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Radiation-induced evolution of microstructure and mechanical properties of stainless steels

A Doctoral thesis

by

G. L. Hankin

Submitted in partial fulfilment
of the requirements for the award of
Doctor of Philosophy of Loughborough University

December 1998

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Abstract

Radiation-induced changes in microstructures often lead to significant changes in mechanical properties of alloys used in the construction of nuclear reactors. It is desirable to test small specimens to make efficient use of the small volumes available in test and commercial reactor cores and also because small specimens are less affected by the sometimes steep flux gradients experienced in reactor cores and the sometimes large temperature gradients developed in the specimens from gamma heating.

Previous work has shown that shear yield and maximum strengths ($\tau_{sy}$ and $\tau_{sm}$) obtained by shear punch test methods (a blanking operation on $\Omega 3$ mm transmission electron microscope disks) can be respectively correlated empirically to tensile yield and ultimate strengths ($\sigma_y$ and $\sigma_{UTS}$) of metallic specimens. When corresponding sets of $\tau$ and $\sigma$ were plotted it was found that they fall along a straight line that extrapolates to a non-zero intercept on the effective shear strength axis. In earlier studies, the slope and offset appeared to be somewhat material dependent. A ductility correlation was also developed that linearly related tensile uniform elongation to effective shear strength data.

In this work, comprehensive yield strength correlations, spanning a wide range of material strengths, have been constructed for a wide range of irradiated and unirradiated austenitic stainless steels. The derived property-property correlations for strength and ductility were shown to be independent of minor compositional changes, thermomechanical starting state, irradiation temperature, dose and dose rate, helium to dpa ratio and details of irradiation history, including the absence of irradiation.

The slope of the yield strength correlation has been experimentally determined to be $\sim 2.1$ for 316 stainless steel which is backed up by theoretical considerations based upon data generated by a finite element model of the shear punch test. In the absence of available tensile specimens, the correlations developed in this work were successfully applied to evaluate tensile yield strength, ultimate tensile strength and uniform elongation of two 304 stainless steel heats and two 316 stainless steel heats that were irradiated in a boiling water reactor.
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Dr. Dan Edwards and dogs, Mic and Tobe, for being such good hosts throughout my stay and the Brew Garden for providing such a rich oasis of fine beers in the desert in which we worked.

M & D Hankin for showing me the way, and finally, but absolutely by no means least, to Miss Louise Mayer for her excellent company and boundless support and affection during the three years of this work.
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1.0 – Introduction

Feelings run high in the debate about nuclear power. It is easy to quote statistics to support each side of the argument. It is, however, a truth universally acknowledged that the energy demand from developing countries will continue to increase and despite the measures that have been taken by developed countries to conserve energy, the global demand on carbon based fossil fuel resources will continue to increase. As everyone is well aware, combustion of carbon based fossil fuels is the main source of carbon dioxide (CO₂) gas emissions that are now known to be responsible for global warming. It has been agreed in recent global energy summits at Kyoto that it is the duty of developed countries to commit to reducing CO₂ emissions.

The solution may lie in the developed countries committing to the long-term use of alternate power sources such as hydroelectric, geothermal and solar power as a supplemental source. In both the short and long term, however, nuclear power can realistically play a large role in committing to the increased energy demands of the world, while satisfying the demand for reduced CO₂ emissions. If there has to be a choice now, the question that needs to be addressed by governmental and environmental agencies is how do we rate the impact of long term nuclear waste storage against that of CO₂ emissions from the domestic and industrial combustion of carbon based fossil fuels.

The work completed in this thesis has been in support of the continuing efforts to develop materials for use in the current line of fission reactors and future applications in fusion reactors. The advance of materials technology and the understanding of neutron irradiation damage mechanisms have been of key importance throughout the development of nuclear reactors and the focus of this work has been to further develop a new technique for obtaining engineering-relevant materials properties from the smallest amount of material.

The strength and mechanical properties of structural materials used in mechanical and civil engineering practice are usually evaluated using a number of different test specimens, e.g., tensile, torsion and impact toughness testing. The geometry of standard specimens has been designed and refined and the practice of each technique has been
documented in international standards such that engineers around the world all work with a common understanding of testing results. In the nuclear power industry, however, standard specimens are, amongst other reasons, physically too big to be cost effectively included in radiation damage experiments. In most situations, scaled down specimens are used. Some small specimen test techniques require size effect correlations and calculations to realise meaningful mechanical properties, but one of the more straightforward practices is the use of miniature tensile test specimens where results can directly be measured.

This work is concerned with the development and application of a small specimen test technique that has the capability of evaluating mechanical properties from a disk of irradiated material that is 3 mm in diameter and 0.25 mm thick. The shear punch test is essentially a blanking operation in which a flat-faced punch is driven at a constant rate through a constrained disk. Prior to this work it was shown, using mostly unirradiated materials, that data from the shear punch test could be related to tensile data for strength and elongation by simple empirical correlations.

In a review paper by Jung in 1996 [1], it was stated that before punch tests can be established as a standard method for determining tensile properties of materials, it would be necessary to answer the question as to whether or not the correlations are valid for irradiated material. It has been the aim of the current work to further develop and demonstrate the validity of the shear punch test technique for a wide range of irradiation-evolved and thermomechanically-induced microstructures.

---

1 Miniature specimens are also desirable with respect to maintaining damage homogeneity in strong neutron flux gradients and the possible effects of gamma heating on temperature gradients within the specimens as well as from the viewpoint of handling radioactive material.
CHAPTER 2 - Background

2.0 – Background

On the 26th September 1944, the initial power run of the first long-term operating nuclear reactor began at Hanford, Washington State, USA, with the express purpose of producing plutonium to make an atomic bomb. The first atomic bomb had a plutonium ($^{239}$Pu) core and was exploded in a test in central New Mexico on the 16th July 1945. The Hiroshima bomb had a uranium core ($^{235}$U) that was produced in a second operation in Oak Ridge, Tennessee, by the physical separation of the fissile $^{235}$U isotope from non-fissile $^{238}$U isotope. The Nagasaki bomb was similar to the New Mexico bomb [2]. In the following years, more nuclear reactors were built to continue the production of plutonium for the cold war effort, but more importantly the first uranium fuelled power reactors were commissioned to produce electricity in the United States and the United Kingdom.

The following sections provide the reader with information about nuclear reactors, detailing their operating conditions and the materials used in them. A literature review on the cause and effects of high-energy neutron irradiation on nuclear reactor materials is included to support some of the findings and as a reference source.

2.1 – The development of nuclear reactors for power generation

The advance of materials technology and the understanding of neutron irradiation damage mechanisms have been of key importance throughout the development of nuclear reactors. Developing techniques for the mechanical testing and qualification of materials used in nuclear reactors is a topic of particular importance for the continuing advance of materials technology. This section concentrates on providing useful background information about thermal and fast reactors and also the current status of fusion reactors. An introduction to the effects and consequences of high-energy neutron irradiation on materials is also presented.

2.1.1 – Thermal reactors

A thermal neutron reactor is configured such that a sustainable nuclear reaction is possible with a fissile fuel controlled by the action of relatively low energy thermal neutrons ($E \approx 1-2$ eV). Normally when an isotope absorbs a neutron, the resulting excitation energy is emitted in the form of a gamma ray. In certain heavy elements, the absorption of neutron tips the balance between the forces of nuclear attraction and
CHAPTER 2 - Background

electrostatic repulsion, which causes the isotope to split into two separate massive fragments, a process known as nuclear fission. A fissile isotope of an element is one that will give rise to nuclear fission on absorption of a thermal neutron (a thermal neutron at room temperature has about 0.025 eV of kinetic energy). $^{235}$U is a natural fissionable isotope of uranium. The first step towards the fission of $^{235}$U involves the absorption of a thermal neutron to form $^{236}$U. The excess energy introduced to the nucleus is sufficient to cause fission and the excited atom splits into two fission fragments, emitting prompt gamma rays and on average 2 - 3 fast neutrons (Fig. 2.1).

\[
^{235}U + _0^1n \rightarrow (^{236}U)^* \\
\text{(absorption of neutron).}
\]

\[
(^{236}U)^* \rightarrow ^{92}_{236}Kr + ^{144}_{56}Ba + 2_0^1n + E
\]

\[
E = 200 \text{ MeV in total.}
\]

Figure 2.1 – Nuclear fission reaction for fissile uranium, after Murray [3].

The emitted neutrons have a range of energies, with neutrons at the high end of the energy spectrum ($E > 0.1$ MeV) being known as fast neutrons. The probability that an energetic neutron will initiate the nuclear fission of $^{235}$U is expressed as the neutron capture cross section ($\sigma$) of the isotope. The neutron capture cross section for the
fission of $^{235}$U is higher for thermal neutrons than it is for the fast neutrons and so it is desirable to slow down or ‘moderate’ the fast neutrons to thermal levels by interaction with a suitable ‘moderating’ medium, such as water or carbon. The moderator slows down the neutrons to thermal energy levels by means of a series of inelastic collisions. More thermal neutrons are then available to cause the fission of more fissile fuel and a sustainable chain reaction is possible. In the case of $^{235}$U, as little as 6.5 MeV of excitation energy is sufficient to yield 200 MeV of energy per fission. Once the chain reaction is initiated, the succession of fission and neutron multiplication continues to operate so long as there is sufficient fuel.

The rate of the chain reaction is regulated by the use of a control medium. The control medium is a material that absorbs neutrons, thus reducing the overall neutron multiplication effect and slowing the chain reaction. Boron is used as a control medium. In the case of an advanced gas-cooled reactor, B$_4$C control rods are lowered into the core to slow the reaction and conversely the rods are retracted in order to increase the reaction rate. In the case of a pressurised light water reactor, boric acid is the control medium, which is contained in the coolant. The concentration of the boric acid is reduced as the fuel is used up in order to maintain a constant power output.

2.1.1.1 - Graphite moderated thermal reactors

The first generation of power reactors to come on line in the UK was the natural uranium fuelled Magnox reactors. The first British commercial nuclear power station to be switched into the national grid was a Magnox reactor at Berkeley, Gloucestershire that was commissioned in 1962.

The key to the success of the Magnox reactor was the design and properties of the fuel element cladding material (seen in Fig. 3.2). The main functions of the fuel cladding is to prevent the radioactive fission products from mixing with the coolant; to resist chemical attack and thermal damage from the fuel and coolant; to resist irradiation damage from energetic particles from the fission reaction and also to provide efficient heat transfer between the fuel and coolant. As natural uranium contains only a low percentage of fissile $^{235}$U, the fuel container or cladding is required to have a low neutron capture cross section, so that as many of the emitted neutrons as possible are available to continue the chain reaction.
Table 2.1 shows the properties of candidate materials for fuel cladding. The Magnox alloy, from which the reactor takes its name, has a low neutron capture cross section and the required thermal properties. The low neutron capture cross section of the Magnox alloy allows for substantial finning on the surface of the fuel capsule which is required for effective heat exchange to the coolant (Fig. 2.2).

Table 2.1 – Candidate cladding and core materials for nuclear reactors [3, 4, 5, 6].

<table>
<thead>
<tr>
<th>Material</th>
<th>Neutron capture cross-section, ( \sigma / 10^{-28} \text{ m (barns)} )</th>
<th>Melting point / K</th>
<th>Comments</th>
</tr>
</thead>
<tbody>
<tr>
<td>Be</td>
<td>0.009</td>
<td>1551</td>
<td>Difficult to fabricate, expensive and toxic</td>
</tr>
<tr>
<td>Al</td>
<td>0.232</td>
<td>933</td>
<td>Low melting point</td>
</tr>
<tr>
<td>Zr</td>
<td>0.185</td>
<td>2125</td>
<td>Zircaloy (98Zr-Sn-Fe-Cr) has a high corrosion resistance</td>
</tr>
<tr>
<td>Mg</td>
<td>0.063</td>
<td>922</td>
<td>Magnesium alloys are limited to low temperature applications</td>
</tr>
<tr>
<td>Magnox</td>
<td>( \sim 0.063 )</td>
<td>( \sim 922 )</td>
<td></td>
</tr>
<tr>
<td>AISI 316</td>
<td>( \text{Fe} = 2.6, \text{Ni} = 4.4, \text{Cr} = 3.1 )</td>
<td>&gt; 1200</td>
<td>High neutron capture cross section limits the use of 316 SS as a cladding material to fast reactors</td>
</tr>
<tr>
<td>PE16</td>
<td>( \text{Ni} = 4.43, \text{Mo} = 2.7 )</td>
<td>( \sim 1500 )</td>
<td>Molybdenum has a high activation</td>
</tr>
<tr>
<td>He</td>
<td>0</td>
<td>-</td>
<td>Coolants used in graphite moderated gas cooled reactors</td>
</tr>
<tr>
<td>CO(_2)</td>
<td>0.036</td>
<td>-</td>
<td></td>
</tr>
<tr>
<td>H(_2)O</td>
<td>( \sim 0.0004 )</td>
<td>-</td>
<td>Moderator and coolant in water moderated reactors</td>
</tr>
<tr>
<td>D(_2)O</td>
<td>( \sim 0.0004 )</td>
<td>-</td>
<td></td>
</tr>
<tr>
<td>Na</td>
<td>0.530</td>
<td>371</td>
<td>Coolant in fast neutron reactors</td>
</tr>
</tbody>
</table>
The coolant and graphite moderator are contained in the same pressure vessel, but the fuel is isolated from both. This configuration is referred to as being heterogeneous. Carbon dioxide is used as a coolant as it also has a low neutron capture cross section. Again, conservation of neutrons is the prime motive for its choice. A complex refuelling operation allows on-line replacement of the Magnox capsules via a 'stand pipe' situated above the core. The use of Magnox as a cladding material in other types of reactor is limited by its low melting temperature. The maximum core temperature (at the core outlet) is around 414°C and the pressure is about 20 Atm. The thermal efficiency of a Magnox reactor is ~30%, which is less than that of a coal-fired power station.

Figure 2.2 – Schematic figure of a Magnox reactor (top) and details of the Magnox fuel elements (below), after Mounfield and Frost [7, 8].
The Advanced Gas-cooled Reactor (AGR) uses an enriched uranium oxide fuel (2-3% \(^{235}\text{U}\)). Neutron conservation is not such an important issue with enriched fuel, and so AISI 316 austenitic stainless steel is used for the cladding as it is less susceptible to corrosion and irradiation damage under the core conditions. The dominant lifetime determinant in the life of an austenitic stainless steel fuel element subject to neutron irradiation is usually dimensional changes due to void swelling [9]. The coolant used is a mixture of carbon dioxide (CO\(_2\)) and helium (He) in a heterogeneous configuration (Fig. 2.3). A higher coolant pressure and temperature is possible (40 Atm. at 650°C), which improves the thermal efficiency of the reactor to ~ 40 % [7]. The enriched fuel allows for a higher power density than is attainable in the Magnox reactor and on line refuelling is possible.

![Figure 2.3 – Schematic diagram of an advanced gas cooled reactor, after Mounfield, [7].](image)

The High Temperature Gas-cooled Reactor (HTGR) works at a higher temperature still than an AGR (~ 785°C), which further improves the thermal efficiency to above 40 %. At this core temperature, the coolant is 100% helium gas since carbon dioxide (CO\(_2\)) would oxidise the graphite cladding. The power density is further improved due to the use of a particle dispersion type fuel. The mixed uranium dicarbide and thorium dicarbide fuel particles are surrounded by a succession of layers of dense impervious graphite and silicon oxide. These fuel pellets are contained within a graphite matrix. This configuration, with fuel and moderator mixed is referred to as being homogeneous. One of the limitations of the HTGR is that refuelling is only possible off load. The HTGR remains an experimental reactor and still requires further investigation.
Table 2.2 summarises the components of graphite moderated thermal reactors. The progression to higher operating temperatures and pressures (where more efficient heat transfer is possible) reflects an improvement in the quality of the pressure vessel steels available.

<table>
<thead>
<tr>
<th></th>
<th>Magnox</th>
<th>Advanced Gas-cooled Reactor (AGR)</th>
<th>High Temperature Gas-cooled reactor (HTGR)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fuel</td>
<td>Natural uranium metal fuelled</td>
<td>2-3 % enriched UO₂</td>
<td>Uranium/thorium carbides</td>
</tr>
<tr>
<td>Cladding</td>
<td>Magnox. Mg alloy with low neutron capture cross-section</td>
<td>Stainless steel or Be alloy tube cladding (P₄ Ma)</td>
<td>Dense impervious pyrolytic graphite cladding.</td>
</tr>
<tr>
<td>Moderator</td>
<td>Graphite</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Reactor Configuration</td>
<td>Heterogeneous</td>
<td>Heterogeneous</td>
<td>Homogeneous</td>
</tr>
<tr>
<td>Control</td>
<td>B₄C</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Coolant</td>
<td>CO₂</td>
<td>He / CO₂</td>
<td>He</td>
</tr>
<tr>
<td>Core pressure</td>
<td>~ 20 Atm.</td>
<td>~ 40 Atm.</td>
<td>~ 38 Atm.</td>
</tr>
<tr>
<td>Coolant outlet</td>
<td>414°C</td>
<td>650°C</td>
<td>785°C</td>
</tr>
<tr>
<td>temperature</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Heat exchanger</td>
<td>Indirect – coolant isolated from fuel</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Power density</td>
<td>0.86 MWm⁻³</td>
<td>3.4 MWm⁻³</td>
<td>6.3 MWm⁻³</td>
</tr>
<tr>
<td>Power output</td>
<td>300 MW</td>
<td>660 MW</td>
<td></td>
</tr>
<tr>
<td>Thermal efficiency</td>
<td>30 %</td>
<td>40 %</td>
<td>&gt; 40 %</td>
</tr>
</tbody>
</table>

2.1.1.2 – Water moderated thermal reactors.

Water moderated reactors can be divided into two categories; Light Water-moderated Reactors (LWR), and Heavy Water-moderated Reactors (HWR). An effective moderator should have a low atomic mass, combined with a low thermal neutron capture cross section. For energy to be imparted from the neutron to the moderating species (an inelastic collision), the molecular mass of the moderator or its constituents should be as low as possible. A neutron colliding with a heavy atom (such as iron) would collide in a more elastic manner, imparting only a small amount of energy to the
iron atom. Light water (H\textsubscript{2}O) and heavy water (D\textsubscript{2}O) are effective moderators since their constituent elements have a low atomic mass.

The first development of a light water reactor was the pressurised water reactor (PWR) as shown in Fig. 2.4. This reactor is both cooled and moderated by light water in a heterogeneous arrangement, with a 2.4 – 4 % enriched \textsuperscript{235}U-oxide fuel. The fuel is contained or clad in helium pressurised tube made from Zircaloy-4 (98Zr-1.2-1.7Sn-Fe-Cr) [3]. The use of stainless steels as a cladding material in light water reactors was abandoned at an early stage, due to both its high neutron capture cross section, and high stress corrosion cracking rate under the core conditions. Zirconium alloys have a low neutron capture cross section (see Table 2.1), and are strongly resistant to corrosion in hot water. A stable ZrO\textsubscript{2} coat is formed on the surface of the alloy, which resists chemical attack from the corrosive environment in the core at the operating temperatures and pressures of pressurised water reactors.

A very high pressure is maintained within the reactor vessel so that the water does not boil. It was feared that changes in the heat exchange characteristics as a result of gas bubbles forming around the heat exchanging interfaces might have a detrimental effect on the performance of the heat exchanger. The concern was that if the heat-transfer between the core and the coolant is restricted, then the core of the reactor might overheat. As a consequence of the high pressure within the reactor core, the fuel has to be pressurised in its cladding with helium.

![Schematic diagram of a pressurised water reactor](image)

Figure 2.4 – Schematic diagram of a pressurised water reactor, after Mounfield [7].
A greater power density is attainable in the core with a liquid coolant than for the gas cooled, graphite moderated reactors (see Table 2.3). Inconel 718 tube heat exchangers are used, as they are particularly good at resisting corrosion from water under high pressures and at high temperatures.

The Boiling Water Reactor (BWR) is a development of the pressurised water reactor that uses the same enriched oxide fuel clad in Zircaloy-2 (98Zr, 1.2-1.7Sn, Fe, Cr, Ni) fuel canisters (Fig. 2.5). Light water (H₂O) is used both as a moderator and coolant at a lower temperature and pressure than in the pressurised water reactor. It was realised through experience with the pressurised water reactor that allowing the water coolant to boil within the core did not have such a detrimental effect as was first thought, and in fact helped the convection flow of the coolant through vertical channels. A direct heat exchanger is used but a lower power density than the PWR is obtained because of poor heat exchange between the boiling water in the secondary heat exchangers. At the lower operation temperature, there are fewer problems with corrosion of core components exposed to the coolant.

The Canadian Deuterium Uranium (CANDU) Reactor uses heavy water as a moderator and a natural uranium oxide fuel clad in Zircaloy. As with the Magnox reactor, the conservation of neutrons from the fission reaction is an important factor when an unenriched fuel is used. The CANDU reactor differs from pressurised and boiling water reactors as it uses heavy water (D₂O) as a coolant, neutron moderator and control
CHAPTER 2 - Background

Heavy water has a low neutron capture cross section, coupled with a high moderating efficiency, the result of which give excellent moderating properties coupled with excellent neutron economy. The moderating ratio for heavy water is 6000, compared to 220 for graphite, which has a moderating efficiency of 0.06 cm\(^{-1}\).

Heavy water, like light water, has good heat exchange properties. The reactor consists of a series of pressure tubes passing through the core, rather than a pressure vessel containing the core (Fig. 2.6). The core is double ended, which allows for on line reloading. The UK’s equivalent to the CANDU reactor is the Steam Generating Heavy Water Reactor (SGHWR), the use of which was dropped in favour of advanced gas cooled reactors.

![Figure 2.6 - Schematic diagram of a Canadian deuterium uranium reactor, after Mounfield [7].](image)

**CANADIAN DEUTERIUM URANIUM REACTOR**

1. Fuel elements
2. Control rods
3. Pressure tubes
4. Calandria
5. Heavy water
6. Light water
7. Steam generator
8. Pump
9. Dump tank
10. Concrete shield
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Table 2.3 – Summary of components of heavy and light water moderated reactors [3, 4, 5, 6].

<table>
<thead>
<tr>
<th></th>
<th>Pressurised Water Reactor (PWR)</th>
<th>Boiling Water Reactor (BWR)</th>
<th>Canadian deuterium uranium reactor (CANDU)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fuel</td>
<td>2.4-4% enriched UO₂ pellets</td>
<td>2.4-4% enriched UO₂ pellets</td>
<td>Natural UO₂ fuel</td>
</tr>
<tr>
<td>Cladding</td>
<td>Sealed Zircaloy-4 pressurised fuel element tubes</td>
<td>Zircaloy-2 cladding</td>
<td>Zircaloy-2 pressurised fuel element tubes</td>
</tr>
<tr>
<td>heat exchanger</td>
<td>Indirect</td>
<td>Direct</td>
<td>Indirect</td>
</tr>
<tr>
<td>Moderator</td>
<td>H₂O</td>
<td>H₂O</td>
<td>D₂O</td>
</tr>
<tr>
<td>Control</td>
<td>B₄C</td>
<td>B₄C</td>
<td></td>
</tr>
<tr>
<td>Coolant</td>
<td>H₂O</td>
<td>H₂O</td>
<td></td>
</tr>
<tr>
<td>Coolant pressure</td>
<td>~ 155 Atm.</td>
<td>~ 72 Atm.</td>
<td>~ 90 Atm.</td>
</tr>
<tr>
<td>Coolant outlet temperature</td>
<td>320°C</td>
<td>285°C</td>
<td>300 - 400°C</td>
</tr>
<tr>
<td>Power density</td>
<td>100 MWM⁻³</td>
<td>55 MWM⁻³</td>
<td>10 MWM⁻³</td>
</tr>
<tr>
<td>Power output</td>
<td>300 – 1300 MW</td>
<td>900 MW</td>
<td>900 MW</td>
</tr>
<tr>
<td>Thermal efficiency</td>
<td>32%</td>
<td>33%</td>
<td>36-40%</td>
</tr>
<tr>
<td>Refuelling</td>
<td>Refuelled off line.</td>
<td>Refuelled off line.</td>
<td>On line refuelling.</td>
</tr>
</tbody>
</table>

2.1.2 – Fast reactors

Fast neutrons were previously introduced as those having energies above ~ 0.1 MeV. A fast neutron is capable of causing the fast fission of either ²³⁵U or the non-fissile ²³⁸U isotope (natural uranium). Fast neutrons are, however, more likely to be captured by ²³⁸U, forming ²³⁹U which subsequently decays to ²³⁹Pu, which is another fissile isotope:

\[
\text{²³⁸ U} + \text{¹n} \xrightarrow{\text{neutron capture}} \text{²³⁹ U} \xrightarrow{\text{β}\text{23.5 min}} \text{²³⁹ Np} + \text{⁰e} \xrightarrow{\text{β}\text{2.35 day}} \text{²³⁹ Pu} + \text{⁰e}
\]

(2.1)

On average, more than one fast neutron is produced during the fast fission of the fuel. The number of neutrons produced per neutron absorbed to sustain the chain reaction is called the reproduction factor. The reproduction factor for fast neutrons from the fission
of $^{235}\text{U}$ is ~ 2.3. The excess fast neutrons are available to convert ‘fertile’ $^{238}\text{U}$ to fissile $^{239}\text{Pu}$ by neutron capture. To maintain a fast fission chain reaction the mixed fuel (83% $\text{UO}_2$ - 17% $\text{PuO}_2$) is enriched with 20-30% $^{235}\text{U}$, and no effective moderator is used.

As the $^{235}\text{U}$ content of the fuel depletes, the fissile $^{239}\text{Pu}$ produced from the $^{238}\text{U}$ in the core of the reactor helps to sustain the fast chain reaction [1]. Additional fissile $^{239}\text{Pu}$ is produced in a ‘breeding blanket’, which surrounds the core. One ‘breeder reactor’ can produce sufficient fissile fuel for several conventional thermal reactors. The non-fissile $^{238}\text{U}$ in the breeder blanket is obtained from natural uranium or depleted uranium left over from other reactors or fuel enrichment plants.

In the breeder reactor a liquid sodium coolant is used, which has excellent heat transfer properties and a low neutron capture cross section (Table 3.1), i.e., does not effectively moderate the fast neutrons. Sodium does however activate in the core heat exchange circuit to form the radioactive isotope $^{24}\text{Na}$. An intermediate heat exchanger is used in a heterogeneous arrangement to prevent radioactive sodium from circulating outside of the reactor containment. Fast reactors in this configuration are referred to as Liquid Metal Fast Breeder Reactors (LMFBR’s). The small reactor core of a LMFBR (Fig. 2.7) has a power density of about 900 MWM$^{-3}$, compared to 100 MWM$^{-3}$ for a pressurised water reactor. This is possible due to the enriched fuel and the fact that there is no requirement for a moderator to be present in the core.

Figure 2.7 – Schematic diagram of a liquid metal fast breeder reactor, after Mounfield [7].
The cladding, coolant channels, reactor vessel and many of the structural parts of LMFBR's are made from austenitic stainless steels or nickel based alloys such as Nimonic PE16 [10]. The materials are chosen for their high temperature properties, void swelling resistance and resistance to corrosion in a liquid sodium environment.

2.1.3 - Fusion reactors

Fusion reactors represent the next step in the generation of nuclear power. Energy in a fusion reactor is released as a result of combining heavy hydrogen nuclei, resulting in the formation of helium. In this reaction, the atomic mass of the products is less than that of the reactants (Fig. 2.8). The difference between the masses of the reactants and products is converted to energy according the equation $E = (\Delta m) \times c^2$. A composite reaction process involving the fusion of deuterium to form tritium and hydrogen makes up the overall reaction, which can be summarised by Equation 2.2.

$$^2_1D + ^3_1T \rightarrow ^4_2He + ^0_1n \quad \text{Energy released} = 17.6 \text{ MeV} \quad (2.2)$$

Figure 2.8 - Overall reaction for the fusion of deuterium and tritium to form helium and a neutron.

The deuterium-tritium reaction will hereafter be referred to as the D-T reaction. In order for a fusion reaction to take place reactants nuclei must have enough energy to overcome the repulsive electrostatic (Coulomb) force and approach each other sufficiently close that short-range attractive nuclear forces become dominant. Immense
temperature and pressure are required in order to physically bring the reactant nuclei together. The criteria which represents the conditions required is referred to as the Lawson criterion:

$$\eta \tau \geq 10^{30} \text{sec.m}^{-3}$$

Where $\eta$ is the fusion fuel particle number density and $\tau$ is the confinement time for the reaction to occur.

The reactant nuclei have to be within a certain proximity of each other by combination of temperature and pressure for a certain period of time for the fusion reaction to occur. The gas temperature must exceed $5 \times 10^7$ K before a significant D-T fusion rate is feasible [11]. At such temperatures, the gaseous products exist as a macroscopically neutral collection of ions and unbound electrons, which together is called a plasma.

Materials technology is the key factor whose advancement will lead to the development of a fusion reactor. The emitted neutrons from the D-T reaction have a very high energy (14 MeV) and will cause extensive irradiation damage to the plasma facing materials (see later sections). A more favourable reaction would be the fusion reaction D-D as the products of the reaction are Tritium and Hydrogen with no emitted neutrons. However, the D-D reaction requires a larger ignition temperature and gives a lower energy yield at a lower reaction rate and so constructing a reactor that can ignite and sustain the D-T reaction is the primary goal [12].

The simplest continuous confinement configuration for a plasma is a toroidal magnetic field. The basis for containment of the plasma is that charged particles spiral around magnetic field lines. If the field lines form a continuous loop, i.e., a toroidal circuit, then the plasma can be contained by the magnetic field indefinitely. The radius of the spiral is inversely proportional to the strength of the magnetic field. The earliest conceptual design for such a plasma confinement was invented in the USSR in the 1960s and was named the ‘Tokamak’, a Russian acronym for toroid-chamber-magnet-coil (Fig. 2.9). In a Tokamak, plasma is heated in a toroidal vacuum vessel and confined away from the vessel walls by a magnetic field.

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1 Void swelling can be the lifetime limiting factor for austenitic stainless steels in fast neutron reactors (see Section 3.3.3).
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Figure 2.9 – Schematic diagram of a developmental Tokamak fusion reactor, after Stacey, Jr. [11].

The toroidal field is produced by a set of toroidal field coils, which encircle the plasma. The poloidal field is produced by axial (toroidal) current in the plasma, which is induced by the transformer action of a set of primary ohmic (poloidal) heating coils. Additional coils, around the outside of the vacuum vessel, shape and position the plasma. The Tokamak concept has shown the greatest potential for success in the design of a fusion reactor. The progress towards ignition conditions achieved in successive generations of fusion experiments is shown in Fig. 2.10.

Each step in the development of fusion reactors has been marked by an increase in the size of the reactor and the strength of the magnetic field used to contain and confine the plasma. The progress of the current generation of experimental fusion reactors (TFTR, JT-60, JET and T-15)\(^2\) towards ignition conditions show that magnetic confinement fusion is scientifically feasible with a larger Tokamak. The International Thermonuclear Experimental Reactor (ITER) represents the next generation of fusion reactors. The volume of the plasma in ITER will be approximately 15 times larger than the largest magnetically produced plasma so far. The magnets that contain the plasma

\(^2\) Acronyms: TFTR (Toroidal Field Test Reactor) JT-60 (Japanese Taurus–60), JET (Joint European Taurus), T-15 (Tokamak 15).
are kept at cryogenic temperatures needed for superconductivity. A cross section of the ITER design is shown in Fig. 2.11.

Figure 2.10 - Progress in controlled fusion.

Figure 2.11 - Cross-section of the ITER design.
Energy is induced in the plasma by resistance heating, and the reaction can be initiated by injecting compressed liquid hydrogen pellets ($10^{20}$ atoms) into the plasma. The products of the reaction carry the energy generated in the D-T reaction. The energy possessed by the helium (3.5 MeV) will radiate into the plasma, helping keep the plasma above the ignition temperature. The 14 MeV neutrons will escape the magnetic confinement of the toroid, as they do not carry any electrical charge. The high-energy neutrons will slow down by a series of collisions with the first wall, blanket and shield structures. The plasma facing material must be able to withstand both the high temperature and high-energy neutron flux. The first wall material will transfer heat to water-cooled stainless steel channels. The water will act as a good moderator of the 14 MeV neutrons and will carry the heat away from the reactor for electricity generation. Beyond the water-cooled channels the blanket structure will circulate liquid lithium to breed tritium for fuel. Finally a shield structure of boronated steel or tungsten carbide balls will provide a neutron shield around the reactor [MO1]. It is likely that the shield and blanket structures will be fabricated from austenitic stainless steel because of the extensive database available on its radiation performance.

The divertor exhausts the flow of energy from charged particles produced in the fusion reactions and removes helium ashes (alpha particles formed during the fusion reaction) and other impurities from the reactions.
**2.2 – The effects of neutron irradiation**

Large-scale effects of radiation on solids were observed only after nuclear fission reactors were developed in the 1940s [13]. The effects of irradiation on these components are usually deleterious to the material properties and are referred to as 'radiation damage'. In an early review on the radiation effects on solids [14], it was recognised that the structural materials within the core of a nuclear fission reactor, i.e. the materials of the cladding, supports, pressure vessel and heat exchanger tubes would be subject to very heavy bombardment by energetic particles such as neutrons and fission fragments. At this time, Wigner had already completed rudimentary investigations on the fractions of atoms that would be displaced from their normal positions in the graphite moderator and concluded that the effects could not be ignored.

Radiation damage describes events occurring on the microscopic level as a result of energetic particles interacting with the atoms of the bombarded material to produce displacements, ionisation, nuclear reactions and localised heating. The macroscopic changes that may occur as a result of radiation damage are known as 'radiation effects'. These include dislocation generation, radiation-enhanced diffusion, radiation-induced segregation, displacement mixing, void swelling and irradiation creep.

Radiation effects ultimately limit the lifetime of a component and so for the continued development of fission reactors and the design of fusion reactors, structural effects of radiation on materials an area of great technological importance.

**2.2.1 – Neutron interactions**

Irradiation damage is measured by the average number of times each atom of the crystalline solid has been moved from its lattice position, or displacements per atom, (dpa). The rate at which this damage occurs, under the effects of a particular neutron flux is referred to as the displacement rate. Typical values of displacement or dose rate are $10^{-6}$ dpa.s$^{-1}$ for a thermonuclear reactor and $10^{-7}$ or $10^{-8}$ dpa.s$^{-1}$ for a thermal reactor. Phenomena such as swelling, creep and non-equilibrium segregation can be linked to the production of dislocation loops and cavities that are driven by vacancy and interstitial supersaturations within the polycrystalline structure.
Neutron irradiation results in the formation of high levels of point defects within the microstructure. The high-energy neutrons bombard the crystalline lattice, displacing solute atoms into interstitial positions and forming vacancies. Since neutrons have no charge, they may pass close to an atom without incident or can collide with the nucleus of an atom. Neutrons can lose their kinetic energy by elastically colliding with the lattice atoms or by nuclear reactions with the lattice atoms. Damage caused by high-energy neutrons (0.1 MeV – 14 MeV) is widely spread out throughout a material since neutrons with a high energy have a low probability (or a low cross section) for collisions with lattice atoms. The sequence of collisions occurring when a high-energy neutron enters a lattice can be spread over a distance of several centimetres unless it terminates in a nuclear reaction.

2.2.1.1 – Electronic excitations
Inelastic interactions between high-energy neutrons and electrons in the solid will lead to excitation, ionisation and the transfer or exchange of electrons within the solid. In metals, electronic disturbances are quickly relaxed, with the main result being heat generation. In insulators, electron losses can cause property changes.

2.2.1.2 – Nuclear reactions
Nuclear reactions occur when an incoming neutron collides with an atom nucleus, as is the case in the fission of $^{235}$U (see Fig. 2.1). The probability of a nuclear reaction with a lattice atom is expressed by its neutron capture cross section.

Nuclear reactions can lead to property changes in several ways. Some nuclear reactions produce gaseous products that can lead to modifications to the microstructure, e.g., the (n, $\alpha$) reaction in structural alloys lead to the formation of helium bubbles, which lead to extreme embrittlement and swelling. Other nuclear reactions that can produce gaseous products (helium and hydrogen) are: (n, $\alpha$), (n, p), (n, t), (n, n', $\alpha$) and (n, d). Each of these reactions are only appreciable for neutron energies above a few MeV and so they are of particular concern in the materials proposed for the first wall and blanket structures in fusion reactors.

Nuclear reaction products will contribute to the level of impurity atoms ($M'$) present in the components, however the levels of impurities produced from the nuclear reactions
during the lifetime of a component are usually lower than their solubility limits in the matrix. The effects of impurities on the phase diagrams obtained under equilibrium conditions may not be applicable under irradiation conditions. A secondary effect of nuclear reactions is that the reaction products usually have recoil energies in the eV and MeV range and can therefore sometimes contribute to displacement damage. For example lithium from the \( \text{(n, } ^{10}\text{B)} \) reaction and iron from \( \text{(n, } ^{58}\text{Ni)} \) can contribute to the damage production [15]. The production of helium by fast neutrons cannot be avoided by selecting alloys of special composition [13], but it can be reduced by avoiding elements that are known to form helium such as boron and nickel.

2.2.1.3 – Elastic collisions

Elastic collisions occur between incoming neutrons and lattice atoms. Energy is transferred to the lattice atom, causing it to recoil. The atom with which the incoming neutron or particle collides with is referred to as the primary knock-on atom [14].

The probability of formation of a primary knock-on atom is a function of the type of particle, its energy and the target atom. If sufficient energy is imparted to the primary knock-on atom during a collision, a replacement collision sequence can be initiated which leaves a vacancy at the starting point and generates an interstitial at some distance (Fig. 2.12). Replacement collision sequences (also known as ‘Dynamic Crowdions’) will occur preferentially in the close-packed directions of \(<110>\) and \(<100>\) in a face centred cubic lattice [16]. A single primary knock-on atom can be responsible for many tens or hundreds of subsequent collisions – hence the term ‘collision cascade’ is used to describe the events occurring as a result of high energy neutron irradiation.
Figure 2.12 – A schematic two-dimensional diagram of a cascade in bcc material created by a fast neutron, after Seeger [17]. Open circles represent lattice atoms, filled circles represent either interstitial atoms or atoms not yet in equilibrium positions and squares represent vacancies. In this representation, the primary knock-on atom (PKA) has initiated a replacement collision sequence that has created an interstitial (top left). The PKA itself is the nearest neighbour to the vacancy created at the site the initial collision.

The primary knock-on atom ends up as the nearest neighbour to the vacancy and the last atom in the replacement collision sequence has insufficient energy to replace its neighbour and so two atoms attempt to share one lattice site. Instead of the interstitial atom occupying one of the possible crystallographic interstitial sites (octahedral or tetrahedral), it takes a dumbbell or split interstitial configuration where two atoms share one lattice site (Fig. 2.13). As a rule, interstitials are formed as a result of replacement collision sequence, and not by direct injection of a low energy primary knock-on atom [18].

The vacancy-interstitial pair of a replacement collision sequence is known as a Frenkel pair. Theoretical and experimental work has shown that about 25eV has to be transferred to an atom to form a Frenkel defect [19]. Production of Frenkel defects is
the dominating cause for property changes in metallic and most other materials in fission and fusion reactors [13].

![Stable self-interstitial atom configurations in f.c.c. and b.c.c. metals.](image)

Recombination of Frenkel pairs may occur if the distance between the vacancy at the start of the replacement collision sequence and interstitial at the end of a replacement collision sequence is less than a certain threshold for stability. The threshold is measured as a volume (~10^2 atomic volumes), containing the vacancy and self-interstitial, within which will the two will spontaneously recombine [20]. The threshold displacement energy, $E_d$, to form a stable Frenkel pair can be measured in 'real time' experiments by irradiation of thin foils in a high voltage electron microscopes (HVEM) with electrons with increasing energies. The introduction of Frenkel defects can be measured as a function of electrical resistivity. Thermal neutrons usually have insufficient energy to cause atomic displacements (depending on the size of $E_d$), but can figure in (n, $\gamma$) nuclear reactions, which lead to recoil energies above $E_d$, contributing indirectly to displacement damage. Low energy recoils, such as the last atom in the replacement collision sequence, transfer only momentum and are called focusons (see Fig. 2.12).

When recoil energies are much larger than $E_d$, the primary knock-on atom will collide with several atoms, giving each them enough kinetic energy to displace them from their lattice sites to make similar collisions of their own in a branching tree-like structure known as a collision cascade. The energy of the primary knock-on atom is dissipated in
a short period of time (~100 femtoseconds) and produces a number of atoms moving with near thermal velocities.

As the primary knock-on atom slows down, the cross section for displacement collisions increases. The remaining energy of the moving atom is distributed to the surrounding atoms in a short distance in a displacement or thermal spike. Each atom in this core region (Seeger zone, S-zone) can have an average energy of 1 eV, which would be equivalent to suddenly raising the temperature to 10,000°C [14].

Wigner, Seitz and Seeger established a good fraction of the fundamental theory behind the nature of radiation damage prior to 1960. The major problem faced when trying to quantify these effects is the high atomic and time resolution that is required in order to study these effects. A collision cascade can complete after only a few picoseconds. A great deal more has been learned about the details of elastic collision cascades with molecular dynamics (MD) models.

2.2.2 - Molecular dynamics

Molecular dynamics are a realistic treatment of atomic motions and crystal effects and can simulate collisions of complex projectiles. MD models solve, in small time steps, the classical equations of motion and so follow the individual positions and kinetic energies of all atoms as a function of time. The static properties of cohesive energy, elastic constants, surface energy and vacancy formation are correctly reproduced by the model. Replacement collision sequences were a prominent feature of the first MD simulations of damage production as seen in the work by Gibson et al. [21] (Fig. 2.14).

---

3 1 femtosecond = $10^{-15}$s and 1 picosecond = $10^{-12}$s.
MD has shown clearly the two stages of defect production: first the ballistic phase, where the primary knock-on atom creates a collision cascade and secondly the thermal spike [22, 23]. The intensity of the motion decreases as the spike cools down after ~10 ps, but during the early stages of the spike, a shock front injects self-interstitials into the surrounding lattice by replacement collision sequences or gliding. The core of the spike cools rapidly from a liquid like phase to the ambient temperature. A vacancy rich depleted zone is left behind. Recombination can then occur. From MD analysis it can be concluded that more than 70% of interstitials formed by high-energy cascades perform only 1 or 2 jumps before recombination [24, 23, 25].

2.2.3 – Defect clustering

When present in large supersaturations in f.c.c materials, such as the Seeger-zone of the cascade, the vacancies tend to arrange themselves in two-dimensional (2D) disks in (111) planes [26]. The vacancy aggregate will collapse to form a vacancy dislocation loop in order to minimise the energy of the aggregate (Fig. 2.15a). When the loop
collapses, a stacking fault is introduced in the stacking order of the (111) planes. The dislocations around the circumference of the vacancy loops have a Burgers vector of $\mathbf{b} = a/3 [111]$ and are referred to as intrinsic or negative Frank faulted loops. Faulted vacancy loops can grow by positive climb if more vacancies diffuse to the loop edge, or shrink by negative climb when interstitials diffuse to the loop edge. Interstitials can also reduce their energy by clustering in 2D platelets on in the (111) plane in f.c.c. materials to form extrinsic or positive Frank faulted loops. An extrinsic stacking fault is introduced into the layering order of the (111) planes (Fig. 2.15c). Faulted interstitial dislocation loops have the same Burgers vector as the vacancy dislocation loops and so both are sessile in nature as their Burgers vectors do not lie on one of the [110] slip directions on which conservative glide can occur. Since faulted loops are essentially immobile objects, they act as barriers to network dislocations in a similar way to precipitates and cavities. Interstitial loops are very stable, even at high temperatures because of the large reduction in energy associated with clustering. Vacancy loops are not, however, thermally stable in face-centred-cubic materials at operating temperatures typical of fission reactors because of the high line tension associated with dislocation loops, and so they tend to reduce in size by thermal vacancy emission. At low irradiation temperatures, the small Frank loops (2-3 nm in diameter) that form under neutron irradiation are known as ‘black spot’ damage.

Faulted loops must first become unfaulted before they can glide as glissile prismatic loops. This can occur by interaction of the faulted loop with a partial dislocation to form perfect or prismatic dislocation loops, which have Burgers vectors in a close-packed direction $\mathbf{b} = a/2 [110]$ as seen in Figs. 2.15 b and d. Prismatic dislocation loops are glissile and can develop into dislocation networks [27].
2.2.4 – Dislocation evolution during irradiation

The dislocation density of unirradiated steel is largely dependent on the initial cold work level, the solute strengthening and type and density of precipitates [9]. The dislocation density of a material is quoted as fraction of the total dislocation line length, divided by the volume analysed. In the absence of irradiation, the dislocation networks found in austenitic (face-centred-cubic) stainless steels consist of \( b = (a_0/2) \langle 110 \rangle \) type dislocations. Network dislocation densities vary from \( <10^{12} \text{ m}^{-2} \) in solution annealed to \( \sim10^{16} \text{ m}^{-2} \) in 20% cold worked materials [28].

Under irradiation conditions, the dislocation microstructure will evolve towards a steady state level that is achieved when the rate of new network dislocation line length equals the rate of dislocation annihilation at grain boundaries and surfaces. The neutron-induced saturation density of network dislocations has been measured for stainless steels at \( \sim 6 \pm 3 \times 10^{17} \text{ m}^{-2} \), a level that is independent of starting state, temperature, displacement rate and He/dpa ratio [9].
2.2.5 – Measuring irradiation damage

The average number of displacements per atom (dpa) occurring is used as a measure of irradiation damage. Incorporated to a first approximation, dpa is a measure of the neutron energy dependent response of the material under irradiation [29]. The dpa unit allows us to directly compare material effects measured at very different irradiation facilities [22] (such as mixed spectrum and fast reactors, particle accelerators etc).

Measurement of the neutron exposure is required to allow us to determine an appropriate measure of irradiation exposure that can be compared with other irradiation conditions. The neutron exposure is a function of the neutron fluence and the neutron energy spectrum. The neutron fluence is the number of neutrons passing through a unit area cross section regardless of direction of travel integrated with respect to time (n/cm²). One also needs to know the recoil spectra for reactions of elements in a particular reactor, e.g., for nickel, iron and chromium in austenitic stainless steels, i.e. taking into account the different collision cross sections of each of the constituent elements.
2.3 – Effects of continuous point defect evolution on materials properties

Radiation damage occurs as a result of energetic particles interacting with the atoms of a material to produce displacements, ionisation and localised heating. As discussed in the previous chapter, point defects such as vacancies and interstitials are introduced into the microstructure as a function of the energy and fluence of the irradiating particles. The small percentages of point defects produced by radiation damage that survive recombination are collectively responsible for radiation effects. The presence of point defects in large supersaturations can lead to changes in the material properties by one of several different irradiation effects.

2.3.1 – Point defects under thermal conditions

Vacancies and interstitials are known as intrinsic point defects and can be introduced into crystal systems by virtue of temperature alone. For all temperatures above zero Kelvin, there is a thermodynamically stable concentration of vacancies and interstitials. The equilibrium concentration of vacancies or interstitials is measured as the fraction of the total number of atomic sites and is an exponential function of the energy of formation of the vacancy or interstitial and the free energy available. The energy of formation of a vacancy is the energy required in removing one atom from the lattice and placing it on the surface of the crystal (~ 1 eV for f.c.c. materials). The thermodynamically stable concentration of vacancies in copper, for instance, at 300 K is \(~ 1.5 \times 10^{-22}\) [28]. The energy of formation of an interstitial is approximately four times greater than that of a vacancy, and as a consequence of the exponential relationship between the thermal energy available and the point defect concentration, the number of interstitials at thermal equilibrium is negligible compared with the number of vacancies.

Since the concentration of thermal vacancies increases with temperature, the effect of excess vacancies created by irradiation damage becomes negligible at temperatures > 0.6 \(T_{mp}\) where the thermal vacancy concentration is extremely high. Greater thermal mobility of the vacancies and interstitials increases the recombination rate and rate of point defect annihilation at sinks. Irradiating at high temperatures decreases the probability that the vacancies and interstitials will have a significant effect on the microstructure.
2.3.2 – Point defects production under irradiation conditions

The concentrations of vacancies and especially interstitials created by irradiation damage far exceed the concentrations present in thermal equilibrium. The excess point defects produced by irradiation that escape recombination during the production phase migrate thermally over significant distances at elevated temperatures before being eliminated by recombination or by annihilation at sinks such as grain boundaries and dislocations. The spatial separation between defect production and annihilation leads to persistent defect fluxes between the grain interior and grain boundary [30]. A self-interstitial in a f.c.c. material is assumed to migrate via orthogonal jumps into nearest neighbour positions on the <100> planes common to the dumbbell axis, i.e., in effect, one atom of the interstitial pair is exchanged between two nearest neighbour lattice atoms [31]. The migrational energy required for an interstitial is appreciably less than that for a vacancy, where an atom in the lattice must move from a nearest neighbour position into the vacancy position. Continuous point defect migration can lead to one of many radiation-enhanced or radiation-induced effects. Radiation effects in steels include radiation-enhanced diffusion, radiation-induced segregation, displacement mixing, void swelling and irradiation creep.

2.3.3 – Radiation effects

The following section describes some different radiation-enhanced and radiation-induced effects. It is important to realise that radiation-enhanced effects are those effects that tend to move a system closer towards the equilibrium state much faster than it would in the absence of irradiation. Irradiation induced effects are those that would never occur in the absence of irradiation.

2.3.3.1 – Displacement mixing

Displacement mixing is one of the simplest effects associated with radiation damage. It includes all atomic arrangements that occur during the cascade event including atom and defect motions associated with the injection of high energy irradiating particles. Displacement mixing is the dominant atomic transport mechanism during irradiations at low temperatures (≤ 0.3 T_{mp}) when point defects are immobile [30].

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2.3.3.2 – Solute segregation and radiation enhanced diffusion (RED)

Solute segregation in metals can occur under thermal equilibrium and non-equilibrium conditions and either in the presence or absence of irradiation. Figure 2.16 shows a schematic of typical profiles associated with three types of solute segregation to a grain boundary. Equilibrium segregation (ES) of impurity atoms to grain boundaries is thermodynamically possible at any temperature [32]. The segregation may be limited to a monolayer of impurities on the grain boundaries and the driving force for this is the reduction of energy associated with placing the impurity atoms in a strain-free environment on the grain boundaries (see Fig. 2.16). It may be thermodynamically favourable for ES to occur at room temperature, but the kinetics are slow and therefore long times are associated with solute build-up at the grain boundaries. At higher temperatures, the ES may be kinetically possible, but the thermodynamics do not favour equilibrium segregation.

![Diagram of equilibrium segregation (ES), thermal non-equilibrium segregation (TNES) and radiation-induced segregation (RIS)](image)

Figure 2.16 – Summary of equilibrium segregation (ES), thermal non-equilibrium segregation (TNES) and radiation-induced segregation (RIS), after Faulkner [32].

Non-equilibrium segregation (NES) can occur as a result of a system moving towards thermal equilibrium. As discussed earlier, the equilibrium concentration of vacancies in a crystalline lattice is a function of the thermal energy available and the equilibrium concentration of interstitials is negligible. If a material is quenched from a high temperature the excess vacancies will diffuse to sinks such as grain boundaries to
restore equilibrium concentrations at that temperature. The flow of vacancies towards
the point defect sinks results in a vacancy concentration gradient in the proximity of the
sinks. Some impurities or solutes form an association or complex with the vacancies
and become concentrated around the point defect sink by a mechanism known as solute
drag. The vacancy-impurity complex, \((v + A)\) in Fig. 2.16 arises due to the relaxation in
the energy of the atoms surrounding the vacancy. It is energetically favourable for the
impurity to reside in this region in the same way as carbon may assume an energetically
favourable position in a dislocation core. Also by this reasoning, an impurity with a
large oversize 'misfit' is likely to take precedence over an impurity with a lesser 'misfit'
in order to reduce the total energy of the system.

Radiation enhanced diffusion (RED) and radiation-induced segregation (RIS) are
processes that occur simultaneously by the influence of point defect fluxes. Radiation
enhanced diffusion refers to the enhanced diffusion of alloying elements in a material
by random motion of point defects during high temperature irradiation. RED tends to
bring a system towards a thermodynamically preferred state quicker than it normally
would by thermal diffusion alone.

The major difference between radiation induced segregation (RIS) and NES is the
involvement of interstitial-solute complexes as well as vacancy-solute complexes.
Under irradiation conditions, interstitials are produced in excess concentrations of
thermal equilibrium levels. Interstitials form complexes with impurity atoms because
the dilatational strain associated with an interstitial atom makes it energetically
favourable for an undersized solute (u/s) to be present in lattice positions immediately
next to or as a part of the interstitial [31, 33]. Interstitial-solute complexes are more
stable and may feature more in RIS than vacancy-solute complexes. The numbers of
freely migrating interstitials can be limited by the production bias mechanism, but
significant numbers are still likely to be available for solute drag. The constant flux of
point defects towards microstructural sinks during irradiation provides an enhanced
driving force for solute drag. As a rule, vacancy-solute complexes can form with either
oversize or undersize solutes, but interstitials only form a strong binding with undersize
solutes [32]. The preference for undersized solutes can lead to negative NES for
oversized solutes, which can result in radiation-induced depletion (RID) of oversized
(o/s) solutes (See Fig. 2.16). An example of RIS and RID is grain boundary segregation.
of nickel (u/s) and depletion of chromium (o/s) in austenitic stainless steels. Depletion of chromium at grain boundaries can contribute to intergranular failure by irradiation assisted stress corrosion cracking (IASCC).

A second mechanism contributing to RIS is the inverse Kirkendall [34] effect. The flux of vacancies and towards defect sinks creates a net flow of atoms away from them, such as when a void grows. The faster diffusing elements will move away quicker and become depleted. Slower elements will retain a degree of enrichment since they do not move away as quickly, e.g., In a Fe-Cr-Ni ternary steel, where \( D_{Cr} > D_{Fe} > D_{Ni} \), nickel will become enriched near a microstructural sink.

It is incorrect to assume that the observed segregation is only due to one mechanism. The segregation that is observed experimentally is the net result of all the possible mechanisms of RIS. It is probably true that certain mechanisms will dominate under certain conditions, for instance, in the Fe-Cr-Ni system the self-interstitial mechanism can be considered be of secondary importance because the inverse-Kirkendall vacancy mechanism, by itself, accounts for measured segregation. The concentration gradients that arise from RIS during irradiation would soon disappear by thermal diffusion if the material were removed from the radiation environment and kept at the same temperature.

2.3.3.3 – Radiation induced precipitation and phase transformations

Austenite is an interstitial solid solution of carbon in f.c.c. iron which, for basic 0.8% C steels, is stable between 723 and 1493°C. Under equilibrium cooling conditions, and below the upper critical (\( A_3 \)) temperature (723°C in the example), the f.c.c. austenite phase would undergo a phase transformation to pearlite (alternating layers of b.c.c. ferrite and cementite) before it reached room temperature. Austenitic stainless steels are as formed by rapid cooling of the Fe-Cr-Ni alloy from the stable austenite regime (above \( A_3 \)). At room temperature the austenite has insufficient thermal energy for atomic diffusion to allow the rearrangement of the crystalline structure to restore equilibrium conditions. During thermal ageing at 550-900°C, there is sufficient thermal energy to allow the thermal diffusion and precipitation of the supersaturations of alloying elements such as Cr, Mo, Si and C to form carbide phases (\( M_{23}C_6 \) \( M_6C \)) and
intermetallic phases (Laves, $\sigma$, $\chi$) [35]. Trace levels of Nb, Ti and P can also lead to other more complex precipitates being formed.

During irradiation, solute segregation and solute drag mechanisms can lead to an enhancement of thermal ageing processes at lower temperatures. There are three classifications for precipitate evolution during neutron irradiation of 316 type stainless steels, namely radiation enhanced (or retarded), radiation modified and radiation induced phase changes. Radiation enhanced or retarded thermal phases are no different than if they were formed during thermal ageing, but they may form faster at lower temperatures (enhanced) or slower at higher temperatures (retarded). Examples of radiation enhanced phases in austenitic stainless steels are $\text{M}_{23}\text{C}_6$, $\text{M}_6\text{C}$, Laves, $\sigma$, $\chi$, etc. [9]. Radiation modified phases occur during both reactor irradiation and during thermal ageing, but the irradiation produced phase compositionally different. They include $\text{M}_6\text{C}$, Laves and $\text{M}_2\text{P}$. Radiation-induced phases are uniquely produced by reactor irradiation and would not be found in the same material during thermal ageing. They include the $\text{Ni}_3\text{Si}$ ($\gamma'$), G-phase silicides ($\text{M}_6\text{Ni}_{16}\text{Si}_7$), MP, $\text{M}_3\text{P}$ and $\text{M}_2\text{P}$.

RIS frequently results in the precipitation of second phases on the surfaces of microstructural sinks and can result in bodily phase transformations in the surrounding area. AISI 304L austenitic stainless steel, for example, contains 9.3% Ni (an austenitic phase stabiliser). The nickel can be depleted from matrix to void surfaces by RIS to such an extent that a phase transformation occurs in the matrix to form b.c.c. ferrite. The only place where austenite can exist is in a thin shell around the void, where the nickel is still present in sufficient quantities. AISI 316 stainless steel (12% Ni) resists early phase transformations as the nickel is present in sufficient quantities to avoid $\alpha$ formation. If RIS can be suppressed, radiation-induced phase formation can be controlled.

2.3.3.4 – Void swelling
Void swelling was first observed in 316 stainless steel fuel pin cladding by Cawthorne and Fulton in 1967 [36]. The phenomenon is connected to excess concentration of vacancies compared to interstitials. The concentration of freely migrating interstitials is low because dislocation cores attract interstitials (and solute atoms) more readily than vacancies, as it is energetically favourable for an interstitial to occupy a dislocation core.
than remain in the matrix. This ‘dislocation bias’ has been used in rate theory to rationalise the presence of excess concentrations of vacancies when equal numbers of vacancies and interstitials are produced homogeneously and the form of Frenkel pairs, e.g., by 1 MeV electrons in a high voltage electron microscopes. Excess vacancy concentrations from dislocation bias are considered to be the driving force for irradiation creep and swelling in rate theory calculations [9]. Application of dislocation bias alone may not be so appropriate for the cascade damage conditions that are occur with 14 MeV fusion reactor neutrons where vacancies and interstitials are produced in a highly localised and segregated fashion [37].

The consideration of interstitial clustering in the cascade yields to a ‘production bias’, which is a second driving force for void swelling in addition to the one provided by dislocation bias [37, 38, 39]. As discussed in Section 2.2.1.3, the vacancies and interstitials that escape recombination in the cascade are highly segregated, with the vacancies in the S-zone and the highly mobile interstitials injected into the surrounding matrix. Vacancies tend to cluster on <111> planes in the S-zone and collapse to form intrinsic faulted loops, and the highly mobile interstitials surrounding the cascade core readily form interstitial clusters on the <111> planes (extrinsic faulted loops). At elevated temperatures, the vacancy loops are thermally unstable because of the high line tension associated with a dislocation loop and so they tend to shrink by thermal vacancy emission. In contrast, the interstitial clusters are thermally stable even at high temperatures. The evaporation of the vacancies from vacancy loops and their escape from the S-zone provides the point defect ‘bias’ of mobile vacancies for microstructural evolution (void growth and irradiation creep) and macroscopic deformation (straining).

Katz and Wiedersich [40] and Russell [41] were among the first to analytically investigate the nucleation of voids. Void nucleation involves the growth of small vacancy clusters that can be stabilised by gaseous impurities such as helium produced from (n, α) reactions. Void swelling is dependent on the mobility and concentration of the vacancies and interstitials and hence the temperature and neutron fluence respectively. Exposure of austenitic stainless steels at 400-500°C to neutron fluences greater than 10^{22} n/cm^2 results in copious void formation and a degree of swelling (sometimes several percent) that is unacceptable in reactor materials. It is possible that voids are nucleated directly in the displacement spike, but more conventional nucleation
CHAPTER 2 - Background

is by the agglomeration of single vacancies [41]. Radiation-induced cavities can be classified as voids or bubbles depending on the driving force for their growth. The critical radius for a cavity is $r^*$. Cavities smaller than $r^*$ grow slowly as bubbles by the absorption of gas atoms of low solubility (usually helium) and vacancies. Above $r^*$, cavities grow rapidly and unstably as voids driven by the available vacancy supersaturation. In the absence of helium, oxygen can act as the void nucleation agent.

Extensive reactor studies on void swelling have been conducted in order to quantify the process of void swelling. It appears that the swelling rate can be described by an incubation period followed by an eventual steady state swelling rate [9]. The sensitivity of void swelling to parameters such as alloy composition and thermomechanical treatment appears only to express itself in the duration of the transient regime of swelling. The incubation period can be prolonged by the addition of certain alloying elements, for example, additions of titanium or titanium and phosphorous in 316 stainless steel (see Fig. 2.17). Radiation-induced precipitation will eventually result in the modifiers such as titanium being depleted and so the onset of swelling is merely delayed [33]. In many of the reactor studies conducted on austenitic stainless steels, the steady state swelling regime proved to be insensitive to most variables, with the steady swelling rate of most Fe-Cr-Ni austenitic alloys being $\approx 1\%/$dpa [42, 43].
Different levels of helium have the possibility to either help or hinder void swelling [9]. Low dose irradiation of solution annealed and cold worked Prime Candidate Application (PCA) austenitic stainless steel in FFTF/ORR/HFIR at 500°C originally suggested that void swelling is at its greatest at the He/dpa ratio close to that expected in a fusion first wall (10-12 appm He/dpa), as shown in Fig. 2.18. It was later shown, however, that the irradiation experiments in ORR were highly non-isothermal due to a frequent number of power reductions throughout the irradiation history [45]. It was proposed that the frequent intermittent reductions in temperature caused periodic reductions in temperature caused periodic and profuse production of small dislocation loops, known to be efficient nucleation sites for helium bubbles, which resulted in a
refined cavity microstructure. As a consequence of this, the relationship between helium and swelling is still not fully understood.

Figure 2.18 – Swelling as a function of He/dpa ratio for SA and CW PCA specimens irradiated in FFTF, ORR and HFIR, after Maziasz [12].

At a given displacement rate, there are three separate temperature regimes of steady state swelling as shown in Fig. 2.19. Generally voids form abundantly in 316 SS above 400°C. At low temperatures (≤ 0.3T_{mp} = ~350°C) vacancies have insufficient mobility to diffuse towards sinks where voids might nucleate. The higher mobility of interstitials at these temperatures increases the likelihood that recombination will occur, reducing the concentration of vacancies. Recently, however, voids have been seen to be present in Russian 304 type austenitic stainless steel components irradiated in a low-flux environment at temperatures approaching 300°C see Fig. 2.20. At higher temperatures (≥ 0.6T_{mp}), the void embryos emit more vacancies than they receive and the voids shrink.

Figure 2.19 – Schematic representation of the three regimes of steady state swelling rate in AISI 316 stainless steel, after Garner [9].

\[4 \text{ Acronyms: FFTF – Fast Flux Test Facility; ORR – Oak Ridge Reactor; HFIR – High Flux Isotope Reactor.}\]
In contrast to austenitic stainless steels, ferritic and ferritic/martensitic steels do not appear to swell at much lower rates than austenitic stainless steels. The difference in behaviour has been attributed to the more open b.c.c. structure in which diffusion is inherently higher than in f.c.c. structures, and the possibility of a lower dislocation bias in ferritic alloys.

2.3.3.5 - Irradiation creep

Creep is a time-dependent strain response to an applied stress that occurs at elevated temperatures at stresses below the yield strength of the material. Thermal creep can become a problem in most polycrystalline materials above \( \frac{1}{2}T_{mp} \) on the absolute temperature scale. It is generally considered that thermal creep has three stages [47]. Fig. 2.21 shows a typical creep test on a standard creep test specimen. The strain represented by \( \varepsilon_0 \) occurs instantaneously on the application of load. This is almost completely elastic strain, but may include some fraction of anelastic (unrecoverable) strain, depending on the material. The strains during the primary creep stage occur as dislocations glide under the applied stress until they reach an obstacle. The creep
resistance of the material increases by virtue of its own deformation, which increases the amount of dislocation pinning. Vacancy assisted climb may permit the dislocation to move around the obstacle allowing it to continue gliding until it reaches another obstacle. This is known as dislocation creep. Thermal creep can also occur by mechanisms that are related to vacancy diffusion. Diffusion creep includes Nabarro-Herring creep\(^5\) (at higher temperatures) and Coble creep\(^6\) (at lower temperatures).

After the primary creep stage, a steady state creep rate (secondary creep) is eventually attained where vacancy assisted climb controls the creep rate (Fig. 2.21) and under constant load conditions, tertiary creep is marked by an acceleration of the creep rate as internal voiding and necking occurs and the applied stress increases until failure occurs.

Irradiation creep in austenitic stainless steels results in orders of magnitude increase in the measured creep rate during relatively low temperature irradiation (See Fig. 2.22). Measured activation energies associated with irradiation creep show that it is a process related to interstitial migration \([9]\). The mechanisms of irradiation creep are strongly dependent on the formation and partitioning of both interstitials and vacancies to the various microstructural sinks, most importantly dislocations and cavities (if present). The formation of interstitials and vacancies via irradiation is an athermal process, and therefore irradiation creep is thought to occur in parallel with thermal creep. The total in-reactor creep rate would be equal to the sum of all contributing thermal and irradiation creep mechanisms.

![Typical creep curve showing the three steps of thermal creep](image)

**Figure 2.21** – Typical creep curve showing the three steps of thermal creep. Curve A, constant load test; curve B, constant-stress test, after Dieter [47].

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\(^5\) Nabarro-Herring creep – creep strains occur as a result of a vacancy flux from a grain boundary experiencing tensile stresses, through the matrix to a grain boundary experiencing a compressive stress.

\(^6\) Coble creep – related to vacancy diffusion along grain boundaries.
The irradiation creep rate is dependent on the stress level, the temperature and the flux of irradiation. Irradiation creep strains are the product of several processes, but the major contributions are thought to be stress induced preferred nucleation (SIPN) of dislocation loops; stress induced preferential absorption (SIPA) of point defects by dislocations; and yielding creep, which is a consequence of loop growth.

In SIPN, the preferential nucleation of Frank interstitial loops is biased on close packed atomic planes that are perpendicular to the applied stress. Fig. 2.23 shows a schematic of the process. The mass transfer associated with self-interstitials moving from the surrounding matrix to nucleate loops results in an elongation in the direction of the applied stress. The continued growth of the interstitial loops by stress induced preferential absorption (SIPA) of more interstitials leads to additional creep strains in the direction of the applied stress. Network dislocations will also undergo SIPA of interstitials with favourably aligned dislocations absorbing more than unfavourably aligned dislocations. Absorption of self-interstitials at edge dislocations with Burgers vectors parallel to the applied stress will contribute to creep strains. At low temperatures ($T \leq 0.3 \ T_m$), the loop density is large, but the point defect flux may be small because of their low mobility. At high temperatures ($T \geq 0.5T_m$), the loop density is small because of recovery and higher vacancy mobility. Somewhere between these limits lies the optimum conditions for irradiation creep to occur.

Figure 2.22 – Comparison of creep rates observed in 20% cold worked 316 stainless steel in uniaxial tests during thermal ageing or neutron irradiation in EBR-II, after Gilbert [48].
Irradiation creep can be divided into four distinct regimes [9]. The first regime exhibits an initial high creep rate that declines with increasing irradiation and is referred to as the transient primary regime. The transient primary regime quickly reaches a steady state secondary regime which continues in the absence of swelling (See Fig. 2.22). During the swelling-independent component of irradiation creep, the strain increases linearly with dose and is relatively insensitive to material variables and irradiation temperature, except at low temperatures where accelerated creep may occur as result of low vacancy mobility [35]. The third regime marks the onset of void swelling, which enhances the creep rate and the final regime marks the disappearance of irradiation creep.

The instantaneous irradiation creep rate, $B$, is measured as the strain-rate per unit stress and dpa and can be written in the form:

$$B = \frac{\dot{\varepsilon}}{\dot{\sigma}} = B_0 + D \dot{S}$$

(2.2)

where $B_0$ is the creep compliance and $D$ is the creep-swelling coupling coefficient and $\dot{S}$ is the instantaneous volumetric swelling rate per dpa.
Irradiation creep and void swelling can be determined from pressurised tube data. The Soderburg relationship [49] states that the length change of a pressurised capsule will arise only from dilatational strains (void swelling) and densification from phase changes but not from creep.

2.3.3.6 – Radiation hardening and severe embrittlement
Austenitic stainless steels undergo significant increases in strength with an attendant loss of ductility for irradiation and test temperatures in the range 60-400°C (see Fig. 2.24) [50]. An increase in yield stress of ~20-40% can occur in reactor pressure vessel steels after irradiation at ~240-290°C to neutron fluence < 10^{24} n m^{-2} [51]. Yield strength and the increase in yield strength of austenitic stainless steels are both maximum after irradiation at ~300°C [35, 52]. The rate and degree of radiation embrittlement in reactor pressure vessel steels can limit the lifetime of a pressure vessel and hence a reactor. As the dose increases, the tensile stress strain curves begin to exhibit a load drop after yield followed by a deformation plateau that transitions from work hardening to perfectly plastic to work softening behaviour. The uniform elongation (defined by the onset of plastic instability) stays relatively high out to a dose of about 7 dpa for all temperatures of interest; but at temperatures in the range 250-350°C, doses beyond 7 dpa appear to result in severe embrittlement, i.e. a very low uniform elongation. Severe ductility-loss or embrittlement has significant implications with respect to loss of fracture toughness.

Figure 2.24 – Engineering stress-strain data for annealed 316(N)-SPN stainless steel irradiated and tested at 250-270°C in HFIR, HFR and R2, after Lucas [50].
3.0 – Experimental procedures and background

The objective of this work has been to demonstrate the validity of the shear punch test technique for generating engineering-relevant mechanical property data for irradiated materials from transmission electron microscope (TEM) specimens. This was achieved by constructing simple correlations between data from the shear punch test and miniature tensile test data for a wide range of irradiation-evolved and thermomechanically-induced microstructures. It is also the aim of this work to combine the results from mechanical properties testing, microstructural information and finite element analysis to provide significant input on the use and limitations of the shear punch test and the associated correlations. The following sections detail the choice of materials tested, the experimental procedures employed, and details of the correlations that were developed.

3.1 – Methodology

Proving the shear punch-tensile correlation technique begins with the demonstration of the validity of the mechanical properties data recorded from miniature tensile specimens. It is important that the miniature tensile data be shown to be valid before correlating it with shear punch test data. While good agreement of data obtained from miniature tensile specimens with full-size specimen data has been reported elsewhere [53, 54] confirmation of this result has also been shown in this work. During the course of this work ASM full-size and miniature tensile specimens fabricated from 5182-O aluminium (nominally Al-4.5Mg-0.35Mn) were available for testing and the results are presented to demonstrate the agreement between the data from the two specimen sizes.

The shear punch test technique, in its current form, had been developed previously using almost entirely unirradiated materials [55, 56 and 57]. The first major task of this work was to establish a concise set of working procedures for punch testing highly irradiated TEM disks in an open facility, i.e., to be able to work on a bench top rather than by remote control in a shielded hot cell\(^1\). The techniques were developed using

\(^1\) Hot cell – a fully enclosed and lead shielded working area used for handling and testing radioactive materials. The hot cell protects the worker from ionising radiation and helps to prevent radioactive material contamination. Work is conducted using special manipulator arms and grips that are inside the cell and operated by remote control. A thick lead-glass window or in some cases camera surveillance enables the operator to conduct the work.
unirradiated control specimens, which were also used to evaluate the effects of various geometrical parameters associated with the shear punch test.

The success of the techniques developed was illustrated with the testing of a set of highly radioactive ferritic stainless steel specimens irradiated in the HFIR\(^2\). Tensile data were not available, but the results from the punch test indicated trends in the mechanical properties that would not have been otherwise available.

Empirical correlations that relate tensile yield strength, ultimate strength, uniform elongation and strain hardening coefficient to shear punch test data were developed for austenitic stainless steels having a variety of irradiation-evolved and thermomechanically-induced microstructures that were irradiated in the FFTF-MOTA\(^3\). These correlations cover a wide range of material strengths and conditions, compiled in a single set of correlations for the first time. The correlations developed were applied to predict some of the tensile properties of austenitic stainless steels irradiated as components in commercial light water reactors for which no tensile specimens were available. Microstructural data were used to back up some of the findings of the mechanical testing and insight into the origin and nature of the slope and offset of the shear punch-tensile correlation for yield strength was obtained through the use of a finite element model simulation of the shear punch test.

\(^2\) HFIR is the High Flux Isotope Reactor in Oak Ridge, Tennessee, USA (see Section 3.3.1).
\(^3\) FFTF-MOTA is the Fast Flux Test Facility Materials Open Test Facility in Richland, Washington, USA (see section 3.3.2).
3.2 – Alloy compositions and backgrounds

3.2.1 – Ferritic alloys irradiated in the High Flux Isotope Reactor

TEM-sized specimens of a set of isotopically tailored ferritic alloys\(^4\) irradiated in the High Flux Isotope Reactor (HFIR), Tennessee, USA were available for shear punch testing. The specimens were from a simple, one variable experiment that had been devised in order to study the effects of different helium levels on the microstructures that evolved in a set of isotopically tailored ferritic alloys [58, 59].

Ferritic stainless steels are attractive as a potential fusion reactor material as, unlike austenitic stainless steels, they are highly swelling resistant [60]. However, it has been suggested by Kluch and co-workers [61, 62, 63] that the presence of ~100 appm helium in ferritic / martensitic stainless steels irradiated at 300 – 400°C causes a shift in the ductile-brittle transition temperature (DBTT) of 200-300°C. These findings were based upon experiments where \(^{58}\)Ni (the most common isotope in naturally nickel) was added to 9Cr-Mo-V-Nb and 12Cr-1Mo-V-W alloys to generate helium at fusion-relevant levels by a two-stage nuclear reaction. In the first stage, \(^{59}\)Ni is formed from \(^{58}\)Ni after a nuclear reaction that involves the capture of a neutron by the nucleus and in the second stage, under continued neutron irradiation, \(^{59}\)Ni subsequently undergoes an (n, \(\alpha\)) reaction to form helium. The results showed a shift in the DBTT and a reduction in the upper shelf energy (USE) in Charpy V-notch specimen tests that were proportional to the helium level. Gelles commented in a letter to the editor [64] that the observed increase in the DBTT observed by Klueh [61] was more likely to be due to irradiation-induced precipitates of nickel that had been observed in HT9 irradiated at 400°C [65] and that further experiments should be carried out to resolve the role of helium in ferritic steels.

The isotopically tailored ferritic experiment was commissioned to clarify the role of helium and nickel content on the mechanical properties of ferritic/martensitic steels. The specimens in this study were nominally of composition Fe-12Cr-1.5Ni. The rate of helium evolution was varied from 0.3 to 10.7 appm/dpa by altering the isotopic content of the nickel. This was done without changing the neutron spectrum or the atomic

\(^4\) Isotopically tailored ferritic alloy – the isotope balance of nickel was changed to favour a nuclear reaction with an isotope of nickel (\(^{59}\)Ni) that produced helium.
displacement rate, which might otherwise mask any effects of helium production on the microstructure and physical properties. The same composition and initial microstructure were maintained for the nickel containing alloys. This idea had been tested and proven in a previous series of FFTF irradiations on Fe-Cr-Ni austenitic alloys [66]. Table 3.1 shows the alloy compositions and isotopic content of nickel in the ferritic alloys.

<table>
<thead>
<tr>
<th>Alloy composition</th>
<th>Nickel content</th>
<th>Nickel isotopes present</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fe-12Cr-1.5(^{60})Ni</td>
<td>(^{60})Ni</td>
<td>(^{60})Ni, by isotopic separation from (^{Nat})Ni</td>
</tr>
<tr>
<td>Fe-12Cr-1.5(^{Nat})Ni</td>
<td>Natural nickel</td>
<td>(^{Nat})Ni (^{a})</td>
</tr>
<tr>
<td>Fe-12Cr-1.5(^{59})Ni</td>
<td>(^{59})Ni</td>
<td>(^{Nat})Ni with 2.3% (^{56})Ni, enriched by neutron irradiation</td>
</tr>
<tr>
<td>Fe-12Cr</td>
<td>None</td>
<td>-</td>
</tr>
</tbody>
</table>
|\(^{a}\) \(^{Nat}\)Ni contains \(67.76\%\) \(^{58}\)Ni, \(26.16\%\) \(^{56}\)Ni, \(1.25\%\) \(^{61}\)Ni, \(3.66\%\) \(^{62}\)Ni, 1.16\% \(^{64}\)Ni [4].

The first alloy contained \(^{60}\)Ni, which was obtained by isotopic separation. In the absence of \(^{59}\)Ni and with significantly reduced \(^{58}\)Ni content, this isotopic variation produces very little helium (< 1 appm He/dpa). The second alloy contained natural nickel, which produces an intermediate level of helium (~45 appm He/dpa) after the delayed development of \(^{59}\)Ni. The nickel content of the third alloy was enriched to 2.3% \(^{59}\)Ni to form relatively large amounts of helium (~10 appm He/dpa in this case). The fourth alloy, which was included as a control to clarify the role of nickel on the properties of these alloys, contained no nickel.

### 3.2.2 \(^{59}\)Ni doped austenitic alloys irradiated in the FFTF-MOTA

This group of materials has been referred to in past experiments as the \(^{59}\)Ni doping series [67]. The \(^{59}\)Ni isotopic tailoring experiment in the FFTF-MOTA used an isotopic tailoring approach to evaluate the effect of helium generation rates typical of both fast reactor and fusion reactor environments on the tensile properties of the three model austenitic alloys. Table 3.2 shows the alloy matrix of the \(^{59}\)Ni experiment. As with the isotopically tailored ferritic alloys in the previous section, helium generation rates relevant to a fusion reactor were produced by an \((n, \alpha)\) reaction involving \(^{59}\)Ni, an isotope which is not found in natural nickel. Nickel enriched in the \(^{59}\)Ni isotope was
extracted from Inconel 600 fracture toughness specimens, which were originally irradiated in the Engineering Test Reactor (ETR). The enriched nickel contained 2% of the $^{59}\text{Ni}$ isotope. Specimens with low and high helium generation rates were irradiated side by side with active temperature control to $\pm 5^\circ\text{C}$. In effect, a truly one-variable experiment was devised to study the impact of helium on the evolution of microstructure and mechanical properties.

<table>
<thead>
<tr>
<th>Alloy composition</th>
<th>Nickel isotopes present</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fe-15Cr-25Ni</td>
<td>$^{\text{Nat}}\text{Ni}^a$</td>
</tr>
<tr>
<td>Fe-15Cr-25$^{59}\text{Ni}$</td>
<td>$^{\text{Nat}}\text{Ni}$ with 2.0% $^{59}\text{Ni}$, enriched by neutron irradiation</td>
</tr>
<tr>
<td>Fe-15Cr-25Ni-0.04P</td>
<td>$^{\text{Nat}}\text{Ni}^a$</td>
</tr>
<tr>
<td>Fe-15Cr-25$^{59}\text{Ni}$ -0.04P</td>
<td>$^{\text{Nat}}\text{Ni}$ with 2.0% $^{59}\text{Ni}$, enriched by neutron irradiation</td>
</tr>
<tr>
<td>Fe-15Cr-45Ni</td>
<td>$^{\text{Nat}}\text{Ni}^a$</td>
</tr>
<tr>
<td>Fe-15Cr-45$^{59}\text{Ni}$</td>
<td>$^{\text{Nat}}\text{Ni}$ with 2.0% $^{59}\text{Ni}$, enriched by neutron irradiation</td>
</tr>
</tbody>
</table>

$^a$ – $^{\text{Nat}}\text{Ni}$ contains 67.76% $^{58}\text{Ni}$, 26.16% $^{59}\text{Ni}$, 1.25% $^{60}\text{Ni}$, 3.66% $^{61}\text{Ni}$, 1.16% $^{62}\text{Ni}$ [4].

The three alloys were prepared in 50-gram buttons, which were normalised at 1250°C for 2 hours in an argon atmosphere. This was followed by a series of cold rolls and thirty-minute anneals at 1030°C in argon until the alloys were given a final 20% reduction in thickness to 0.25 mm (0.01 in.). TEM and miniature tensile specimens were punched from the sheet stock, deburred and then engraved with identifying codes. The solution annealed specimens were heat treated at 1030°C for 30 minutes after they were deburred and engraved [67].

Previous papers in the $^{59}\text{Ni}$ series have addressed the influence of helium on radiation-induced evolution of microstructure and void swelling using microscopy disks, and the evolution of mechanical properties using miniature tensile specimens [66, 68, 69, 70, 71]. Garner et al. [66] reported the tensile results for the $^{59}\text{Ni}$ series in a previous experiment. The results showed in general that all the $^{59}\text{Ni}$ alloys approached saturation levels of strength and ductility that were independent of He/dpa ratio and starting condition, but that were sensitive to the irradiation temperature and dpa rate.
3.2.3 – Three variations of 316 stainless steel irradiated in the FFTF-MOTA

Three variants of a 20% CW 316 stainless steel were available for shear punch and tensile testing. The first alloy, designated CN13, was one of the well controlled heats used to construct first core of the Fast Flux Test Facility (FFTF), and A1 and A61 were experimental heats with slightly different levels of minor alloying elements (C, P, Ti and Si), as shown in Table 3.3.

Table 3.3 – Compositions of three 316 austenitic type austenitic stainless steel alloys, form PNNL archive logbooks.

<table>
<thead>
<tr>
<th>Element</th>
<th>CN13</th>
<th>A1</th>
<th>A61</th>
</tr>
</thead>
<tbody>
<tr>
<td>C</td>
<td>0.04</td>
<td>0.05</td>
<td>0.02</td>
</tr>
<tr>
<td>Cr</td>
<td>15.26</td>
<td>15.73</td>
<td>15.75</td>
</tr>
<tr>
<td>Ni</td>
<td>12.48</td>
<td>13.77</td>
<td>13.60</td>
</tr>
<tr>
<td>Mo</td>
<td>2.05</td>
<td>2.45</td>
<td>2.49</td>
</tr>
<tr>
<td>P</td>
<td>0.020</td>
<td>0.016</td>
<td>0.017</td>
</tr>
<tr>
<td>Si</td>
<td>0.69</td>
<td>0.62</td>
<td>0.38</td>
</tr>
<tr>
<td>Ti</td>
<td>0.002</td>
<td>0.012</td>
<td>0.010</td>
</tr>
<tr>
<td>Mn</td>
<td>1.38</td>
<td>1.99</td>
<td>1.77</td>
</tr>
<tr>
<td>S</td>
<td>0.024</td>
<td>0.018</td>
<td>0.019</td>
</tr>
<tr>
<td>Cu</td>
<td>0.02</td>
<td>0.02</td>
<td>0.02</td>
</tr>
<tr>
<td>Co</td>
<td>0.01</td>
<td>0.07</td>
<td>0.07</td>
</tr>
<tr>
<td>Al</td>
<td>0.023</td>
<td>0.032</td>
<td>0.034</td>
</tr>
<tr>
<td>Fe</td>
<td>Base</td>
<td>Base</td>
<td>Base</td>
</tr>
<tr>
<td>Nb</td>
<td>0.01</td>
<td>0.02</td>
<td>0.02</td>
</tr>
<tr>
<td>Ta</td>
<td>0.01</td>
<td>0.01</td>
<td>0.01</td>
</tr>
</tbody>
</table>
3.2.4 – Four cold worked levels of a commercial 316 stainless steel

TEM disks and matching miniature tensile specimens were available for an unirradiated, commercial austenitic stainless steel (exact composition not available) in four thermomechanical conditions (solution annealed and 25, 50 and 75 percent cold worked). The cold worked conditions were achieved through a series of cold rolls to achieve the desired thickness and the specimens were then fabricated from the 0.25 mm thick sections of the material by electrical discharge machining. These specimens, together with the three 316 stainless steel variations that were irradiated in the FFTF-MOTA were used to establish whether the same shear punch-tensile correlation for strength and ductility could be used for similar materials in the irradiated and unirradiated conditions.

3.2.5 – BWR-irradiated stainless steels for tensile property predictions

Irradiated TEM disks were available for two 304 stainless steel heats and two 316 stainless steels heats from commercial boiling water reactor components. An IASCC research group required strength and ductility properties for the materials, but had insufficient irradiated material to produce miniature tensile specimens. Throughout this work, the 304 stainless heats are designated L and M and the 316 stainless steel heats were designated N and O. The funding client did not disclose the exact compositions of these materials. Miniature tensile specimens and TEM disks were available for some of the heats in the unirradiated condition for proof testing the correlation, but the tensile properties of the irradiated heats were to be evaluated from shear punch tests using the shear punch correlation developed during this study.

IASCC – Irradiation Assisted Stress Corrosion Cracking (Section 3.3.3).
3.3 – Irradiation facilities, details and sequences

Miniature tensile and TEM specimens from the $^{59}$Ni experiment and 316 variations experiment were irradiated in the Fast Flux Test Facility’s Materials Open Test Facility (FFT-MOTA) in Richland, Washington, USA. The isotopically tailored ferritic alloys were irradiated in the high flux isotope reactor (HFIR) at Oak Ridge, Tennessee, USA in the HFIR-MFE-JP23 capsule and the 304 and 316 stainless steel heats (designated L, M, N and O for this work) were irradiated as components in an unspecified BWR.

3.3.1 – Irradiation facility and details for isotopically tailored ferritic steels

The isotopically tailored ferritic alloys were irradiated in the high flux isotope reactor (HFIR) at Oak Ridge, Tennessee, USA in the HFIR-MFE-JP23 capsule. The HFIR is a 100 MW, beryllium reflected, pressurised light-water cooled reactor that was designed for the production of isotopes [72]. The target region is situated between two concentric annular fuel elements (see Fig. 3.1). This core region experiences a high power density that leads to a high thermal neutron flux. Irradiation capsules for experiments can be placed in the target region or positions in the surrounding reflector.

Figure 3.1 – Plan view of the high flux isotope reactor (HFIR) showing reactor components, fuel and experiment access, after Hicks [72].
Specimens were irradiated at 300, 400, 500 and 600°C in the HFIR-MFE-JP23 capsule for a period of 110.2 effective full power days. The resulting fluence levels, dose levels and helium production levels corrected for positions in the reactor are shown in Table 3.4 [59]. Specimens were also irradiated to 20, 40 and 70 dpa, but none as yet were available for shear punch testing.

Table 3.4 – Dose level and helium production levels for isotopically tailored ferritic stainless steels irradiated in the HFIR-MFE-JP23 capsule, after Gelles [59].

<table>
<thead>
<tr>
<th>Alloy composition</th>
<th>Irradiation temperature, fluence, dose level and helium production level</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>300°C 3.45×10^{22} ncm^{-2}</td>
</tr>
<tr>
<td></td>
<td>Dpa appm He</td>
</tr>
<tr>
<td>Fe-12Cr</td>
<td>6.5 2.1</td>
</tr>
<tr>
<td>Fe-12Cr-1.5^{60}Ni</td>
<td>6.4 2.1</td>
</tr>
<tr>
<td>Fe-12Cr-1.5^{59}Ni</td>
<td>6.5 41</td>
</tr>
<tr>
<td>Fe-12Cr-1.5^{59}Ni</td>
<td>6.6 70</td>
</tr>
</tbody>
</table>

3.3.2 – Irradiation facility for {59}Ni materials and 316 stainless steel variations

The Fast Flux Test Facility (FFTF) is a liquid sodium-cooled fast breeder reactor (see section 3.1.2). The FFTF was designed to accommodate experimental test assemblies in the core of the reactor. The Materials Open Test Assembly (MOTA) was one of the test assemblies that were used to provide a controlled irradiation environment for materials testing experiments. The assembly is a complex structure that houses temperature-controlled canisters arranged in sets of six around a central column at eight levels through the reactor (Fig. 3.2). Specimens were contained in perforated packets, such that they would be in contact with the liquid sodium coolant. The packets fitted into baskets within the cylindrical canisters. Gamma heating, proportional to atomic mass, is the source of heating within the specimens, coolant and core components. The liquid sodium coolant removes the heat from the core and test assemblies. The temperature of each canister is controlled by three mechanisms: the inlet temperature of the liquid
sodium coolant, its flow rate through each canister and by a helium/argon gas gap that surrounds each canister. The radial heat transfer through the gas gap can be varied by tailoring the thickness of the gap and the thermal conductivity of the gas gap can be 'fine tuned' between that of helium and that of argon by control of the gas composition. By these methods, active temperature control of each canister could be maintained at temperatures between 365 and 750°C to within ±5°C.

The neutron flux varies across the core, but at the core centre is around $3.0 \times 10^{15}$ neutrons/cm$^2$-sec (see Fig. 3.3). Each MOTA irradiation cycle ran a total of ~200 full power days over a period of about a year. Interim reconstitution with new hardware was necessary to ensure the canisters remained operational [73]. At the end of each irradiation cycle, specimens could be removed for post irradiation examination or reinserted in the next test cycle.

In both the $^{59}$Ni experiment and the 316 stainless steel variations experiments, miniature tensile specimens were irradiated side-by-side with TEM disks (for microscopy) in the same packet in the FFTF-MOTA so that the two specimens experienced exactly the same irradiation and temperature history. In many experiments, TEM disks were irradiated in sufficient numbers to allow multiple types of testing to be performed. In

---

Figure 3.2 – MOTA test-train (30 in-core canisters shown) and capsule configuration, after Bauer [73].
the case of the $^{59}$Ni experiment, it was fortuitous for correlation development that redundant TEM specimens were available for shear punch testing. Most of the specimens had previously been used for microscopy.

![Figure 3.3 - Representation of the axial fast flux distribution in the Fast Flux Test Facility, after Bauer [73].](image)

3.3.2.1 - Irradiation details for isotopically tailored $^{59}$Ni series materials

Transmission electron microscope (TEM) disks of the three Fe-Ni-Cr alloys were in general irradiated side-by-side with the miniature tensile specimens as part of the $^{59}$Ni experiment. When the specimens were irradiated at various levels in FFTF, the helium to dpa (displacements per atom) ratios obtained were of the order of 0.3-0.5 appm He/dpa for the undoped alloys and 4.4-62 appm He/dpa for the $^{59}$Ni-doped alloys.

Due to some limitations on specimen availability, only three of the five original irradiation temperatures could be examined in the current study (365, 490 and 495°C). TEM disks were available for 20% CW and SA starting conditions for all three irradiation temperatures. At 495°C, two different irradiation sequences were available: one three-increment sequence that was completely isothermal, and a second, three-increment sequence in which the first irradiation increment was initially isothermal, then subject to a short over-temperature event, followed by prolonged irradiation at temperatures below 495°C. In the following two increments, the irradiation was again isothermal at 495°C. A comparison of the tensile behaviour of the two different irradiation sequences is shown in Fig. 3.4 for Fe-15Cr-25Ni. The high strength
observed in the specimens in the first increment of the non-isothermal sequence is a result of prolonged irradiation at temperatures below 495°C, which occurred after the overtemperature event in irradiation increment 1D (see Fig. 3.5). In the following irradiation increments, temperature control was maintained at 495°C and the material responded to reflect the higher temperature conditions and saturate at the same strength as the isothermal sequence. Similar behaviour was observed in the other two alloys.

At 365 and 490°C, specimens were available for most conditions that were previously included in the tensile experiment. These specimens did not experience the overtemperature event that the specimens irradiated at 495°C were subjected to as can be seen in Fig. 3.5.

Figure 3.4 – Two irradiation sequences conducted in FFTF-MOTA [66]. In one sequence (solid line), there was a very irregular temperature history in the first of three irradiation increments, while in the other sequence, all increments proceeded isothermally (dashed line).
**59Ni ISOTOPIC DOPING EXPERIMENT IN FFTF-MOTA**

<table>
<thead>
<tr>
<th>MOTA - ID</th>
<th>MOTA - IE</th>
<th>MOTA - IF</th>
<th>MOTA - IG</th>
</tr>
</thead>
<tbody>
<tr>
<td>Non-thermal</td>
<td>Isothermal</td>
<td>Isothermal</td>
<td>Isothermal</td>
</tr>
<tr>
<td>Below-Core</td>
<td>365°C</td>
<td>365°C</td>
<td>365°C</td>
</tr>
<tr>
<td>5.5 appm He/dpa without 59Ni</td>
<td>6.1 dpa*</td>
<td>10.2 dpa</td>
<td>24 dpa</td>
</tr>
<tr>
<td>Level 1</td>
<td>495°C</td>
<td>495°C</td>
<td>495°C</td>
</tr>
<tr>
<td>5.5 appm He/dpa without 59Ni</td>
<td>13.9 dpa</td>
<td>27.9 dpa</td>
<td>52 dpa</td>
</tr>
<tr>
<td>Level 1-repeat</td>
<td>495°C (R)</td>
<td>495°C (R)</td>
<td>495°C (R)</td>
</tr>
<tr>
<td>5.5 appm He/dpa without 59Ni</td>
<td>14.6 dpa</td>
<td>~29 dpa</td>
<td>~39 dpa</td>
</tr>
<tr>
<td>Level 6-repeat</td>
<td>490°C (R)</td>
<td>490°C (R)</td>
<td>490°C (R)</td>
</tr>
<tr>
<td>0.3 appm He/dpa without 59Ni</td>
<td>2.13 dpa</td>
<td>~4.8 dpa</td>
<td>~6 dpa</td>
</tr>
</tbody>
</table>

*Specimens unaffected by over-temperature event.

Figure 3.5 – Irradiation sequences for 59Ni experiment in FFTF-MOTA.

### 3.3.2.2 – Irradiation details for 316 stainless steel variations

The three different heats (designated Al, A61, CN13) of 20% cold-worked 316 stainless steel were irradiated in Fast Flux Test Facility (FFTF) at five temperatures between 400 and 730°C to doses ranging from 12 to 88 dpa. The miniature tensile and microscopy disks were irradiated side-by-side in perforated stainless steel subcapsules in contact with flowing sodium in the Materials Open Test Assembly (MOTA), with active temperature control within any one irradiation segment to ±5°C. The target temperature sometimes varied a little from irradiation segment to segment, however. Unirradiated control specimens were also available and the initial compositions of the alloys are shown in Table 3.3.

### 3.3.3 – Irradiation details for stainless steel heats from BWR components

The two 304 stainless steel heats and two 316 stainless steel heats were irradiated as components in an unspecified boiling water reactor. The TEM disks were fabricated from components irradiated to dose levels between ~0.4 and 9 × 10²¹ n/cm² (~0.5 to 13 dpa).
3.4 – Methods for measuring mechanical properties

Miniature specimens are desirable with respect to maintaining damage homogeneity in strong flux gradients and the possible effects of gamma heating on temperature gradients within the specimens, as well as from the viewpoint of handling radioactive material. Miniaturisation is also important from the standpoint of the effective use of irradiation sources and reactor space. In recent years, small specimen tests have been developed in response to the need for mechanical property data. Many of these have been periodically reviewed [74, 75, 76, 1, 77, 78] and several ASTM Special Technical Publications (STP) symposium have been held on small specimen test techniques. Some small specimen test techniques involve simply scaling down full size specimens, as is the case with miniature tensile testing, Charpy impact testing and compact tension testing [79, 80, 81, 82], but others, such as the shear punch and ball punch tests rely upon innovative ideas to extract useful mechanical property data.

At this point it is felt necessary by the author to point the differences between some of types of small punch tests that are currently in use and under investigation, since they are often confused. The bulge test, which is often referred to as the small-punch (SP) or ball-punch test has a similar test fixture and configuration to the shear punch test that is used in this work. Shear punch tests and small specimen bulge tests are both carried out on transmission electron microscope (TEM) disk specimens, but are used to extract quite different mechanical properties. The bulge test uses a hemispherical punch that is driven into the face of a TEM disk at a slow strain rate. It is claimed that a load versus punch displacement trace from the bulge test can be used to obtain mechanical properties data such as fracture toughness, $J_{1C}$, [83, 84 and 85] and the ductile to brittle transition temperature (DBTT) [86, 87, 88 and 89]. The shear punch test uses a flat-faced, circular cross-section punch that is driven at a slow strain rate through a TEM specimen in a blanking/cutting type operation.

In the following sections, the development of the miniature tensile test and the shear punch test technique will be reviewed in detail.
3.4.1 – Miniature tensile testing

The use of miniature tensile specimens is unique to the nuclear materials community for reasons as discussed in the previous section. Miniature tensile testing has been periodically reviewed [54, 74, 78, 53, 75, 1] and has been widely accepted in the nuclear materials testing community as a useful means of extracting valid mechanical properties data from irradiated specimens. The applicability of these small tensile specimens to irradiation damage studies was established in a variety of other experiments [53, 90]. Table 3.5 lists the dimensions of a number of scaled-down tensile specimens that have been used and Fig. 3.6 shows the manufacturing drawing for the ‘US – RTNS-II sheet’ specimen, which is the ‘miniature’ tensile type referred to and used throughout this study.

Table 3.5 – Compilation of miniature tensile specimen geometry, after Lucas [75].

<table>
<thead>
<tr>
<th>Type</th>
<th>Length (mm)</th>
<th>Width (mm)</th>
<th>Thickness (mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Full size ASM</td>
<td>76.2</td>
<td>12.7</td>
<td>2.4</td>
</tr>
<tr>
<td>SS-1</td>
<td>20.3</td>
<td>1.52</td>
<td>0.76</td>
</tr>
<tr>
<td>SS-3</td>
<td>9</td>
<td>1.52</td>
<td>0.76</td>
</tr>
<tr>
<td>US – RTNS-II sheet</td>
<td>5.10</td>
<td>1.03</td>
<td>0.254</td>
</tr>
<tr>
<td>‘minitensile’</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

As the gauge lengths of miniature tensile specimens are so small, extensometers are not a practical means of measuring the elongational properties of the specimens and so crosshead displacement is used [54]. Panayotou et al. [78] demonstrated that the miniature tensile specimens used in many size effects studies yielded representative strength and ductility data at room temperature for unirradiated 316 stainless steels, HT9 and age-hardenable Cu-Be-Ni materials. As long as a sufficient number of grains are maintained in the cross section of the tensile specimen, the miniaturised specimen strength data have been in fairly good agreement with the larger tensile specimen data. Studies have indicated that at least 25 grains in the cross section are required to represent bulk properties of a material, although good results can be produced with as few as 10 grains across the smallest dimension [91]. The agreement between small and standard size specimen elongation data is not as good, however.
As can be envisaged, miniature specimens are likely to be more sensitive to the fabrication method and final surface condition than the full size versions would be. Small surface flaws on miniature tensile specimens are likely to act as stress concentrators, to which full size specimens made by the same fabrication techniques may be insensitive. In a study by Hamilton [54], three different fabrication techniques were evaluated against each other for the manufacture of US - RTNS-II sheet miniature tensile specimens. They were punching, chemical etching and electric discharge machining (EDM). Miniature tensile specimens were originally developed with the intention that they would be punched from sheet stock that had been rolled to the desired specimen thickness. The problem with punching is that the operation produces unavoidable deformation in the specimen, especially when the clearance between the punch and die is not optimised for a given material or in softer materials, that results in specimens becoming bowed across the width of the gauge length and the formation of a burr around the perimeter of the punched specimen. It is, however, possible to generate reproducible results for the changes in tensile behaviour using punched specimens if the punch/die clearance is optimised for a given material to reduce deformation and the specimens are deburred after punching [54, 66, 92]. The two other fabrication techniques studied by Hamilton [54] were chemical milling (a selective etching technique) and electrical discharge machining (EDM). The EDM technique was shown
to produce the best surface finish of the three techniques since chemical milling resulted in some extent of undercutting occurring around the periphery of the specimen. EDM is now the preferred method of fabrication, although punching is useful for a quick turn around for material characterisation.

The miniature tensile specimens are held in wedge-type grips and tested on a screw-driven, horizontal test frame at a strain rate of $1 \times 10^{-4} \text{ s}^{-1}$. Load and displacement transducers measure the crosshead movement and load and a computer records the data, with a chart recorder used for backup. Fig. 3.7 illustrates the small scale of the specimens and the test frame used.

An example of the good agreement between the data from full sized and miniature specimen geometry will be shown in the ‘Experimental results’ section. In this study, miniature tensile specimens were EDM-fabricated from an aluminium sheet product fabricated from 5182-0 aluminium. The data obtained from miniature specimens were in good agreement with that from full sized specimens. Fig. 3.8 shows the two types of specimen side by side.

Figure 3.7 – Horizontal test frame and miniature US – RTNS-II sheet-type tensile specimens used at PNNL, after garner, [53].
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Figure 3.8 – Miniature (US-RTNS-II sheet) and full sized (ASM) specimens side by side. Both specimens shown were fabricated with a welded section in the gauge length to characterise the strength of the weld.

3.4.2 - Shear punch testing

The shear punch test technique was developed in response to the needs of the nuclear materials testing community for small-scale mechanical property tests. In the interests of developing an understanding of the test technique, a brief history of the shear punch test technique will be included as part of this experimental section. The shear punch test is essentially a blanking operation in which a flat-faced punch is driven at a constant rate through a sheet sample. The original experimental concept was to punch out Ø3mm disks from irradiated sheet to provide mechanical property data and then to prepare the punched disk for transition electron microscopy (TEM) analysis. This test sequence thus satisfies the requirements for the efficient use of irradiated materials. Preliminary investigations were conducted on several unirradiated materials (Cu, Al brass and stainless steels) with Ø6.25, Ø3 and Ø1 mm punches [74]. Data from the smallest punch was in good agreement with that from the largest and in subsequent experiments [55, 56, 93], a Ø1 mm punch was used on a Ø3 mm TEM specimen. The decision to adopt this configuration was made in anticipation that irradiated specimens would be tested in the future and reactor test assemblies already had facilities for
irradiating TEM disks. Other attractions of using TEM disks are that they are almost always produced in excess for irradiation experiments and their small size means that the dose limitations placed on upon testing radioactive specimens are rarely exceeded for out of cell testing. This is an important consideration when considering the resources required for setting up a tensile test in a lead-shielded hot cell.

In the current configuration of the shear punch test, a nominally 1 mm diameter punch is driven at a constant rate of 0.127 mm/min. (0.005 in./min.) through a transmission electron microscope (TEM) sized disk (nominally 0.25 mm thick and 3.0 mm in diameter). The disk is constrained along both its upper and lower surfaces in a test fixture, which also guides the punch (Fig. 3.9). The clearance between the punch and lower die as a fraction of the specimen thickness in the shear punch test is maintained at less than 10% (Fig. 3.9). This is the maximum clearance that was recommended by Chang [138] (on the subject of circular blanking operations) in order to obtain a clean fracture surface with least amount of strain work involved. A maximum clearance limit of 10% was therefore applied to the punch test so that the test specimen material did not deep draw into the die. In the current configuration of the shear punch test, the diameter of the punch that is used is chosen as a function of the specimen thickness in order that the clearance does not exceed 10% (see Appendix 1).

In early tests, the load on the punch was measured against the displacement of a cup or flat ended transducer that was held up against the lower side of the specimen. It was realised early on that crosshead displacement was a better measure of punch travel, as the underside of the specimen tended to bow and exaggerate the displacement measured by the transducer [94].

The progression of the punch test is followed in a series of partial punch tests on specimens made from solution annealed 304 stainless steel shown in Fig. 3.10. The test was stopped at various points on the loading curve (1 to 6) and the specimens were sectioned and polished to show the profile of the punched specimens.
The load versus displacement curve obtained from a shear punch test is of a similar form to that obtained from a tensile test (Fig. 3.10). After initial take-up, a linear relationship exists between load and punch displacement, during which no large-scale plastic deformation occurs (Fig. 3.10 # 1). Damage is limited to a faint indentation where the edge of the punch was pressed against the specimen causing a limited amount of local plastic deformation. A deviation from linearity (the effective shear yield strength) occurs in the loading curve at the point where the punch permanently penetrated the surface of the specimen (Fig 3.10 # 2). The yield load is taken at the point of deviation from linearity rather than at a 0.2% strain offset value, as is the case in a tensile test, since defining an offset strain would be inappropriate for the test geometry and nature of the shear punch test. Beyond the yield point, further deformation forms a shear process zone between the die and punch. Work hardening compensates for reduced ‘shear area’ until a maximum load is achieved [94] (Fig.3.10 # 4). Beyond the maximum shear strength, the remaining ligament cannot support the punch load and the solution annealed specimens typically fail when the remaining ligament is less than a half to a third of the original thickness. The effective shear yield
strength ($\tau_{sy}$) and an effective shear maximum strength ($\tau_{sm}$) can be evaluated from the yield and maximum loads, respectively, by the following equation [55]:

$$\tau_{sy,sm} = \frac{P_{sy, sm}}{(2\pi rt)}$$

In Eqn. 3.1, $P_{sy, sm}$ is the appropriate load, $r$ is the average of the bore and punch radii and $t$ is the specimen thickness.

Typically, for good quality EDM-fabricated specimens, $\tau_{sy}$ is reproducible with a standard deviation of 5-7 % and $\tau_{sm}$ is reproducible with a standard deviation of 2-3 %. The difficulty in determining the deviation from linearity or yield point for a shear punch test also contributes to lower the accuracy of $\tau_{sy}$. This is especially true for softer materials that do not exhibit extended linear elastic behaviour in the loading curve.
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1 - Localised yielding at the punch tip before yield
2 - Permanent punch penetration beyond yield
3 - Further beyond yield

Figure 3.10 - Development of punch test illustrated by pictures 1 - 6 of cross sections of partially punched, solution annealed 304 stainless steel specimens
3.4.3 – Shear punch testing facility for testing highly irradiated materials

As outlined in the methodology section, the first task of this project was to devise a facility and develop working procedures that would enable shear punch tests to be performed ‘out of cell’ on highly irradiated specimens. It is unusual to perform any kind of test on highly irradiated specimens without having to conduct the test in a lead-shielded hot cell by remote control. This is the case for miniature tensile testing and Charpy impact testing. However, TEM disks are small enough that, in some cases, even materials containing highly activated elements do not breach the limiting radiological conditions imposed by radiological control for conducting ‘bench top’ work.

To satisfy the Radiological Control requirements at PNNL, it was necessary to set up an appropriate testing facility and develop a safe operating procedure (SOP) and technical working document (TWD) for conducting the shear punch test on highly irradiated materials. The SOP is a meticulous set of instructions, detailing specifically the steps during operations that deal with handling and moving radioactive materials. The procedures were developed using unirradiated control specimens and then the techniques were further refined using the set of highly radioactive ferritic alloys described in Section 3.2.1. A copy of the latest revision of the SOP is contained in Appendix 1. The radiological control regulatory body at PNNL approved the procedures and issued a radiological work permit (RWP), which is used to establish radiological controls for entry into areas used for radiological purposes. Radiological work permits serve to inform workers of area radiological conditions, entry requirements into the areas, and provide a means to relate radiation doses received by workers to specific work activities. The more detailed technical working document (TWD) was written to deal with the specific procedures required for performing the shear punch test (see Appendix 2).
Fig. 3.11 shows the bench top loading area, where specimens are transferred from a lead shielded temporary storage area (not shown) to the shear punch test fixture prior to testing. The temporary storage area is essentially a lead brick cave designed to store a limited number of radioactive specimens. The area including the bench top and temporary storage area is designated as a radiation and contamination area.

The operating procedures require that whenever a specimen is handled, it be done by remote handling techniques, i.e., maintaining distance between the worker and the radiological source. Some of the ferritic specimens tested in this development stage measured individually 2000 mrem/hr at contact. The rem (Roentgen Equivalent Man) is a unit for measuring dose equivalence. It is the most commonly used unit for dose in the USA and pertains to the effect of radiation on the human body (1 rem = 10^{-2} Sv). However, the dose from TEM specimens that measured 2000 mrem/hr (20 Sv/hr) at contact reduces to < 100 mrem/hr (0.2 Sv/hr) at a distance of 30 cm because of the inverse square law relationship between dose and the distance from the source. The law states that if you double the distance from the source to the reference point, the dose rate falls to 1/4 of the original dose rate. If you triple the distance, the dose rate falls to 1/9 of the original dose rate.
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Specimens are removed from the temporary storage area using the long-handled tweezers (seen above the fixture holding device) and thickness measurements are taken using the dial micrometer that is situated behind a lead-glass block. Fig. 3.12 shows the fixture used for the shear punch test. The fixture is located in the holding device while it is loaded using long-handled tweezers. The fixture is then transferred to the test frame using one of the fixture handles (Fig. 3.11). A screw driven Instron test frame is used that is completely contained inside a fume hood. Fig. 3.13a shows the view of the test frame through the fume hood and Fig. 3.13b shows the test fixture in position. The test frame has a furnace for conducting tests at temperature, and also has the capability to conduct tests in an inert argon atmosphere. Data from the load cell is recorded as a function of time using Labview software, and a chart recorder is used as a backup data acquisition system.

As mentioned, the procedures and capability of the current shear punch test facility were first developed for, and qualified on, a set of isotopically tailored ferritic alloys. At the time, a recently completed study had reported on a simple, one-variable experiment that was devised in order to study the effects of different helium levels on the microstructures that evolved in a set of isotopically tailored ferritic alloys, nominally of composition Fe-12Cr-1.5Ni [59]. In the absence of available tensile specimens, the shear punch test was used to demonstrate qualitative trends in the mechanical properties of the ferritic alloys as a function of helium content.
Figure 3.12 - Picture of the test fixtures used for the shear punch test

Figure 3.13 - (a) Picture of the Instron test frame within its fume hood that is used for the shear punch test. (b) The test fixture in position (inside the furnace) viewed through the open window of the fume hood in which it is contained.
3.5 – Method for constructing shear punch – tensile strength correlation

A number of experiments have been carried out [94, 56, 55, 95, 57, 96, 97, 98] to develop a relationship between uniaxial tensile strength and effective shear strength, as determined using the shear punch test. The work has shown that effective shear yield and maximum strengths obtained by shear punch test methods can be linearly correlated to tensile yield and ultimate properties by a simple empirical relationship. When corresponding sets of $\tau$ and $\sigma$ are plotted, they fall along a straight line (Fig. 3.14). A linear regression is performed on the data to obtain the constants $m$ and $\tau_0$ in Eqn. 3.2:

$$\sigma_{y,UTS} = m(\tau_{sy,sm} - \tau_0)$$  \hspace{1cm} (3.2)

![Figure 3.14](image)

Figure 3.14 – Plot showing corresponding sets of $\tau_{sy}$ (shear punch) and $\sigma_y$ (miniature tensile) data plotted against each other. The data fall along a straight line with slope $m$ and x-axis intercept $\tau_0$.

The offset parameter $\tau_0$ (the x-axis intercept of the regression line) is used to indicate that the offset is thought to be some characteristic associated with the shear punch test and not the tensile test. $\tau_0$ has been ascribed in past to punch-die-specimen friction [74], with both $m$ and $\tau_0$ being somewhat material-dependent as can be seen in Table 3.6.

The correlations shown in the table are in terms of $m$ and $K$ (the y-axis intercept) rather than $m$ and $\tau_0$ (the x-axis intercept). Although the $\tau_0$ offset was not reported in the earlier studies, its equivalent value is shown for comparison. In each of these studies, it
was shown that different alloy sets could be combined to form a single correlation by choosing an appropriate value of \( \tau_0 \) for each alloy set. Fig. 3.15 shows the yield and ultimate shear punch-tensile correlation for the unirradiated aluminium alloys (5052 and 6061) in Table 3.6. Fig. 3.16 shows such a plot for the shear punch ultimate strength data from the unirradiated alloys in Fig. 3.15 together with irradiated aluminium shear punch data and full size torsion-tensile data from various other aluminium alloys.

Table 3.6 – Shear punch tensile correlation coefficients published prior to the current work by Lucas [98] and Hamilton et al. [55].

<table>
<thead>
<tr>
<th>Alloys</th>
<th>No. of data points</th>
<th>Test type</th>
<th>Punch size used</th>
<th>Yield strength correlation</th>
<th>Maximum strength correlation</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>m  K¹  ( \tau_0 )</td>
<td>m  K¹  ( \tau_0 )</td>
</tr>
<tr>
<td>Al, brass, Cu, stainless / carbon steels</td>
<td>&gt; 100</td>
<td>Punch</td>
<td>Ø6.25mm</td>
<td>1.85 -² -</td>
<td>1.61 -² -</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Ø3mm</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Ø1mm</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Various aluminium</td>
<td>126</td>
<td>Torsion</td>
<td>-</td>
<td></td>
<td>1.7 -8 4.7</td>
</tr>
<tr>
<td>Vanadium alloys</td>
<td>2³</td>
<td>Punch</td>
<td>Ø1mm</td>
<td>2.8 -129 46</td>
<td>1.8 -38 21</td>
</tr>
<tr>
<td>Stainless steels</td>
<td>10</td>
<td>Punch</td>
<td>Ø1mm</td>
<td>1.7 29 17</td>
<td>2.2 -425 193</td>
</tr>
<tr>
<td>5052 (O + T6) 6061 (O + T6)</td>
<td>8</td>
<td>Punch</td>
<td>Ø1mm</td>
<td>2.6 -73 28</td>
<td>2.1 -67 31.9</td>
</tr>
<tr>
<td>Cu-Zn</td>
<td>72</td>
<td>Torsion</td>
<td>-</td>
<td></td>
<td>2.9 -335 115</td>
</tr>
<tr>
<td>Copper alloys</td>
<td>3⁴</td>
<td>Punch</td>
<td>Ø1mm</td>
<td>-</td>
<td>-</td>
</tr>
</tbody>
</table>

¹K is the y-axis intercept of the effective shear stress-tensile strength correlation (K= - m\( \tau_0 \))
²K was considered as a frictional component, and was not reported
³Not enough data for a regression
⁴Insufficient data to fit a reasonable straight line
Lucas [56] noted that the regression coefficient in a tensile-shear punch correlation for yield data from a variety of materials when combined was close to $\sqrt{3} = 1.73$. This is the ratio of shear to uniaxial stress in the von Mises yield criterion i.e. $\sigma_y = \sqrt{3}\tau$. This result would only be expected if a state of pure shear can be assumed in the process zone of the specimen during a shear punch test (see Appendix 3 for a full derivation). However, the method by which the load is applied to a specimen during a shear punch test is not ideal for creating a state of pure shear. Kullen [99] previously applied the
Tresca yield criterion with some success to predict the tensile yield strengths of a number of materials after conducting a series of Ø3 mm punch tests to produce TEM discs. The work of these authors will be addressed in more detail in Section 4.3.

**3.6 – Method for constructing shear punch – tensile ductility correlation**

Tensile uniform elongation has also been predicted from shear punch properties [94, 93]. Toloczko et al. [93] showed that a linear correlation existed between tensile uniform elongation and data from the shear punch test for a variety of unirradiated alloys. The first step in the development showed that a linear relationship existed between the measured tensile strain-hardening exponent, \( n \) (from tensile test data), and a strain-hardening exponent, \( n_\sigma \), calculated from the ratio of ultimate tensile stress to tensile yield stress, \( \sigma_m/\sigma_y \), according to the following expression:

\[
\left( \frac{n_\sigma}{0.002} \right)^{n_\sigma} = \frac{\sigma_m}{\sigma_y}
\]

Eqn. 3.3 was derived by Lucas et al. [94] from the definition of power law strain hardening \((\sigma = K\varepsilon_{pl}^n)\) at yield and maximum strength, where the true plastic strain \(\varepsilon_{pl}\) equals 0.002 at yield, and \(\varepsilon_u\) equals \(n\) at the onset of necking or maximum strength (see Appendix 4). The measured strain-hardening exponent, \(n\), was determined from the slope of \(\sigma\) vs. \(\varepsilon_{pl}\) traces on log-log plots of the tensile data. Analysis of the tensile tests verified that \(\varepsilon_u \approx n\) for the materials tested. Fig. 3.17 shows the linear relationship between the measured tensile strain hardening coefficient, \(n\), and \(n_\sigma\) from Eqn. 3.3 that Toloczko obtained from tensile data on a variety of materials.
Figure 3.17 – A linear relationship is observed between the measured tensile strain hardening coefficient, \( n \), and a value of \( n_o \) calculated by Eqn. 3.3, after Toloczko et al [93]. Toloczko wrote that the two solution annealed aluminium alloy data points are thought to deviate from the linear relationship because of biaxial loading conditions brought about by a high ratio of gauge width to gauge thickness which is thought to promote early instability and necking.

In the next step, \( \sigma_m/\sigma_y \) in Eqn. 3.3 was replaced with the corresponding ratio of effective shear maximum stress to effective shear yield stress \( (\tau_{sm}/\tau_{sy}) \), resulting in \( n_r \), analogous to \( n_o \). A plot of \( n \) versus \( n_r \) was then constructed, which is shown in Fig. 3.18. When considering that \( \sigma_m \) and \( \sigma_y \) represent true stresses and \( \tau_{sm} \) and \( \tau_{sy} \) are more like engineering stresses, this step was quite an approximation, but nevertheless a linear relationship was also found to exist between \( n \) and \( n_r \) with a slope similar to that of the \( n \) vs. \( n_o \) correlation. The linear nature of these relationships \( (n_r \propto n \text{ and } \varepsilon_{ui} \approx n) \) makes it possible to obtain finally a linear relationship between true uniform tensile elongation, \( \varepsilon_{ui} \), and \( n_r \) which is shown in Fig. 3.19.
Figure 3.18 - A linear relationship is also observed between the measured tensile strain hardening coefficient, $n$, and a value of $n$, calculated from a equation similar to 3.3, involving the ratio $\tau_{\text{sm}}/\tau_{\gamma}$, in place of $\sigma_{\text{m}}/\sigma_{\gamma}$, after Toloczko et al [93].

Figure 3.19 - A linear relationship is observed between $n$, calculated from shear punch data and true uniform tensile elongation $\varepsilon_u$, after Toloczko et al [93].
3.7 – **Techniques employed for quantitative microstructural analysis**

Transmission electron microscopy was conducted using a JEOL JEM 1200 EX by Dr D.S. Gelles at the Pacific Northwest National Laboratory on selected irradiated CN13 specimens (from the 300 series alloys) to observe the microstructural development of the materials irradiated at 430°C and 550°C up to 88 dpa. In the absence of any TEM disks remaining from the 300 series experiment, thin foils of three of irradiated specimens were prepared from the end tabs of un-tested miniature tensile specimens. The end tabs were punched using a Ø3 mm punch, and then manually thinned to approximately 0.15 mm on an abrasive surface within a shielded fume hood. The specimens were electropolished to produce thin foils for transmission electron microscopy. At least one foil was produced for each material condition.

Stereo pair pictures were taken to facilitate the evaluation of the number density of voids and faulted loops and the network dislocation line density so that a barrier hardness calculation could be performed by the method described in Section 3.8.

3.7.1 – **Measurement of network dislocation density**

Stereo pair pictures were taken in dark field contrast for g = 111, 200 and 020 imaging conditions for selected regions in each specimen foil. A region of the picture was chosen for network line density count and the thickness was measured using a stereo viewer. The distance between the top and bottom of the foil was repeatedly measured across the area of interest and a thickness measurement was calculated with knowledge of the magnification of the images and the angle between the stereo pair. The number of dislocations (including perfect loops) intersecting the surface of a box grid was counted and with knowledge of the foil thickness, the network dislocation line density was calculated. Repeated measurements were made in different regions of the foil to obtain the average network dislocation line density for each specimen. With g = 111 imaging conditions, half the network dislocations are visible and with g = 020 and 200 imaging conditions, two thirds of the network dislocation density [100].
3.7.2 – Measurement of cavity and faulted loop densities

Stereo pair pictures were also taken in bright field contrast for the same regions with $g = 111, 200$ and $020$ imaging conditions to image the voids and faulted loops in each specimen foil. The thickness of the region of interest was measured in the same way as for the network dislocations. Voids shapes in face centred cubic materials can vary between truncated octahedra and truncated cubes; to measure the correct void volume, an averaging calculation is performed and so the voids were all measured across the $<110>$ direction. A computer program developed by Gelles et al [101] was used to measure the actual void volume and average void diameter. A separate and similar method was used to measure the faulted loop average diameter and number density.

3.8 – Method applied to dispersed barrier hardening calculation

Where sufficient microstructural information is available, it is possible to estimate the yield stress of a material from a microstructure-yield stress relationship or dispersed barrier hardening calculation [102, 103, 104, 35 and 57]. Barrier hardening calculations were performed for selected material conditions from the $^{59}$Ni series and the 316 stainless steel variations to clarify the results of some of the mechanical testing.

As discussed in Section 3.3, microstructural features that serve as obstacles to dislocation slip control the yield strength of a material. The different types of obstacle present in the microstructure are divided into those that are short range, such as cavities, precipitates, faulted loops and grain boundaries, and those that are long range, such as network dislocations. Range refers to the distance from the obstacle at which it can affect the movement of a dislocation. Examples of long-range forces are those due to the interaction of moving network dislocations with each other.

The yield stress of the material is measured as the sum of the intrinsic yield strength, $\sigma_{y,int}$ of the solid solution of the matrix (in this case austenite), the root mean square of the critical resolved tensile stresses of the short range obstacles, $\sigma_{sr,i}$, and the sum of the critical resolved tensile stresses of the long range barriers, $\sigma_{lr,j}$. The superposition law is expressed by Eqn. 3.4 [57].

$$\sigma_y = \sigma_{y,int} + \sqrt{\sum_i \sigma_{sr,i}^2} + \sum_j \sigma_{lr,j}$$  (3.4)
It is generally assumed that the sum of the intrinsic yield strength, \( \sigma_{y,\text{int}} \), of the matrix and the critical resolved tensile stress of the grain boundaries, \( \sigma_{y,gb} \), is equal to the yield strength of the material in the solution annealed condition \([102]\) and it is also assumed that \( \sigma_{y,\text{int}} \) does not change during irradiation \([103]\).

The change in yield strength caused by each type of barrier is measured as the increase in applied stress required to move a dislocation through a field of obstacles of strength \( \alpha \) and inter-obstacle spacing \( l \), such that

\[
\Delta \sigma_y = \frac{m \alpha G b}{l}
\]  

(3.5)

where \( G \) is the shear modulus of the matrix, \( b \) is the Burgers vector of the moving dislocation, \( m \) is a factor relating the shear stresses on a slip plane in a single crystal to the applied tensile stress (3.1 for a f.c.c. material) and \( l \) is the inter-obstacle distance on the glide plane based on a square array of obstacles. It was shown by Foreman and Makin \([105]\) that for random arrays of obstacles, the effective stress required to move dislocations through these obstacle fields is about 80% of that assumed for a periodic (square) array and so \( f \) is a random array efficiency factor that is equal to \( \sim 0.8 \) (for perfectly hard obstacles).

Studies to date \([102, 103, 57]\) have assumed that for discreet obstacles, the value of \( l \) is equal to \( (n d)^\gamma \), where \( n \) is the number density of the obstacles and \( d \) is their average diameter. In the case of faulted loops, \( l \) is further adjusted by a factor of 0.56 to relate the average height of loops when intersecting glide planes in a face centred cubic material \([96]\). In the case of network dislocations, the spacing \( l \) is proportional to \( \rho^{\frac{1}{2}} \), with the constant of proportionality being equal to the fraction of network dislocations intersecting glide planes (in a face centred cubic material this is equal to \( 2/\pi \times l \)).

The obstacle strength, \( \alpha \), is a function of the critical bowing angle \( \theta_c \) above which a dislocation is released. Table 3.7, compiled by Lucas \([35]\) shows different obstacle strengths that have been assumed or derived from fits to experimental data. It is generally assumed that voids and large precipitates act like Orowan barriers, with \( \alpha \) equalling one; Frank faulted loops have intermediate barrier strengths and network
dislocations have low barrier strengths. In his Masters thesis, Toloczko [96] showed that the random array factor scales with alpha as shown in Table 3.8 and graphically in Fig. 3.20.

Table 3.7 – Comparison of different obstacle strength, α, for use in barrier hardness calculations, compiled by Lucas [35].

<table>
<thead>
<tr>
<th>Relative strength</th>
<th>Barrier Type</th>
<th>System</th>
<th>α</th>
<th>Reference</th>
</tr>
</thead>
<tbody>
<tr>
<td>Strong</td>
<td>Orowan</td>
<td>-</td>
<td>1</td>
<td>-</td>
</tr>
<tr>
<td></td>
<td>Voids</td>
<td>Austenitic</td>
<td>~1</td>
<td>[106]</td>
</tr>
<tr>
<td></td>
<td>Voids</td>
<td>Nickel</td>
<td>~1</td>
<td>[107]</td>
</tr>
<tr>
<td></td>
<td>Voids</td>
<td>Austenitic</td>
<td>~1</td>
<td>[102]</td>
</tr>
<tr>
<td></td>
<td>Voids</td>
<td>Austenitic</td>
<td>~1</td>
<td>[103]</td>
</tr>
<tr>
<td></td>
<td>Large precipitates</td>
<td>Austenitic</td>
<td>~1</td>
<td>[103]</td>
</tr>
<tr>
<td>Intermediate</td>
<td>Frank loops</td>
<td>Austenitic</td>
<td>0.33</td>
<td>[102]</td>
</tr>
<tr>
<td></td>
<td>Frank loops</td>
<td>Austenitic</td>
<td>0.45</td>
<td>[104]</td>
</tr>
<tr>
<td></td>
<td>Frank loops</td>
<td>Austenitic</td>
<td>0.45</td>
<td>[103]</td>
</tr>
<tr>
<td></td>
<td>Small MC ppts.</td>
<td>Austenitic</td>
<td>0.33-0.45</td>
<td>[104]</td>
</tr>
<tr>
<td>Weak</td>
<td>Small bubbles</td>
<td>Austenitic</td>
<td>0.2</td>
<td>[102]</td>
</tr>
<tr>
<td></td>
<td>Small loops/clusters</td>
<td>Austenitic</td>
<td>0.2</td>
<td>[108]</td>
</tr>
<tr>
<td></td>
<td>Vacancy clusters</td>
<td>-</td>
<td>&lt;0.25</td>
<td>[109]</td>
</tr>
<tr>
<td></td>
<td>Dislocations</td>
<td>-</td>
<td>0.15-0.3</td>
<td>[110]</td>
</tr>
<tr>
<td></td>
<td>Dislocations</td>
<td>Austenitic</td>
<td>~0.11</td>
<td>[102]</td>
</tr>
<tr>
<td></td>
<td>Dislocations</td>
<td>Austenitic</td>
<td>0.2</td>
<td>[103]</td>
</tr>
</tbody>
</table>
Table 3.8 – Table showing the relationship of obstacle strength $\alpha$ and random array factor $f$ used in this work, after Toloczko [96].

<table>
<thead>
<tr>
<th>Barrier strength, $\alpha$</th>
<th>Random array factor, $f$</th>
<th>$f \times \alpha$</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.15</td>
<td>0.41</td>
<td>0.06</td>
</tr>
<tr>
<td>0.20</td>
<td>0.46</td>
<td>0.09</td>
</tr>
<tr>
<td>0.25</td>
<td>0.51</td>
<td>0.13</td>
</tr>
<tr>
<td>0.30</td>
<td>0.55</td>
<td>0.17</td>
</tr>
<tr>
<td>0.35</td>
<td>0.59</td>
<td>0.21</td>
</tr>
<tr>
<td>0.40</td>
<td>0.62</td>
<td>0.25</td>
</tr>
<tr>
<td>0.45</td>
<td>0.64</td>
<td>0.29</td>
</tr>
<tr>
<td>0.50</td>
<td>0.67</td>
<td>0.33</td>
</tr>
<tr>
<td>0.55</td>
<td>0.69</td>
<td>0.38</td>
</tr>
<tr>
<td>0.60</td>
<td>0.71</td>
<td>0.43</td>
</tr>
<tr>
<td>0.65</td>
<td>0.73</td>
<td>0.47</td>
</tr>
<tr>
<td>0.70</td>
<td>0.75</td>
<td>0.52</td>
</tr>
<tr>
<td>0.75</td>
<td>0.77</td>
<td>0.58</td>
</tr>
<tr>
<td>0.80</td>
<td>0.78</td>
<td>0.62</td>
</tr>
<tr>
<td>0.85</td>
<td>0.80</td>
<td>0.68</td>
</tr>
<tr>
<td>0.90</td>
<td>0.80</td>
<td>0.72</td>
</tr>
<tr>
<td>0.95</td>
<td>0.80</td>
<td>0.76</td>
</tr>
<tr>
<td>1.00</td>
<td>0.81</td>
<td>0.81</td>
</tr>
</tbody>
</table>

Figure 3.20 – Graph showing the effect of the random array factor, $f$, on barrier strength for hardness calculations, after Toloczko [96].
3.9 – **Application of developed correlations to predict tensile properties**

The correlations developed for strength and ductility in this work will be used to predict the tensile yield strength, ultimate strength and uniform elongation of a set of materials irradiated in a commercial boiling water reactor (BWR), for which no tensile specimens were available in the irradiated condition. Miniature tensile and shear punch specimens were, however, available for a set of four unirradiated 316 stainless steels that had different thermomechanical treatments, i.e., the same materials that are included in a correlation for 316 stainless steel variations in Section 4.2.1. In a ‘proof-test’ of the correlations developed, tensile values predicted for the four stainless steel thermomechanical treatments were compared to the miniature tensile test results in a ‘feedback’ exercise designed to test the accuracy of the strength and ductility correlations. The correlations were then used to predict tensile yield strength, ultimate strength and uniform elongation for the BWR-irradiated stainless steel heats, previously outlined in Section 3.2.5, for which no tensile specimens were available in the irradiated condition.

3.10 – **Finite element simulation of shear punch test**

A finite element model of the shear punch test was used to investigate certain characteristic aspects of the shear punch-tensile yield strength correlation. In particular, it was of interest to learn why the correlation slope for yield strength deviates from the ideal von Mises value of $\sim 1.73$ (see Section 3.5) and also to learn more about the nature and origin of the $\tau_0$ offset, which from experimental work prior to this study appears to be material dependent. The aim of this part of the work was to be able to evaluate shear yield strength from the model and to construct an ‘FEM-tensile’ correlation for yield strength that could be compared to the experimentally produced shear punch-tensile correlation for yield strength. The uniradiated 316 stainless steels in four thermomechanical conditions (Section 3.2.4) were chosen for the model since they represented a wide range of strengths and ductility and both tensile and shear punch specimens were available.

A schematic of the model is shown in Fig. 3.21. The finite element model was constructed using the MARC code in 2 dimensions and was axisymmetric$^6$ about an axis of revolution or angularly symmetric.

---

$^6$ Axisymmetric - symmetrical about an axis of revolution or angularly symmetric.
centriline through the middle of the punch. This reduces the complexity of the model, and hence the processing time required for completing an analysis. The model was capable of simulating material yielding, but no failure criteria were specified, as the model would only be run to a point just beyond the perceived yield point on the punch test load-displacement trace. Load on the punch is measured as a function of its displacement, as is the case experimentally. The geometry of the specimen, punch and die, including all corner radii, can be changed, and the material properties of the specimen can be changed to simulate different materials or material conditions. The die and punch are modelled as being rigid, and a non-penetration constraint between the working piece, punch and die is handled by a subroutine in the MARC code. The coefficient of friction between contacting surfaces can be changed to reflect different material/tooling variations. A summary of the details of the model, including diagrams of the model is shown in Appendix 5.

![Finite Element Model Diagram](image)

Figure 3.21 – Schematic diagram of finite element model for shear punch test showing magnified view of elements. There are 50 elements across the thickness of the specimen and the sizes of the elements are graded towards the region in the specimen where the majority of the deformation occurs (see Appendix 5 for more details of model and for screen pictures of the model).

The finite element model requires uniaxial tensile properties of the particular material that is being modelled. The true stress versus true plastic strain plots for the four cold-work conditions of the 316 stainless steels are shown in Fig 3.22. The tensile data, which were obtained from miniature tensile test specimens, were corrected for machine
compliance. The model was run for each of the four material conditions using the same punch and specimen geometry as in the experiments. The corner radius of a sharpened punch was determined to be approximately 0.010 mm from a shadowgraph apparatus and the corner radius of the fixture bore was assumed to be the same. The model was also used to evaluate whether friction between the punch, specimen and die was in any way part or wholly responsible for the $\tau_0$ offset that is a feature of shear punch-tensile correlations for yield and ultimate strength.

Figure 3.22 – True stress versus true plastic strain traces used for the finite element model for four cold work conditions of 316 stainless steel.

### 3.11 – Parametric study

To complete the investigation, a parametric study was completed on the shear punch test parameters. Throughout the development of the shear punch test, it was apparent to this and previous authors that the shear punch test is likely to be sensitive to factors such as the punch alignment, the relative sharpness of the punch and bore and the specimen thickness. To aid in the understanding of test results, a finite element method was used to model the shear punch test to evaluate the stress state in the deformation zone of the TEM disk up to and slightly beyond large scale yielding.
CHAPTER 4 - Results

4.0 Results

The mechanical and microstructural properties of all the material groups tested will be presented first, followed by the correlations developed and their subsequent application to predict tensile strength and ductility properties of a set of irradiated austenitic stainless steels. Also presented are the findings from a study to determine the sensitivity of the shear punch test technique to experimental and physical parameters. The questions that arise from the materials properties will be addressed in the discussion section.

4.1 Application of results toward specific fundamental considerations

The following section details the physical testing results of the materials tested during the course of this work. The results are presented with limited discussion at this stage.

4.1.1 - Comparison of full size and miniature tensile test specimens

Fig. 4.1 shows a direct comparison between two miniature tensile specimens and one full-size tensile specimen made from 5182-O aluminium sheet (annealed Al-4.5Mg-0.35Mn alloy). Comparable results are obtained from the two miniature sized specimens and the full-size specimen despite the difference in specimen size. For a comparison, the maximum recorded load for the full size specimen is \( \sim 850 \) kg and the maximum recorded load for the miniature tensile specimens is \( \sim 9.5 \) kg, which reflects a factor of \( \sim 100 \) between the cross-sectional area of the gauge lengths of the two specimen sizes. The strain serrations that are observed in the loading curve are thought to be the result of dynamic strain ageing or the Portevin-Le Chatelier effect \([111, 112, 47 \text{ and } 28]\). In the case of the full size specimen, multiple Lüders bands could be seen in the gauge length with the eye on the specimen surface at an angle approximating \( 45^\circ \) to the applied stress. For further discussion, see Section 5.1.1.1.

The data presented here are from monolithic specimens that were tested to demonstrate the validity of results from the miniature specimen geometry. The data for the full size specimen test were supplied by a client interested in the capabilities of the miniature tensile test for characterising the welded region between two butt-welded plates of different thicknesses. It is not known why the strain serrations are not as pronounced in the results from the full size specimen test as in those from the miniature specimen test.
but the supplied data may have been sampled at a different frequency or a smoothing function may have been used when the data were recorded.

![Graph showing comparison of typical results from miniature versus full size tensile specimen made from 5182-O aluminium sheet (nominally Al-4.5Mg-0.35Mn).](image)

**Figure 4.1** – Comparison of typical results from miniature versus full size tensile specimen made from 5182-O aluminium sheet (nominally Al-4.5Mg-0.35Mn).

### 4.1.2 - Shear punch testing of isotopically tailored ferritic alloys

The effective shear yield and maximum shear strengths of a series of irradiated, isotopically tailored ferritic alloys were evaluated using the shear punch test as an exercise in testing highly radioactive materials. Two tests per specimen condition were performed with good reproducibility in the data: effective shear yield strength typically varied by no more than ±15 MPa between duplicate specimens. Since this test series was not directly relevant to the development of the correlation and this author completed no microscopy on the materials tested, the reader is referred to the publication in Appendix 6. However, a short summary of the work follows:

It is known from previous experiments that shear punch yield and ultimate properties can be linearly correlated to the equivalent tensile properties [56, 55]. Qualitative determination of trends in material properties of irradiated materials can therefore be made in the absence of any available tensile specimens by conducting shear punch tests.
on TEM sized specimens. The shear yield and shear maximum strengths of an irradiated series of isotopically tailored ferritic alloys were evaluated using the shear punch test. The composition of three of the alloys tested was nominally Fe-12Cr-1.5Ni. A different balance of nickel isotopes was used in each alloy to produce different helium levels (see Section 2.2.1). A fourth alloy, which contained no nickel, was also irradiated as a control. Figs. 4.2 and 4.3 show that the addition of nickel at any isotopic balance to the Fe-12Cr base alloy significantly increased the shear yield and maximum strengths of the alloys, and as expected, the strength of the alloys decreased with increasing irradiation temperature. It can also be seen that helium itself, up to 75 appm at 7 dpa, appears to have little effect on the mechanical properties of the alloys.

Figure 4.2 - Effective shear yield strengths ($\tau_{yy}$) in Fe-12Cr-1.5Ni as a function of helium content (an open symbol signifies the control alloy [Fe-12Cr] at the same condition as the corresponding filled symbol).
CHAPTER 4 - Results

The testing exercise demonstrated that the shear punch test facility was capable of handling highly radioactive specimens within the strict limitations imposed by the need for radiological control. As the ferritic specimens were initially considered potentially friable by the radiological control staff, it was a concern that spreading contamination within the test facility would be possible. During the post-testing period, radiological control personnel declared that the spread of radioactive contamination was limited to the cutting edges of the shear punch test fixture, which by the design of the test fixture, could be replaced. In completing this preliminary testing, the potential of the shear punch test for predicting qualitative trends in irradiated material properties was realised.

4.1.3 - Shear punch testing of isotopically tailored $^{59}$Ni alloys

TEM disks of three neutron-irradiated, simple model alloys (Fe-25Ni-15Cr, Fe-25Ni-15Cr-0.04P, and Fe-45Ni-15Cr) were tested. Two shear punch tests were performed per specimen condition in the case of the irradiated disks and five in the case of the unirradiated controls. The standard deviations in the measured effective shear strength and maximum shear yield strength of the controls were of the order of 15 MPa and 8 MPa respectively.
Fig. 4.4 shows both the yield strength data from the shear punch test and the original yield data from the miniature tensile testing for materials irradiated at 365°C. It can be seen for both tensile and shear punch tests that the yield strength measurement of each alloy in both starting conditions saturates before 10 dpa. The yield strength of the 25Ni+P alloy saturates at a slightly higher level than the 25Ni, which in turn saturated with a higher yield strength than the 45Ni alloy. There is no obvious effect of helium level, and the saturation strength is independent of the thermomechanical starting state for the materials irradiated at 365°C. The only significant difference between results of the two types of test is the characteristic lower level of shear yield, compared with that of the tensile yield.

Figure 4.4 – The influence of thermomechanical starting state and different He/dpa ratios on the tensile yield and shear yield strengths of three austenitic alloys irradiated below the FFTF core at 365°C. He/dpa ratios of 0.5 and 15 appm He/dpa were generated in the undoped and doped alloys, respectively.
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The shear punch test results for the alloys irradiated at 490°C at a lower dpa rate (Fig. 4.5) produce the same general findings as the original tensile results [66], i.e., the yield strength tends to approach a saturation level that is independent of the starting condition. The total exposure to the materials irradiated at 490°C in this sequence was low (6 dpa) and therefore a common saturation strength for the CW and SA material starting states has not yet been achieved. Once again there was no obvious influence of helium/dpa ratio. The scatter in the data measured from specimens irradiated at 490°C by the shear punch test was larger than was seen in the miniature tensile data.

![Figure 4.5](image)

Figure 4.5 – The influence of thermomechanical starting state and helium generation rates of 0.3 and 20 appm He/dpa on the effective shear yield strength of specimens irradiated at a relatively lower dpa rate above the FFTF core at 480°C (dashed lines indicate SA starting state and solid lines indicate the CW starting state).

The alloys irradiated at 495°C in the fully isothermal sequence, which have accumulated up to 39 dpa at a higher dpa rate, show that they are at a more advanced stage in the saturation of the yield properties than the alloys irradiated at 490°C (Fig. 4.6). There would appear to be some effect of helium in this series, however, which acts as to strengthen especially the SA Fe-15Cr-45Ni, and to a lesser extent, the 25Ni+P alloy. It is unclear whether there is any significant effect of helium on the shear yield properties in the 25Ni alloy. Most interestingly, the SA and CW specimens in this sequence appear to approach common saturation levels as expected, but levels that are different for the low and high He/dpa specimens. This phenomenon will be addressed in more detail in the discussion section.

The behaviour of specimens irradiated at 495°C, which had experienced the non-isothermal sequence initially, are shown in Fig. 4.7. In the later stages of the irradiation,
the tensile results showed that the yield strength of the materials was still recovering from a peak induced by the lower temperatures in the last portion of the non-isothermal event (Fig. 4.8). The shear punch test results shows the same downward trend in yield strength for the two 25Ni alloys. The convergence in shear yield strength from the SA and CW starting conditions is complete after 52 dpa. The saturation level of the shear yield strength of materials irradiated in the two 495°C sequences is higher than that in the 490°C sequence, reflecting primarily a difference in the displacement rate.

Figure 4.6 – The influence of starting state and He/dpa ratios of 0.5 and 5 appm He/dpa on the effective shear yield strength of specimens irradiated in the fully isothermal sequence at the bottom of the core of FFTF at 495°C (dashed lines indicate the SA starting state and solid lines indicate the CW starting state).

Figure 4.7 – The influence of starting state and He/dpa ratios on the effective shear yield strength of specimens irradiated in the initially non-isothermal sequence at the bottom of the core of FFTF at 495°C.
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Figure 4.8 – Convergence of tensile strengths in solution annealed Fe-15Cr-25Ni in two irradiation sequences conducted in FFTF-MOTA [66]. In the first sequence (solid line), there was a very irregular temperature history in the first of three irradiation increments, while in the second sequence, all increments proceeded isothermally (dashed line).

The shear yield data at 495°C and 29 dpa from the non-isothermal sequence matches that from the fully isothermal sequence at the same dpa level. After 52 dpa, the shear yield strengths of the non-isothermal sequence are showing further convergence at a level that is consistent with that of the isothermal sequence (Figs. 4.6 and 4.7). This result further emphasises the fact that the saturation strength of these alloys is independent of the thermomechanical starting state.

4.1.4 – Barrier hardening calculation for $^{59}$Ni materials

A barrier hardening calculation was performed using microstructural data from a selected few irradiated conditions of Fe-15Cr-25Ni-0.04P and Fe-15Cr-45Ni where there appeared to be an effect of helium on the effective shear yield strength that was evaluated from shear punch tests. The model was based upon microstructural data evaluated in a previous study by Stubbins and Garner [70], which are reproduced in Table 4.1. The details of the barrier hardness calculation are shown in Section 2.8.
Table 4.1 – Microstructural data for selected alloys in the $^{59}\text{Ni}$ series, after Stubbins and Garner [70].

<table>
<thead>
<tr>
<th>Material, Condition, helium content</th>
<th>Voids</th>
<th>Large Faulted loops</th>
<th>Small Faulted Loops</th>
<th>Network dislocat-ions</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>Number density</td>
<td>Average diameter</td>
<td>Number density</td>
</tr>
<tr>
<td></td>
<td>$n_v$ $\times 10^{21}$ m$^{-3}$</td>
<td>$d_v$ (nm)</td>
<td>$n_L$ $\times 10^{21}$ m$^{-3}$</td>
<td>$d_L$ (nm)</td>
</tr>
<tr>
<td>Fe-15Cr-25Ni-0.04P, SA</td>
<td>1.67</td>
<td>19.3</td>
<td>22.0</td>
<td>0.8</td>
</tr>
<tr>
<td>Fe-15Cr-25Ni-0.04P, SA, +He</td>
<td>2.13</td>
<td>18.8</td>
<td>16.9</td>
<td>1.7</td>
</tr>
<tr>
<td>Fe-15Cr-45Ni, SA</td>
<td>0.39</td>
<td>18.2</td>
<td>33.7</td>
<td>0.3</td>
</tr>
<tr>
<td>Fe-15Cr-45Ni, SA, +He</td>
<td>0.51</td>
<td>17.8</td>
<td>31.0</td>
<td>1.0</td>
</tr>
<tr>
<td>Fe-15Cr-45Ni, CW</td>
<td>0.12</td>
<td>18.6</td>
<td>27.8</td>
<td>2.0</td>
</tr>
<tr>
<td>Fe-15Cr-45Ni, CW, +He</td>
<td>0.11</td>
<td>28.6</td>
<td>20.1</td>
<td>1.7</td>
</tr>
</tbody>
</table>

As can be seen from the results of the barrier hardness calculation in Table 4.2, the model predictions for yield strength fall short of the measured tensile values. A similar disparity between the measured and barrier hardness-evaluated yield strength values was observed in similar materials Fe-15Cr-XNi \{X=25-45\} in an experiment by Brager et al. [113]. The difference between the measured and barrier hardness-evaluated yield strength increased with increasing nickel content and was attributed to a decomposition of the matrix into alternate nickel-rich and nickel-poor regions that resulted in additional hardening (See Section 5.3.3.1 for additional discussion).

The model predictions agree with the shear punch data in that there is a consistent difference between the yield strengths of the $^{59}\text{Ni}$-doped (high helium content) and undoped alloys of the same composition. The barrier hardness predictions show a difference of ~80 MPa between the alloys with a high helium content and the alloys with a low helium content and the corresponding tensile data shows a difference of ~50 MPa.
Table 4.2 – Barrier hardening theory applied to the microstructural data from materials irradiated at 495°C to 14 dpa. Stubbins and Garner, [70], completed the microstructural data used in this calculation. Eqsns. 3.4 and 3.5 were used, with \( m = 3.1 \); \( \mu = 75 \text{GPa} \); \( b = 2.55 \times 10^{-10} \); \( f = 0.8 \) and \( \alpha = 0.9 - 1.0 \) for cavities; \( f = 0.8 \) and \( \alpha = 0.45 \) for faulted loops and \( f = 0.4 \) and \( \alpha = 0.25 \) for network dislocations.

<table>
<thead>
<tr>
<th>Specimen details</th>
<th>Contribution to hardening, ( \Delta \sigma _{(\text{obstacle})} )</th>
<th>Total change in yield strength</th>
<th>Tensile yield strength of solution annealed condition</th>
<th>Model prediction for yield strength</th>
<th>Yield strength from tensile test</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>CAVITIES</td>
<td>FAULTED LOOPS</td>
<td>NETWORK DISLOCATIONS</td>
<td>( \Delta \sigma _{(\text{obstacle})} )</td>
<td>( \Delta \sigma _{y(t)} )</td>
</tr>
<tr>
<td>Material</td>
<td>Condition</td>
<td>( \Delta \sigma _{c} )</td>
<td>( \Delta \sigma _{L} )</td>
<td>( \Delta \sigma _{l} )</td>
<td>( \Delta \sigma _{n} )</td>
</tr>
<tr>
<td></td>
<td></td>
<td>(MPa)</td>
<td>(MPa)</td>
<td>(MPa)</td>
<td>(MPa)</td>
</tr>
<tr>
<td>Fe-12Cr-25Ni-0.04P</td>
<td>SA</td>
<td>273</td>
<td>61</td>
<td>43</td>
<td>49</td>
</tr>
<tr>
<td></td>
<td>SA+He</td>
<td>303</td>
<td>78</td>
<td>76</td>
<td>56</td>
</tr>
<tr>
<td>Fe-12Cr-45Ni</td>
<td>SA</td>
<td>128</td>
<td>47</td>
<td>23</td>
<td>56</td>
</tr>
<tr>
<td></td>
<td>SA+He</td>
<td>145</td>
<td>82</td>
<td>52</td>
<td>52</td>
</tr>
<tr>
<td>Fe-12Cr-45Ni</td>
<td>CW</td>
<td>72</td>
<td>109</td>
<td>39</td>
<td>63</td>
</tr>
<tr>
<td></td>
<td>CW+He</td>
<td>85</td>
<td>86</td>
<td>46</td>
<td>63</td>
</tr>
</tbody>
</table>

* Theoretical change in yield strength due to microstructural development during irradiation
* Measured yield strength of unirradiated, solution annealed starting state
* Predicted yield strength after irradiation
* Tensile specimens not available
4.1.5 – Shear punch and tensile testing of 316 stainless steel variations

Fig. 4.9 shows typical stress-strain curves for the miniature tensile specimens, showing that irradiation at lower temperatures and higher dpa levels increases the yield strength and reduces the ductility of the steel. The yield strength of the steel was found to saturate after ~10 dpa at a level that is a strong function of irradiation temperature, as shown in Fig. 4.10. Generally, the transition from irradiation hardening to overall softening from the 20% cold worked starting state occurs between 470°C and 550-570°C for all three alloys, with that of CN13 being around 470°C. Both the starting yield strength and saturation yield strengths were dependent on the heat identity. The yield strength of alloys A1 and A61 saturate at a higher level than CN13 for each irradiation temperature.

As shown in Fig. 4.11, the effective shear yield strengths exhibited all of the same features as the tensile yield strength data although numerically the effective shear yield strengths were roughly a factor of two lower, resulting from the difference in stress state of the two specimen types. The larger level of data scatter seen in the shear punch test data was partly a consequence of the fabrication technique of the specimens, which had been prepared by punching disks from sheet stock. This can introduce a shear lip around the punched edge and also an element of ‘dishing’ to the specimen cross section. Both these factors make it more difficult to obtain consistent results.

Immersion density measurements show that the specimens covered a wide range (<0 to 8%) of swelling levels as seen in Fig. 4.12. The duration of the incubation period prior to swelling and the transient regime is dependent on irradiation temperature and somewhat on composition. The microstructural details of swelling were also evaluated for select conditions as shown in Section 3.1.6.
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Figure 4.9 – (a) Typical stress strain curves derived from tensile specimens of the CN13 heat irradiated in the first segment of the FFTF-MOTA experiment, showing primarily the effect of irradiation temperature and (b) Typical stress-strain curves derived from tensile specimens of the CN13 heat irradiated at 400-430°C in the FFTF-MOTA over four irradiation segments, showing the influence of dpa level in reducing the ductility.
Figure 4.10 – The influence of heat identity, irradiation temperature and dpa level on the evolution of tensile yield stress of the three heats of 20% cold worked 316 stainless steel.
Figure 4.11 - The influence of heat identity, irradiation temperature and dpa level on the evolution of shear yield stress of the three heats of 20% cold worked 316 stainless steel.
Figure 4.12 – Swelling of AISI 316, as measured by an immersion density technique, showing that a wide range of swelling levels and sometimes slight densification were obtained in this experiment.
4.1.6 – Microscopy and yield strength calculation for irradiated 316 SS

The microstructural data from 20% cold worked 316 stainless steel (CN13) irradiated in the FFTF MOTA are presented in Table 4.3. Transmission electron microscopy pictures of the microstructures of the three irradiated conditions are shown in Figs. 4.13 – 4.15. Table 4.4 shows the results of a barrier hardness calculation that was conducted on the basis of the microstructural data evaluated and using the same method described in Section 3.7. The predicted yield strengths are shown, together with the experimentally measured values.

At 430°C and 17.5 dpa (Fig. 4.13), no voids are seen in the microstructure of the CN13 heat, which is in agreement with the immersion density swelling measurement for CN13 at 17.5 dpa in Fig. 4.12. The high network dislocation line density dominates the microstructure of the material but a significant number of faulted interstitial loops have also been introduced into the microstructure from fast neutron interactions with the matrix atoms (see Section 2.2.3). The network dislocation density of the specimens irradiated at 430°C appears to have saturated at \( \sim 4 \times 10^{15} \text{ m}^{-2} \) which may well represent a slight increase from the original network dislocation density level for a 20% CW austenitic stainless steel material, which is of the order of \( 1 \times 10^{15} \text{ m}^{-2} \). The increase in the yield strength of CN13 irradiated to 17.5 dpa at 430°C in Fig. 4.10 is therefore due to the increased introduction of sessile faulted interstitial loops that were introduced by the irradiation. The results of the barrier hardness calculation using the microstructural data in Table 4.2 shows that significant contributions to the yield strength of CN13 irradiated to 17.5 dpa at 430°C are made from both the network dislocations and the faulted loops. The barrier hardness calculation predicts the yield strength at 611 MPa, which compares to the experimentally measured value of 873 MPa. Only one tensile specimen was tested in this material condition and a more reliable indication is the saturation yield strength of CN13, which is closer to 800 MPa. Little or no precipitation was seen in these alloys (Fig. 4.15a) and so they were not included in the yield strength calculation. It is possible that the faulted loop number density was underestimated which is why the predicted value is low.
### Table 4.3 - Microstructural data for 316 stainless steel (CN13) irradiated in the FFTF-MOTA.

<table>
<thead>
<tr>
<th>Irradiation temperature</th>
<th>430°C</th>
<th>430°C</th>
<th>550°C</th>
</tr>
</thead>
<tbody>
<tr>
<td>Displacements per atom</td>
<td>17.5</td>
<td>88</td>
<td>60</td>
</tr>
<tr>
<td>Cavity number density (m⁻³)</td>
<td>-</td>
<td>2.1 \times 10^{21}</td>
<td>0.015 \times 10^{21}</td>
</tr>
<tr>
<td>Mean cavity diameter (nm)</td>
<td>-</td>
<td>36.6</td>
<td>190.0</td>
</tr>
<tr>
<td>% Swelling (from void measurements)</td>
<td>-</td>
<td>4.0</td>
<td>5.1</td>
</tr>
<tr>
<td>% Swelling from density measurements</td>
<td>0.28</td>
<td>7.5</td>
<td>8.0</td>
</tr>
<tr>
<td>Faulted loop number density (m⁻³)</td>
<td>1.8 \times 10^{21}</td>
<td>-</td>
<td>2.85 \times 10^{21}</td>
</tr>
<tr>
<td>Faulted loop mean diameter (nm)</td>
<td>25.2</td>
<td>-</td>
<td>18.5</td>
</tr>
<tr>
<td>Network dislocation line density (m⁻²)</td>
<td>4.2 \times 10^{15}</td>
<td>3.7 \times 10^{15}</td>
<td>0.48 \times 10^{15}</td>
</tr>
</tbody>
</table>

### Table 4.4 - Barrier hardening calculation for 316 stainless steel irradiated in FFTF-MOTA using Eqns. 3.4 and 3.5 with \( m = 3.1, \mu = 75\text{GPa}, \) \( b = 2.55 \times 10^{-10}, \) \( f = 0.8, \) and \( \alpha = 0.9 - 1.0 \) for cavities; \( f = 0.8, \) and \( \alpha = 0.45 \) for faulted loops and \( f = 0.4, \) and \( \alpha = 0.25 \) for network dislocations.

<table>
<thead>
<tr>
<th>Irradiation temperature</th>
<th>430°C</th>
<th>430°C</th>
<th>550°C</th>
</tr>
</thead>
<tbody>
<tr>
<td>Displacements per atom</td>
<td>17.5</td>
<td>88</td>
<td>60</td>
</tr>
<tr>
<td>Tensile yield strength (MPa)</td>
<td>873^a</td>
<td>797</td>
<td>460</td>
</tr>
<tr>
<td>Hardening from cavities (MPa)</td>
<td>-</td>
<td>391</td>
<td>80</td>
</tr>
<tr>
<td>Hardening from faulted loops (MPa)</td>
<td>108</td>
<td>-</td>
<td>116</td>
</tr>
<tr>
<td>Hardening from network dislocations (MPa)</td>
<td>305</td>
<td>286</td>
<td>104</td>
</tr>
<tr>
<td>Total change in yield strength form barriers (MPa)^b</td>
<td>412</td>
<td>677</td>
<td>245</td>
</tr>
<tr>
<td>Predicted yield strength (MPa)</td>
<td>611</td>
<td>876</td>
<td>444</td>
</tr>
</tbody>
</table>

^a - result from a single test
^b - weighted contribution
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Figure 4.13 - Microstructure of 20% CW 316 SS (CN13) irradiated at 430°C to 17.5 dpa seen in dark field and in dislocation contrast.

Figs. 4.14a and 4.14b shows the microstructure in bright and dark field for the specimen irradiated to 88 dpa at 430°C. The microstructure of the CN13 in this condition is dominated by the presence of voids, although a high network dislocation density still exists in the surrounding matrix. The barrier hardening calculation is reasonably accurate for the material irradiated to 88 dpa at 430°C, with the calculation slightly over-predicting the yield strength that was evaluated from miniature tensile testing.

The microstructure of the specimen irradiated at 550°C to 60 dpa is shown in Figs. 4.15a and b. A relatively low density of large voids was observed in the material that was irradiated at the higher irradiation temperature and a significant amount of Frank faulted loops were observed in the matrix away from the voids. The barrier hardness calculation accurately predicts the measured tensile yield strength.
Figure 4.14 – Microstructure of 20% CW 316 stainless steel (CN13) irradiated at 430°C to 88 dpa seen in (a) bright field for void contrast and (b) dark field contract for network dislocations.
Figure 4.15 – Microstructure of 20% CW 316 stainless steel (CN13) irradiated at 550°C to 60 dpa seen in (a) low magnification and bright field for void contrast and (b) dark field for network dislocations.
4.1.7 – Tensile and shear punch testing of unirradiated 316 stainless steels

Fig. 4.16 presents the results of miniature tensile and shear punch tests on the unirradiated 316 stainless steel specimens. The results from miniature tensile tests on the different cold worked levels follow the expected trends: there is an increase in yield and ultimate strength and a reduction in ductility with increasing cold work treatment. The shear punch test curves show similar trends in terms of the increase in effective shear yield and shear maximum strengths. The displacement to failure does not correlate well with uniform elongation, which instead can be evaluated from shear punch test data by a ductility correlation that will be reported later in this work (Section 4.2.3).
Figure 4.16 – (a) Tensile yield and (b) effective shear yield strengths observed in unirradiated 316 stainless steel, heat CN13.
4.2 – Empirical correlation for strength and ductility

The shear punch-tensile correlations for strength in this work were constructed by plotting coordinate pairs of tensile strength (ordinate) versus effective shear strength (abscissa). The resulting correlations take the form $\sigma_{y,UTS} = m(\tau_{sy,sm} - \tau_0)$, where $m$ is the slope of a linear regression of the data and $\tau_0$ is the x-axis intercept of the regression line (see Section 3.5 for details). The shear punch-tensile correlation for ductility relates tensile uniform elongation and the tensile strain-hardening coefficient to the ratio of $(\tau_{sm}/\tau_{sy})$ by the method described in Section 3.6.

Two TEM specimens were tested for each irradiated condition and five specimens for each unirradiated condition for both the $^{59}$Ni doping series and the 316 stainless steel variations. The tensile testing for the $^{59}$Ni alloys was completed in a previous study [66], and two miniature tensile specimens were available for each irradiated condition for the 316 stainless steel variations. At least three shear punch tests and three tensile tests were completed for the unirradiated CW 316 SS alloy conditions and also for the irradiated heats that required tensile property evaluation. From the results of shear punch tests on the unirradiated material, it was established that the effective shear yield and maximum strengths of duplicate specimens typically exhibited a standard deviation of $\sim 15$ and $\sim 8$ MPa, respectively.

4.2.1 – Shear punch – tensile correlation for yield strength

The correlations between effective shear yield strength and uniaxial tensile yield strength for the three model alloys from the $^{59}$Ni doping experiment were plotted individually and in all cases the slopes were approximately 2, with a slightly different $\tau_0$ offset in each case (see Fig. 4.17). In an effort to derive a simple and consistent correlation between tensile yield and effective shear yield stresses for this work it was decided that all the data would be fit to a single master slope. As the value of the regression slopes for yield strength correlation for each of the $^{59}$Ni materials was on or about 2, this was the value chosen for the master slope. Regressions were recalculated for the three $^{59}$Ni materials with the slope set equal to 2, producing a new $\tau_0$ value for each alloy. The three $^{59}$Ni data sets are combined on a plot of $\sigma_y$ vs. $(\tau_{sy} - \tau_0)$ in Fig. 4.18.
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Figure 4.17 - Correlation between effective shear yield strength, from the shear punch test, and uniaxial tensile yield strength from miniature tensile specimen testing for three model austenitic alloys from the 59Ni experiment. The data have not been adjusted for fixed slope or $\tau_0$ offset.

Figure 4.18 - Correlation between $\sigma_y$ and $(\tau_{sy} - \tau_0)$ for three model austenitic alloys from the 59Ni series. The $\tau_0$ value for each material was determined after setting a slope of 2 to each data set in a correlation of $\sigma_y$ and $\tau_{sy}$.

The effectiveness of this yield strength correlation is measured by the standard deviation of the measured tensile strength from the value that the correlation predicts from the effective shear yield strength. It can be seen from Fig. 4.18 that the standard deviation of the predicted tensile value was calculated at 53 MPa. It is likely that the
standard deviation observed in the correlation could be reduced since each plotted point represents only two shear punch tests and one or two miniature tensile tests. The difficulty of defining a standard procedure for determining the yield point in a shear punch test also contributes to the prediction accuracy. It is difficult, especially for softer materials, to extract effective shear yield strength data. The scatter is clearly greatest for the data from the lower strength materials. For \( ^{59}\text{Ni} \) materials having a measured tensile yield strength greater than 400 MPa, the standard deviation in predicting the tensile yield strength was reduced to 43 MPa. This is equivalent to an error of ±10% at 400 MPa or ±5% at 800 MPa.

The shear punch-tensile correlation for yield strength of the 316 stainless steel variations is shown in Fig. 4.19. A number of alloys (Irradiated austenitic alloys CN13, A1, A61 and unirradiated 316 SS) are included in the same correlation since there was less variation in alloy composition than in the case of the \( ^{59}\text{Ni} \) alloys. The graph shows two regression lines. The first, with slope 2.2 and \( \tau_0 = 117 \) MPa, is the actual regression line for the data shown and the second line has a fixed slope of 2 with a corresponding value of \( \tau_0 = 88 \). The second line was added to investigate the application of the same slope used in the \( ^{59}\text{Ni} \) series correlation, although the slope of 2.2 seems more appropriate for the data shown. The offset parameter is slightly larger than was seen for the \( ^{59}\text{Ni} \) alloys, which enforces conclusions by previous authors that this value is material dependent [55, 74].

The standard deviations of the measured tensile data from the correlation predictions for the true and forced-slope regression lines are 57 and 62 MPa, respectively, which is slightly larger than was previously seen in the \( ^{59}\text{Ni} \) correlation.

A very important result in Fig. 4.19 is that the same slope and offset parameter can be defined for the yield strength correlation of the unirradiated 316 stainless steels as for the corresponding unirradiated controls and the three fast neutron-irradiated 316 stainless steel variations.
4.2.2 - Shear punch – tensile correlation for ultimate strength

The shear punch-tensile correlation for maximum strength for the $^{59}$Ni series has an unadjusted slope of 2.0 and offset value of 133 MPa (Fig. 4.20), while the unadjusted 316 stainless steel variations has a slope of 1.8 and an offset value of 170 MPa (Fig. 4.21). A previous correlation on unirradiated stainless steels gave a slope of 2.2 and a $\tau_0$ offset of 212 MPa [55]. Again, irradiated and unirradiated material data fit on the same correlation.
Figure 4.20 - Shear punch – tensile correlation between $\sigma_{\text{UTS}}$ and $\tau_{\text{sm}}$ for three model austenitic alloys from the $^{60}$Ni series.

Slope = 2.0
$\tau_0 = 133$ MPa

Figure 4.21 - Shear punch – tensile correlation between $\sigma_{\text{UTS}}$ and $\tau_{\text{sm}}$ for three irradiated 316 stainless steel variations and an unirradiated set of 316 SS with various CW levels.

Slope = 1.8
$\tau_0 = 170$ MPa
4.2.3 – Shear punch – tensile correlation for ductility

Figs. 4.22 - 4.25 show the stages in the development of the ductility correlation for the $^{59}$Ni materials. Fig. 4.22 (cf. Fig. 4.17) shows a tight linear relationship between the measured tensile strain hardening component, $n$, and $n_\sigma$, predicted from Eqn. 4.3. In Fig. 4.23, the value of $n_\tau$ was obtained by exchanging $\sigma_m/\sigma_y$ with $\tau_m/\tau_y$ in Eqn 4.3. As in the previous work by Toloczko et al. [57], a linear relationship with a similar slope to that seen in the $n$ vs. $n_\sigma$ comparison was obtained (cf. Fig. 4.18). The lack of one-to-one behaviour seen in both the $n$ vs. $n_\sigma$ and $n$ vs. $n_\tau$ plots is due to the method for estimating $n_\sigma$ or $n_\tau$, and not due to any factor peculiar to the shear punch test. Fig. 4.24 shows a linear relationship between $n$ and true tensile uniform elongation that provides the link between $n_\tau$ and true uniform elongation, shown in Fig. 4.25. The result is that tensile elongation data can be estimated from yield and maximum stresses from shear punch test data.

Fig. 4.26 shows a plot of true uniform elongation versus $n_\tau$ for the irradiated 316 stainless steel variations and unirradiated 316 stainless steels. The data for the 316 stainless steel variations show less scatter than in the equivalent plot for the $^{59}$Ni alloys. A similar slope was obtained in the plots of true uniform elongation vs. $n_\tau$ for both the $^{59}$Ni and the 316 stainless steel variations. The two data sets are combined in Fig. 4.27 where it is clear that all the data falls on a single line which has a regression slope of 2.4 and an x-axis intercept of 0.05.

The above ductility correlations were constructed using materials with both thermomechanically-induced and radiation-evolved microstructures. As was the case with the yield strength and ultimate strength correlations, the unirradiated and irradiated material data from a particular alloy series can be plotted on the same correlation.
Figures 4.22 - 4.25 - The development of a ductility correlation that shows a linear relationship between tensile true uniform elongation and shear punch test results.
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Figure 4.26 - Correlation between tensile true uniform elongation, $\epsilon_u$, and a strain hardening exponent, $n_\tau$, for three 316 SS variations and their unirradiated controls. The correlation slope is 2.8 and the x-axis intercept is 0.06.

Figure 4.27 - Correlation between $\epsilon_u$ and $n_\tau$ for a variety of materials, thermomechanical starting states and irradiation-evolved microstructures. The correlation slope is 2.4 and the x-axis intercept is 0.05.
4.3 – *Finite element model-tensile correlation for yield strength*

A finite element model was constructed to investigate the origin and nature of the slope and offset of the yield strength correlation (Section 3.10). The standard deviations in the experimentally measured effective shear strength and maximum shear yield strength of the controls were of the order of 15 MPa and 8 MPa respectively and the shear punch test curves show the same trends as the tensile results in terms of the increase in effective shear yield and shear maximum strengths with increasing cold work. The load-displacement traces generated by the finite element model of the shear punch test that was run to a point just beyond the effective shear yield strength are shown with the corresponding shear punch test results in Figs. 4.28a and 4.28b, respectively. The loading curve generated by the finite element model is quite stiff when compared to the experimental results (note the expanded x-axis scale in Fig. 4.28a relative to Fig. 4.28b), despite machine and test rig compliance having been removed from the experimental results. However, the important result is that the effective shear yield strength can be clearly defined from the model results for all the material conditions.

![Figure 4.28](image_url)

*Figure 4.28* – (a) Effective shear strength versus punch displacement as evaluated for four thermomechanical conditions of 316 stainless steel by a finite element model of the shear punch test. (b) Equivalent traces for the same materials evaluated by the shear punch test.
4.3.1 – Finite element model-evaluated shear punch-tensile yield correlation

The correlation that was developed from the FEM-evaluated effective shear yield strength and the experimentally determined tensile yield strength is shown in Fig. 4.29, together with the experimentally determined yield strength correlation for the same material conditions. The slopes of the two correlations are approximately equal, but the FEM-tensile correlation passes through the origin. Both these results will be discussed in Section 5.2.1.

![Graph showing tensile-shear punch (red) and tensile-FEM (blue) evaluated correlation for yield strength for unirradiated 316 stainless steel in four thermomechanical starting conditions.](image)

Figure 4.29 – Tensile-shear punch (red) and tensile-FEM (blue) evaluated correlation for yield strength for unirradiated 316 stainless steel in four thermomechanical starting conditions.

Figs. 4.30 - 4.32 show the development of the plastic zone in the region of the specimen between the punch and die for the solution annealed 316 stainless steel material condition. The stress contours show the intensity of the deviatoric stress (equivalent von Mises stress) within the specimen. A scale has been chosen such that stresses exceeding the level at which plastic strain occurs (~260 MPa) are shown in colour. The load-displacement curve in each figure shows the point during the test that corresponds to the contour plot. Fig. 4.30 shows that localised plastic yielding is occurring in the region near the punch and die. Fig. 4.31 shows the extent of the plastic zone at the point where the effective shear yield strength was determined. As can be seen, plastic deformation is occurring in a region that is wider than the clearance region of the punch and die. Fig. 4.32 shows the results from same model run to a point beyond yield.
Fig. 4.33 shows the magnitude of the major contributing stress components sampled across a line AA' that spans the plastic zone for the yielding condition shown in Fig. 4.31. The shear component $\tau_{xz}$ reaches a maximum at the centre of the deformation zone and the component $\sigma_{zz}$ reaches a maximum compressive value either side of the deformation zone. Within the clearance region, the deviatoric stress (equivalent von Mises) is a function of mostly of these two resolved stress components. The deviatoric stress reaches a broad plateau across the deformation zone that indicates yielding in a region wider than the clearance zone.

The fact that the $\tau_0$ offset is not observed in the FEM-tensile correlation suggests that there is either some systematic error involved in determining the effective shear yield strength experimentally, or that the finite element model may be deficient in some way.
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Figure 4.31 - Finite element model showing the extent of plastic deformation at the point perceived to be the effective shear yield strength in solution annealed 316 stainless steel.

Figure 4.32 - Finite element model showing the extent of plastic deformation at a point beyond the effective shear yield strength in solution annealed 316 stainless steel.
4.3.2 - The effect of friction on the FEM-evaluated effective shear yield stress

It has been proposed in the past that the $\tau_0$ offset could be attributed to friction between the punch, specimen and die which caused an increase in the experimentally evaluated effective shear yield stress [94]. When different coefficients of friction were used in FEM runs for the solution annealed material, no significant difference in the generated value of effective shear yield strength was observed. Fig. 4.34 shows two runs of the model for the solution annealed 316 stainless steel material condition, with ($\mu = 0.204$) and without a frictional coefficient. The same value for effective shear yield strength would be chosen for both iterations, which implies that friction has no effect on the effective shear yield strength, and is therefore not responsible for the $\tau_0$ offset in the yield strength correlation. This is not surprising since in the instant that yielding begins to occur, the contacting surfaces are unlikely to be moving.
Figure 4.34 - The effect of different frictional coefficients on the effective shear strength determined by a finite element model of the shear punch test that was determined for a solution annealed 316 stainless steel.
4.4 – Application of SPT-tensile correlations for strength and ductility

The correlations developed for predicting tensile properties from shear punch test data from the various irradiated and unirradiated 316 stainless steel heats were used to predict tensile yield strength, ultimate strength and uniform elongation of two sets of austenitic stainless steels in an exercise to test the accuracy of the correlations.

4.4.1 – Tensile property predictions for unirradiated 316 stainless steels

Both miniature tensile and shear punch test TEM disk specimens were available for testing for the various cold-work levels of the unirradiated 316 stainless steels. Tensile properties were predicted from the shear punch test results using the correlations developed and then compared to the measured tensile results in a ‘feedback’ exercise. Table 4.5 shows the experimental and predicted values of the tensile properties of the 316 stainless steels with various CW levels. The subscript (p) denotes a predicted value.

Table 4.5 – Measured and predicted tensile properties of 316 stainless steel in four thermomechanical conditions

<table>
<thead>
<tr>
<th>CW level (%)</th>
<th>Average experimental values</th>
<th>Predicted tensile properties</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Shear punch test*</td>
<td>Tensile test**</td>
</tr>
<tr>
<td></td>
<td>$\tau_{sy}$ (MPa)</td>
<td>$\tau_{sm}$ (MPa)</td>
</tr>
<tr>
<td>0</td>
<td>243</td>
<td>525</td>
</tr>
<tr>
<td>25</td>
<td>65</td>
<td>644</td>
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<td>50</td>
<td>570</td>
<td>727</td>
</tr>
<tr>
<td>75</td>
<td>620</td>
<td>782</td>
</tr>
</tbody>
</table>

* Average of 3 or 4 shear punch tests  
** Average of 2 miniature tensile tests

Fig. 4.35 compares the predicted with the measured values from the tensile test data. The predicted and measured yield strengths are in reasonably good agreement for all cold work levels. The agreement for the ultimate tensile strength is not quite as good, with the measured values being somewhat under-predicted as the cold work level increases. The most likely reason for this is that the maximum strength correlation is not as well defined as the yield strength correlation, since the range of values of strength was not as wide for the maximum load condition as it was for the yield condition. Thus
the slope and offset of the maximum strength correlation are not as well known for the ultimate tensile strength correlation, and the error can be expected to increase slightly at the higher strength levels.

The predicted and measured UE values are in very good agreement for the three cold worked conditions, but not for the annealed (0% CW) condition. The true uniform elongation of the two tensile specimens tested in this condition showed rather more variability than was observed in most cases; one value of UE was close to the predicted value but the other was ~10% higher. Thus the predicted value may be more valid than the data appear to imply. Additional tensile specimens were not available from the funding client.

Figure 4.35 – Comparison of mechanical properties of 316 stainless steels as a function of cold work level obtained from miniature tensile testing (open) and those predicted from shear punch test data (filled) using the developed shear punch-tensile correlations for strength and ductility.
4.4.2 – Tensile property predictions for irradiated 304 and 316 stainless steels

The correlations developed were then applied for the first time to predict tensile data for two 304 stainless steel heats and two 316 stainless steel heats irradiated in a commercial boiling water reactor where no tensile specimens were available. Table 4.6 shows the predictions that were made.

Table 4.6 – Measured and predicted tensile properties of irradiated 304 and 316 stainless steel.

<table>
<thead>
<tr>
<th>Dose (dpa)</th>
<th>Heat N</th>
<th>Experimental values</th>
<th>Predicted tensile property values</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>Shear punch test*</td>
<td>Tensile test**</td>
</tr>
<tr>
<td></td>
<td></td>
<td>( \sigma_{y(t)} )</td>
<td>( \sigma_{UTS(t)} )</td>
</tr>
<tr>
<td></td>
<td></td>
<td>TSY</td>
<td>TS</td>
</tr>
<tr>
<td>0.0</td>
<td>187</td>
<td>495</td>
<td>231</td>
</tr>
<tr>
<td>1.0</td>
<td>405</td>
<td>592</td>
<td>562</td>
</tr>
<tr>
<td>1.6</td>
<td>384</td>
<td>611</td>
<td>634</td>
</tr>
<tr>
<td>3.7</td>
<td>470</td>
<td>641</td>
<td>641</td>
</tr>
<tr>
<td>6.0</td>
<td>498</td>
<td>713</td>
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</tr>
<tr>
<td>13.3</td>
<td>543</td>
<td>744</td>
<td>744</td>
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<tr>
<td>Heat O</td>
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<td>611</td>
</tr>
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</tr>
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<td>5.0</td>
<td>528</td>
<td>722</td>
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<tr>
<td>Heat L</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>0.0</td>
<td>231</td>
<td>550</td>
<td>279</td>
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<tr>
<td>0.7</td>
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<td>728</td>
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</tr>
<tr>
<td>1.6</td>
<td>527</td>
<td>750</td>
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<td>539</td>
<td>827</td>
<td>539</td>
</tr>
<tr>
<td>Heat M</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>0.0</td>
<td>217</td>
<td>520</td>
<td></td>
</tr>
<tr>
<td>0.6</td>
<td>294</td>
<td>561</td>
<td></td>
</tr>
<tr>
<td>0.9</td>
<td>400</td>
<td>639</td>
<td></td>
</tr>
<tr>
<td>1.7</td>
<td>381</td>
<td>687</td>
<td></td>
</tr>
</tbody>
</table>

* Average of 3 or 4 shear punch tests
** Average of 2 miniature tensile tests
*** UE likely to be invalid due to failure in fillet
Figs. 4.36 - 4.39 show the trends in the tested specimens as a function of dpa. For three of the heats, one or sometimes two unirradiated miniature tensile control specimens were available. With the exception of the uniform elongation of heats N and O, the measured and predicted tensile results are in excellent agreement for the unirradiated controls. In the case of the single miniature tensile test that was carried on heat N, the specimen broke next to the fillet of the gauge length, which might explain the unusually low value uniform elongation measured for the solution annealed condition. Typically the uniform elongation in unirradiated 316 stainless steel is significantly larger than in irradiated conditions, and this is what is observed in the predictions. In the CW conditions, the lower UE values of the cold worked materials were in good agreement with the predictions. It is probably therefore reasonable to assume that the lower UE values predicted for the irradiated condition are reasonably accurate as well.

It is assumed that the yield and ultimate strength predictions for the irradiated conditions have the same level of accuracy as was observed in the unirradiated specimens and the CW 316 stainless steels and therefore that the strength predictions are reasonably accurate.
Figure 4.36 – Tensile yield strength, ultimate strength and uniform elongation values predicted as a function of dpa for components made from heat N that were irradiated in a commercial light water reactor. Properties were predicted from shear punch-tensile correlations developed on a wide range of irradiated and unirradiated austenitic 316 stainless steel.

Figure 4.37 – Tensile yield strength, ultimate strength and uniform elongation values predicted as a function of dpa for components made from heat O that were irradiated in a commercial light water reactor. Properties were predicted from shear punch-tensile correlations developed on a wide range of irradiated and unirradiated austenitic 316 stainless steel.
Figure 4.38 – Tensile yield strength, ultimate strength and uniform elongation values predicted as a function of dpa for components made from heat L that were irradiated in a commercial light water reactor. Properties were predicted from shear punch-tensile correlations developed on a wide range of irradiated and unirradiated austenitic 316 stainless steel.

Figure 4.39 – Tensile yield strength, ultimate strength and uniform elongation values predicted as a function of dpa for components made from heat M that were irradiated in a commercial light water reactor. Properties were predicted from shear punch-tensile correlations developed on a wide range of irradiated and unirradiated austenitic 316 stainless steel.
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Figure 4.38 - Tensile yield strength, ultimate strength and uniform elongation values predicted as a function of dpa for components made from heat L that were irradiated in a commercial light water reactor. Properties were predicted from shear punch-tensile correlations developed on a wide range of irradiated and unirradiated austenitic 316 stainless steel.

Figure 4.39 - Tensile yield strength, ultimate strength and uniform elongation values predicted as a function of dpa for components made from heat M that were irradiated in a commercial light water reactor. Properties were predicted from shear punch-tensile correlations developed on a wide range of irradiated and unirradiated austenitic 316 stainless steel.
4.5 – Developments and insights in the shear punch test technique

Many of the insights into the effects of various parameters on the shear punch test gained during the course of this work have been incorporated in the technical work document (TWD) and safe operating procedure (SOP) that are contained in appendices Y and X. Some of the main points will be presented here, together with other insights gained during the course of this work.

The reproducibility of the shear punch test technique has been investigated in a comprehensive parametric study using solution annealed 304 and 316 stainless steel control specimens that were fabricated by electrical discharge machining (EDM) from rolled sheet stock. The effect of punch and bore condition on the recorded effective shear yield strength of solution annealed 316 stainless steel was investigated together with the importance of fixture alignment, punch-to-bore and bore-to-bore concentricity.

Tests were conducted at elevated temperatures on solution annealed 304 stainless steel, revealing an apparent temperature effect on the value recorded for the effective shear maximum strength. This lead to an exhaustive parametric study being carried out to investigate the effect of parameters associated with the test machine, punch test fixture and testing environment to determine the cause of the apparent temperature effect.

Finally, the progression of the deformation in a specimen during a shear punch test was followed in a series of partial punch tests on solution annealed 304 stainless steel specimens. The specimens that were tested to various stages of completion were sectioned in profile to reveal the progression of the test. The fracture surfaces of punched specimens of cold worked 316 stainless steel were observed using SEM to reveal the failure mode.
4.5.1 – The effect of punch and bore condition

Shear punch testing solution annealed 316 stainless steel control specimens has shown that maintaining a sharp punch edge and an edge that is square to the bore of the lower fixture half can affect the measured effective shear yield strength. The punches are made from hardened tool steel gauge pins. The cutting edge of the punch pin is usually sanded with 600-grit sandpaper to give it a square edge for the punching operation. The top half of an old test fixture is used to hold the punch perpendicular to the abrasive surface. A small weight is applied to the top of the punch and then the punch and fixture are polished in a figure 'eight' motion on the sandpaper. In recognition of the fact that small misalignments and changes to the punch test apparatus can affect the results, the punches were prepared for other control tests on a glass surface with a 6 µm diamond paste. Fig. 4.40 shows the condition of the punch with a 600-grit finish (a) alongside a punch polished to a 6-µm finish (b). The radius of a sharpened punch was estimated at less than 10 µm when using a shadowgraph. The third picture (Fig. 4.40c) shows the condition of the punch after approximately 50 punch tests when the edge of the punch has become slightly rounded.

Similarly, the condition of the lower bore is important in the shear punch test. Fig. 4.41 shows the condition of the bore in the as received condition (a) and after finishing on a glass surface with 6-µm diamond paste (b).

Fig. 4.42 shows the results from punch tests conducted on solution annealed 316 stainless steel first with a punch and bore that were finished using 600 grit sandpaper (the original condition), secondly with punches that were ‘sharpened’ on a glass surface with 6-µm diamond paste and thirdly with a sharpened punch and lower fixture bore.
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Although the effective shear maximum strength is not sensitive to the condition of the punch and lower fixture bore, it would appear that the effective shear yield strength determined varies with the condition of the bore and punch. The transition from a punch and bore with a 600-grit sandpaper finish to a punch and bore with a 6-µm finish is marked by an increase in the measured effective shear yield strength.

Figure 4.41 - a) Condition of the lower fixture bore as received and b) condition of the lower fixture bore after replacement and polishing to 6µm finish.

Figure 4.42 - Effect of different finishes on the cutting edges of the punch and bore on the effective shear yield and maximum strengths of solution annealed 316 stainless steel.
4.5.2 – Fixture alignment and punch to bore concentricity

The precise alignment of punch, specimen and bore in the shear punch test is an issue of importance recognised by all previous authors [56, 55 and 57]. In the current configuration of the shear punch test fixture, the upper fixture bore has a slightly smaller diameter bore than the lower fixture bore, so that the punch pin is guided concentrically into the lower fixture bore as shown in Fig. 4.43. The current engineering drawings for the shear punch test-fixture are shown in Appendix 7. One recent modification has been to move the position of the location pins that guide the two fixture halves together from the lower fixture half to the upper fixture half. This allows the lower fixture bore to be skimmed and sharpened without it being necessary to remove the location pins each time. It has been found that removing and replacing the fixture alignment pins tends to reduce the concentricity of the upper and lower fixture bores in subsequent tests.

![Figure 4.43 - Schematic drawing (not to scale) of shear punch test fixture showing punch and upper and lower bore diameters. All units in inches as per supplied stock.](image-url)
4.5.3 – Shear punch testing at elevated temperatures

In a recent development of the shear punch test technique, tests were conducted on 304 stainless steel control specimens at elevated temperatures. The test facility for the shear punch test was assembled with a furnace in anticipation that tests would be conducted at different temperatures in the future (Fig. 4.13). The furnace was built in-house and can be heated or cooled; and tests can be conducted in a vacuum or an argon atmosphere. The furnace temperature was controlled from a fixed thermocouple that was situated close to the shear punch test fixture. An overtemperature thermocouple was situated close to the control thermocouple that was usually set 30-40°C above the test temperature. It is impractical to measure the specimen temperature directly via a thermocouple on the shear punch test fixture as this would require the fixture to be coupled to the furnace controls before each test is conducted. The specimen temperature was therefore monitored with a thermocouple that was inserted into a hole drilled into a cradle base that supported the shear punch test fixture. It was determined in separate trials with a wire thermocouple that was attached to a specimen that the specimen temperature closely followed the temperature recorded by the base thermocouple. The technical working document (Appendix 2) includes a more detailed account of the relationship between the specimen temperature and the base thermocouple temperature as a function of test temperature and rate of furnace heating.

Tests were carried out on solution annealed 304 stainless steel specimens at room temperature, 50°C and 100°C as a control prior to conducting a run of tests on various specimens irradiated by a spallation neutron source. Fig. 4.44 shows the results obtained from a variety of tests on the 304 stainless steel control specimens tested at three different temperatures. The effective shear yield strength of the 304 stainless steel at each test temperature does not change, but the effective shear maximum strength is changing as a function of the test temperature. This is surprising when considering that temperature-induced softening in 304 stainless steel would not be expected below 300 - 400°C. Fig. 4.45 shows typical punch test traces for the solution annealed 304 stainless steel tested at room temperature, 50 and 100°C. An exhaustive parametric study was initiated to determine whether any combination of parametric variations in the set up of

1 Spallation neutrons are high-energy neutrons resulting from the breaking up of a nucleus by a very high-energy proton, which is accelerated towards the target.
the shear punch test at room temperature and at 50°C were responsible for the differences observed in the effective shear maximum strength.

Figure 4.44 - Shear punch tests on EDM fabricated 304 stainless steel controls at three different temperatures. Data scatter includes results of tests designed to measure the effects of various deleterious parameters.

Figure 4.45 - Shear punch test traces for EDM-fabricated 304 stainless steel controls at three different temperatures.
The details and development of the parametric study will be reported in later discussion (Section 5.3.2.2). The conclusion of the parametric study was that no combination of machine or fixture parameters could account for such large variations in the effective shear maximum strengths.

The apparent temperature effect that was observed in the effective shear maximum strength of shear punch tests on 304 stainless steel was later explained by a strain-induced transformation from austenite to martensite [114] in the deformation region of the specimen. At room temperature, the transformation from austenite to the harder martensite phase gives rise to an apparent increase in the level strain hardening that occurs, which then results in a higher value being recorded for the effective shear maximum strength. The transformation occurs to a lesser degree at higher temperatures [114] and so an apparent loss in strength was observed. This phenomenon is discussed in greater detail in Section 5.3.2.3.

4.5.4 – Failure mode of shear punch tested specimens

Fig. 4.46a shows the fracture surface of a 50% cold worked 316 stainless steel specimen that was punch tested. The punch was moving from right to left in the picture. The right hand side shows the smeared surface where the material has been sheared and subsequently smeared by the punch and the left-hand side shows where the ligament finally failed by ductile-dimple fracture. Final ligament failure occurred in a tensile mode leaving a ductile-dimple fracture surface as seen in Fig. 4.46b. Fig. 4.47 shows a cross section of a 50% cold worked 304 stainless steel specimen that was tested almost to failure. When the specimen was etched to reveal the grain structure, flow lines that presumably were formed during the cold rolling operation appeared parallel to the rolling direction. The lines were quite useful in showing how the material deformed and flowed during the punch test. It appears that extensive flowing (deep drawing) has occurred in the specimen in the clearance region during the test. The flow lines are concentrated in the clearance region and rotated to be almost vertical. It would appear that final ligament failure would then occur in a tensile mode, which explains the ductile-dimple fracture surface seen in Fig. 4.46b.
Figure 4.46 - a) fracture surface of 50% cold worked punch-tested specimen (the punch was moving from right to left and the image is of the inside surface of the hole that was punched in the specimen) b) detail of ductile-dimple fracture surface.
Figure 4.47 - Specimen punch tested almost to failure then etched to reveal flow lines. The flow lines in the bulk material are parallel to the surface but are near vertical in the clearance region.

<table>
<thead>
<tr>
<th>Material</th>
<th>$d_{min, b, max}$ (mm)</th>
<th>$\sigma_b$ (ksi)</th>
<th>% CRSS</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.3607</td>
<td>0.05</td>
<td>0.0106</td>
<td>3.7</td>
</tr>
<tr>
<td>0.9007</td>
<td>0.01</td>
<td>0.03906</td>
<td>7.5</td>
</tr>
<tr>
<td>0.5002</td>
<td>0.02</td>
<td>0.05906</td>
<td>11.9</td>
</tr>
</tbody>
</table>

The discussion above is largely speculative as to the reason for the differences seen in the measured yield strengths. Further testing with a polished punch and brass wedge would be needed to confirm the idea that punch radius affects the recorded effective yield strength because of changes in the "effective" thickness between the punch and the punch die being larger for a punch with broader edges. It would be expected that there would be an similar effect of punch and bore radius on a larger scale model of the punch test since the relative size of the punch-tip radius compared to the punch diameter would be greater.
5.0 – Points of discussion

The discussion that is presented will be divided into four areas. First a discussion of the small specimen test techniques and various insights gained in the understanding of the mechanical testing will be presented and secondly, details of the correlations developed will be presented. Discussion of some of the more interesting materials properties uncovered during the course of the current work, some of which influence the correlations developed will follow, and finally the benefits and limitations of applying the correlations developed to irradiated materials will be realised.

5.1 – Discussion of the small specimen test techniques used in this study

5.1.1 – Miniature tensile specimen testing

The success of the correlations developed for irradiated materials in this study depends on using miniature test specimen geometry, since it would be impractical to irradiate full sized specimens side-by-side with TEM disks. Even if this were possible, the effects of gamma heating and flux gradients would result in poor damage homogeneity in the full sized specimens, which would invalidate the correlation. Miniature tensile specimen testing was introduced in Section 3.4.1 as a widely accepted test technique in the nuclear materials testing community for obtaining mechanical properties data from irradiated specimens. In summary, as long as a sufficient number of grains are maintained across the smallest dimension of the specimen (approximately 25, and perhaps as little as 10 [91, 78]), and the specimens are fabricated by electrical discharge machining [54], accurate results can be obtained which are consistent with the results from full size specimens. This was demonstrated in Section 4.1.1 where results of tensile tests on full sized and miniature tensile specimen fabricated from 5182-O aluminium were in good agreement (Fig. 4.1). This result further improves the confidence with which the correlations developed can be applied to obtain accurate tensile data.
5.1.1.1 – Dynamic strain ageing of 5182-O aluminium alloy

Tests were conducted on full size and miniature tensile specimens to investigate the
validity of results from miniature tensile specimens. The results of tensile tests on full
size and miniature tensile specimens made from 5182-O aluminium were in good
agreement. In tensile tests conducted on both the full size and miniature specimens, the
recorded stress-strain curves exhibited a ‘saw-tooth’ profile (Fig. 4.1). It was proposed
that the observed ‘strain serrations’ in the stress-strain curve of 5182-O aluminium alloy
were a result of dynamic strain ageing. A brief review of yield point phenomenon
follows in support of this argument:

In body centred cubic steel, the upper yield point, as shown in Fig. 5.1a, occurs when
interstitial solutes (carbon and nitrogen) pin network dislocations in the matrix by
occupying energetically favourable interstitial positions in dislocation cores. When
sufficient stress is applied, the dislocations will break free of the solutes which, under
constant strain loading conditions, will result in a load drop and a small amount of strain
(the lower yield point). The strain will occur in a discrete band of metal at an angle of
45° to the tensile axis, which is often visible to the eye. These shear bands are known
as stretcher strains or Lüders bands. Typically, several Lüders bands will form at the
lower yield point during a period known as yield point elongation (Fig. 5.1a). After the
Lüders bands have propagated to cover the entire length of the specimen gauge length,
the material strain hardens by normal mechanisms before eventually failing.

At sufficiently high temperatures and low strain rates, carbon and especially nitrogen
can diffuse at a faster rate than dislocations can move and so either the solutes catch up
with the dislocations, or other solutes intervene, to pin the dislocations once more. The
applied stress must increase before the dislocations are released once more. The process
repeats until failure creating ‘strain serrations’ in the stress-strain curve in a
phenomenon referred to as dynamic strain ageing or the Portevin-Le Chatelier effect
[111] as shown in Fig. 5.1b.
Figure 5.1 - (a) typical yield point behaviour of low carbon steel and (b) the Portevin-Le Chatelier effect in low carbon steel at different temperatures, after Dieter, [47].

In materials with interstitial solutes, i.e., carbon and nitrogen atoms in body centred cubic low carbon steel, serrated yielding may occur at temperatures close to room temperature. Dynamic strain ageing has also been observed in face centred cubic and hexagonal crystal structures [112, 115], but for alloys having substitutional solutes like the 5182-O aluminium alloy, the strain serrations are normally seen only at elevated temperatures or in quenched or irradiated alloys where diffusion of the solute (in this case magnesium) has been accelerated by the presence of supersaturations of vacancies in the microstructure. In work by Chung et al. [116] a quenched Al-Zn-Mg alloy was observed to exhibit strain serrations at 30°C and work by King et al. [115], quenched 7010 aluminium alloy (Al-Zn-Mg-Cu) exhibited strain serrations at 20°C (Fig. 5.2).

Figure 5.2 - Tensile test on quenched 7010 aluminium, exhibiting the Portevin-Le Chatelier effect at 20°C, after King et al. [115].

It is therefore surprising that the Portevin-Le Chatelier effect was observed in the 5182-O alloy at room temperature. King et al. [115] pointed out that the heat-effected zone around a weld is subjected to a thermal cycle, which is equivalent to a re-solution
treatment followed by rapid cooling. The rapid cooling effectively represents quenching conditions due to the high metallic conduction of aluminium. The tested specimens were produced by electrical discharge machining from sheet samples of the material far away from any welded region. It was concluded that either the miniature tensile samples were taken from an area of the heat affected zone, or that the samples were subjected to a heat cycle during electrical discharge machining, which left the samples in the as-quenched condition.

5.1.2 – The shear punch test technique
In this section, different aspects of the shear punch test technique are presented. The results of a parametric study are discussed and some details of factors that affect the accuracy of punch tests are outlined.

5.1.2.1 – Factors affecting the accuracy and reproducibility of shear punch test
Aside from specimen condition (fabricated by EDM vs. punched) and punch/bore sharpness, the punch test is remarkably tolerant of small variations in the test set-up (a parametric study is described in Section 5.3.2.2). The effects of numerous deleterious parameters were evaluated in a parametric study that was conducted to evaluate whether any combination of parametric variations could account for the anomalous trends observed in effective shear maximum strength that was measured from punch tests on solution annealed 304 stainless steel specimens as a function of temperature (Figs. 4.44 and 4.45). These limits represent the worst case, when considering that the results were founded on tests designed to estimate the effects of changes known to be deleterious to the punch test results. Fig. 4.44 shows a standard deviation of 10 MPa for the effective shear maximum strength and 30 MPa for the effective shear yield strength. This scatter includes all the tests conducted on 304 stainless steel during the course of the parametric study that was aimed at resolving the observed temperature effect. The reader is referred to Section 5.3.2.2 for a more complete account of the parametric study and for an explanation of the origin of the apparent temperature effect.

It has been shown for the irradiated 316 stainless steel variations that the effective shear yield and maximum strengths of duplicate specimens typically exhibit a standard deviation of 15 and 8 MPa [97]. The difficulty of defining a standard procedure for determining the yield point in a shear punch test contributes to the lower reproducibility
of the effective shear yield strength. It is difficult, especially for softer materials, to extract effective shear yield strength data. In a recent paper by Kasiviswanathan et al. [117] acoustic emission monitoring was successfully used during shear punch tests to reduce the error involved in the estimation of the effective shear yield strength.

5.1.2.2 – Effects of the shear punch test fixture condition

In Section 4.5.1 it was reported that the effective shear yield strength that is measured for a particular material condition varies with the condition of the test fixture bore and the punch. Tests conducted on solution annealed 316 stainless steel specimens, using a sharpened lower fixture bore and punch, consistently leads to an effective shear yield strength measurement that is higher than if the same specimens were tested using a blunt punch and bore combination.

Insight gained from running the finite element model of the shear punch test with two different corner radii to simulate sharpness of the punch and bore has shown that with larger radii, the compliance of the loading curve increases. This may lead to the perceived effective shear yield strength appearing to be lower for the punch tests with a ‘blunter’ tip, i.e. a systematic error.

Fig. 5.3 shows punch load-displacement traces obtained from the finite element model of the punch test on solution annealed 316 stainless steel, for runs with a small and a large punch and bore corner radii to simulate punch and bore wear. The iteration of the model with a blunt punch and bore shows a more compliant loading curve after the point where yielding starts to occur. The result of this is that the compliance of the system with a blunt punch and bore may lead to a lower value of the effective shear yield strength being interpreted experimentally.

The additional compliance that is observed in the finite element model is most likely due to the ‘effective’ clearance, up to the point of yielding, between the punch and lower die being larger for a punch with blunter edges. Up to the point of yield, the punch load is transmitted to the specimen only by the area of the punch that is in contact with the specimen (this excludes the portion that has a corner radius). The stresses in the specimen during loading emanate from the point of contact between the outermost tip of the punch and the specimen and from the point of contact between the specimen
and lower fixture bore. The path between the two points is highlighted in Fig. 5.4. It can also be seen that if the punch has a larger corner radius, i.e., is blunter \((R > r)\), then the point of contact moves towards the centreline of the punch, and the 'effective' clearance distance between the punch and bore at the point of yield increases \((C > c)\). Table 5.1 expresses the effective clearance at yield \(c\) as a percentage of the lower fixture bore radius \(R_B\) as function of the punch and bore corner radius, \(r\). The value of \(r\) for a sharpened punch has been estimated, using shadowgraph equipment, at 0.01 mm. The calculation assumes that the corner radius on the lower fixture bore is equal to the corner radius of the punch and that a \(\Omega 1.0033\) mm \((0.0395''\) punch is used in a \(\Omega 1.0414\) mm \((0.0410''\) lower fixture bore. It can be seen that when \(r\) equals 0.01 mm, the effective clearance between the punch and bore at yield \(c\) is 0.039 mm, which is equal to 7.5 \% of the radius of the lower fixture bore. This value increases to 11.3 \% for a blunt punch and bore combination.

In the case of the blunt punch, the horizontal distance from the punch to the fulcrum of the specimen, i.e., the corner of the lower fixture bore, is increasing. It can therefore be envisioned that the specimen is likely to be more compliant under loading from the punch with the larger corner radius.

Figure 5.3 – The results of a FEM-punch test on solution annealed 316 stainless steel, with different values of punch and bore corner radii to simulate punch wear.
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Figure 5.4 – Schematic diagram showing the effect of punch corner radius on the effective clearance distance at the point of large-scale yielding in the shear punch test.

Table 5.1 – Relationship between punch corner radius \( r \) and the effective clearance at yield.

<table>
<thead>
<tr>
<th>( R_B ) (mm)</th>
<th>( r_{(punch, bore)} ) (mm)</th>
<th>( c ) (mm)</th>
<th>% ( c/R_B )</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.5207</td>
<td>0.0</td>
<td>0.01905</td>
<td>3.7</td>
</tr>
<tr>
<td>0.5207</td>
<td>0.01</td>
<td>0.03905</td>
<td>7.5</td>
</tr>
<tr>
<td>0.5207</td>
<td>0.02</td>
<td>0.05905</td>
<td>11.3</td>
</tr>
</tbody>
</table>

The discussion above is largely speculative as to the reason for the differences seen in the measured yield strengths. Further testing with controlled punch and bore radii would be needed to confirm the idea that punch radius affects the recorded effective yield strength because of changes in the 'effective' clearance between the punch and lower die being larger for a punch with blunter edges. It would be expected that there would be no similar effect of punch and bore radius on a larger scale model of the punch test since the relative size of the punch tip radius compared to the punch diameter would negligible.
It should also be pointed out that the effects of changes in the corner radius of the punch do not explain the presence of the $\tau_0$ offset. In fact, as discussed above, the effect of sharpening the punch is to increase the recorded effective shear yield strength, which would result in an increased value in the $\tau_0$ offset.

5.1.2.3 – Fracture and Failure of a shear punch test specimen

The progress of a punch test can be followed from start to finish in Figs. 3.10, 4.46 and 4.57. Fig. 4.46a shows two distinct regions in the sheared surface of the specimen. The region on the right hand side of the sheared surface shown in Fig. 4.46a shows a region of plastic indentation where the punch pushed through the specimen (~60% of the specimen thickness). Tool marks can be seen along the face of this region. The remainder of the thickness shows extensive voiding and stretched ligaments after the last uncut portion of the thickness failed by tensile tearing. These two features were noted by Atkins et al. on the subject of ‘surfaces produced by guillotining’ [118, 119]. Fig 4.47 shows a cross section of a specimen that was tested almost to failure, i.e., the point of the tests that marks the transition from plastic indentation to failure.

It can be seen by the grain flow in Fig. 4.47 that the stress state in the deformation zone of the specimen during a shear punch test is not pure shear, but has a tensile component at an oblique angle to the cutting plane. When the specimen finally fails, the crack propagates from the tip of the punch to the tip of the lower fixture bore and can be seen from the cross section of a failed specimen in Fig. 3.10 (Part 6). The crack propagates across the remaining ligament at an angle that, on average, approximates 40-50° to the direction of grain flow observed in Fig. 4.47 as is shown schematically in Fig. 5.5 below.
Tensile failure of ductile materials most often occurs at an angle of \(45^\circ\) to the applied stress, i.e., the angle at which the greatest resolved component of shear occurs. This might explain why so many voids are intersected by the fracture surface as shown in Fig. 4.46b.
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5.2 – Shear punch-tensile correlations for strength and ductility

When the results of the $^{59}$Ni and AISI 316 series are combined, it is obvious that the mechanical property measurements obtained from both miniature tensile specimens and TEM shear punch tests are very consistent, and that the derived property-property correlations describing their relationship are independent of composition and starting state, temperature, dose and dose rate, helium to dpa ratio and details of irradiation history, including the absence of irradiation. Thus, the tensile-shear punch correlation is effectively independent of starting state and irradiation condition over a very wide range of microstructures whether they are induced either by thermal-mechanical treatment and/or irradiation. This independence allows the prediction of tensile properties from highly irradiated material, using the smallest amount of material, as long as a TEM disk can be produced from the irradiated materials.

In the following sections the nature of the correlations are discussed with particular attention paid to the values of the slope and x-axis offset that are common to the yield and ultimate strength correlations.

5.2.1 – Shear punch-tensile correlation for yield strength

The yield strength correlation developed prior to this work, summarised by Hamilton et al. [55] on a variety of Fe, Cu, V and Al based alloys, showed the correlation slope and offset to be somewhat variable when regressions were performed for individual alloys. For example, the yield strength correlation for stainless steels appeared to have a slope of $\sim 1.7$ whereas values of 2.8 and 2.6 were seen for vanadium and aluminium alloys respectively. In the study by Hamilton, it was shown that the alloy sets, some of which had only small strength ranges, could be combined to form a single correlation by choosing an appropriate value of $\tau_0$, i.e., the values of $\tau_0$ were chosen to obtain the best fit to a line with an intercept through the origin.

In the current study, however, a variety of materials having wide ranges of radiation-evolved and thermomechanically-evolved microstructures were available for shear punch and tensile testing. Comprehensive yield strength correlations, spanning a wide range of material strengths, have been constructed for the first time with the confirmation that the tensile-effective shear correlation does not change with the previous data base on unirradiated materials. It has been possible to derive a more
simple and consistent approach for producing correlation than previous methods, where an appropriate $\tau_0$ offset was chosen for each material.

Fig. 5.6 shows the yield strength correlation for the three model alloys from the $^{59}$Ni doping experiment. The data have not been adjusted for offset or slope, but in each case, the slopes are $2.0 - 2.3$. Table 5.2 shows the unadjusted correlation slopes and $\tau_0$ offsets for each of the other material sets tested in the current study. The slopes all lie in the same range, although the $\tau_0$ offset values still appear to be material dependent. The wide range of material strength data available in the current study was considered to increase confidence in the accuracy of the correlations, and so the question arose whether there might be some fundamental reason for the regression slopes to tend to this value.

Figure 5.6 – Correlation between effective shear yield strength, from the shear punch test, and uniaxial tensile yield strength from miniature tensile specimen testing for three model austenitic alloys from the $^{59}$Ni experiment. The data have not been adjusted for fixed slope or $\tau_0$ offset.
Table 5.2 – Summary of the yield strength correlations produced in this study

<table>
<thead>
<tr>
<th>Materials</th>
<th>Number of data points</th>
<th>Slope</th>
<th>$\tau_0$ offset, MPa</th>
<th>Range of tensile yield strengths</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fe-12Cr-25Ni $^a$</td>
<td>33 $^d$</td>
<td>2.3</td>
<td>85</td>
<td>131 - 801</td>
</tr>
<tr>
<td>Fe-12Cr-25Ni-0.04P $^a$</td>
<td>29 $^d$</td>
<td>2.0</td>
<td>38</td>
<td>145 - 837</td>
</tr>
<tr>
<td>Fe-12Cr-45Ni $^a$</td>
<td>29 $^d$</td>
<td>2.1</td>
<td>49</td>
<td>151 - 750</td>
</tr>
<tr>
<td>316 variations $^b$</td>
<td>36 $^d$</td>
<td>2.2</td>
<td>117</td>
<td>206 - 1069</td>
</tr>
<tr>
<td>CW 316 SS $^c$</td>
<td>4</td>
<td>2.1</td>
<td>108</td>
<td>275 - 1100</td>
</tr>
</tbody>
</table>

$^a$ - 3 irradiation temperatures up to 52 dpa from CW + SA starting states with and without helium generation plus unirradiated controls

$^b$ - 3 alloys with 5 irradiation temperatures up to 88 dpa from a 20% CW starting state with unirradiated controls plus 4 unirradiated cold work conditions of AISI 316 stainless steel

$^c$ - Four individual cold work conditions of AISI 316 stainless steel used in FEM-tensile correlation

$^d$ - Each data point on a correlation represents a different material condition and is the average of 2-3 shear punch tests and 1-2 tensile tests

In preliminary studies on the yield correlation, Lucas [56] noted that the regression coefficient or slope in a tensile-shear punch correlation for yield data from a variety of materials when combined was close to $\sqrt{3} = 1.73$ (See Appendix 4). This is the ratio of pure shear yield stress to uniaxial tensile yield stress in the von Mises yield criterion, i.e., $\sigma_y = \sqrt{3} \tau_y$. This result would be expected, given that the tensile test specimen experiences pure uniaxial tension and that the process zone in the specimen during a shear punch test experiences a state of pure shear. These assumptions lead to an idealised correlation having a slope of $\sqrt{3}$ that passes through the origin as shown schematically in Fig. 5.7.
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Figure 5.7 – Idealised shear punch-tensile correlation for yield strength leading to a correlation of slope $\sqrt{3}$ and passing through the origin (also see Appendix 4).

As can be seen, this idealised correlation does not match the experimentally determined correlation that has a slope of 2.0-2.3 and does not pass through the origin. Kullen [99] previously applied the Tresca yield criterion, with some success, to predict the tensile yield strengths of a number of materials after conducting a series of Ø3mm punch tests on TEM disks. The Tresca yield criterion is a simplified and more conservative version of the von Mises yield criterion that is often used for materials prone to brittle fracture. This criterion gives a ratio of 2 between a state of pure shear and a stress state of uniaxial tension that would give rise to a slope of 2 on the yield correlation. However, it may not be reasonable to assume using either criterion that the stress state in the deformation zone of the shear punch test specimen is that of pure shear. Additional stresses that are likely to exist from compression, stretching and bending in the deformation region between the punch and die should be considered in the application of either yield criterion. It may have been fortuitous that the Tresca criteria ‘fitted’ the data. If these additional stresses are inserted into the von Mises yield criterion for the state of stress during the shear punch test, the ratio of uniaxial tensile yield stress to effective shear yield stress will be greater than $\sqrt{3}$. If this were the case, then a rationale may have been found to explain the convergence of the experimental slopes of the yield correlation at ~2.1.
In the early part of this work (the $^{59}$Ni materials), it was recognised that there probably would be additional stresses present in the shear zone of the specimen in the shear punch test at the point of yielding. In an effort to derive a simple and consistent correlation between tensile yield and effective shear yield stresses, it was decided that all the data would be fit to a single master slope of 2.0. By this method, $\tau_0$ has been determined in a more controlled manner than by the previous method of choosing the 'best fit' value, and it can be seen that the data now fit a single correlation line. As more materials have now been tested, the value of the yield correlation slope appears to be more likely in the region of 2.1 – 2.2.

The finite element model was constructed to investigate whether a more complex stress state exists in the deformation zone in the shear punch test specimen during yielding, that might be responsible for the correlation slope being larger than $\sqrt{3}$. The model has shown that a compressive stress component in the deformation region contributes significantly to the deviatoric stress that causes deformation in the specimen during the shear punch test. It was concluded that the stress state is not pure shear and the visible result of this is that the slope of the correlation increases from the idealised value of 1.73 towards the experimentally determined value of ~2.1.

5.2.1.1 – The origin of the $\tau_0$ offset in the yield strength correlation

In the studies by previous authors [56, 55], it was shown that some alloy sets, which generally had small strength ranges, could be combined to form a single correlation by choosing an appropriate value of $\tau_0$ for a given slope, i.e., the values of $\tau_0$ were chosen to obtain the best fit to a line with an intercept through the origin. In this study it has been shown that for a particular alloy set (similar composition and structure), $\tau_0$ is a constant. The preferred format for the correlations in this work is to use an x-axis ($\tau_0$ offset), rather than to remove it from the correlations.

The fact that the $\tau_0$ offset is not observed in the FEM-tensile correlation suggests that there is either some systematic error involved in determining the effective shear yield strength experimentally, or that the finite element model is deficient in some way. According to the finite element model, a load of 100 N is required to cause yielding in the solution annealed 316 stainless steel, but in the corresponding shear punch test on
the same material, a load of 208 N is required at the point perceived to be the effective shear yield strength. A difference of 100 – 150 N is seen between the experimentally measured and the FEM evaluated effective shear yield strength for all four conditions of the 316 stainless steels. It is this constant that is equivalent to the $\tau_0$ offset.

The fact that the tensile yield strength is measured as 0.2% strain offset and that the effective shear strength is measured as the deviation from linearity does not account for the offset. If the tensile data were adjusted to represent the deviation from linearity, or conversely, if the effective shear yield stress was recorded at some offset strain value, the $\tau_0$ offset would only increase in size. It was pointed out in Section 5.1.2.2 that the effects of changes in the corner radius of the punch do not explain the presence of the $\tau_0$ offset. It was shown that the effect of sharpening the punch is to increase the recorded effective shear yield strength, which would result in an increased value in the $\tau_0$ offset.

### 5.2.2 – Shear punch-tensile correlation for ultimate strength

The correlation between ultimate tensile strength and effective shear maximum strength generally shows larger $\tau_0$ values and slopes comparable to the yield correlation. This is consistent with the findings of previous authors. Unlike in the case of the yield strength correlation, there is no reason why the slope of the ultimate strength correlation should have a slope approaching the value of 2.1. No explanation is offered as to the origin of the offset, but as in the case of the yield strength correlation, $\tau_0$ appears to be material dependent.

It is difficult to envisage that the maximum shear strength is related to tensile ultimate strength considering the vastly different modes of failure, but nevertheless the empirical correlations developed appear to work. After the punch has started to penetrate the specimen during a shear punch test, further deformation forms a shear process zone between the die and punch.

It would appear from the sectioned specimens in Fig. 4.47 that there is a rotation to a tensile failure mode as suggested by the ductile-dimple fracture surface. Rudimentary calculations in a model that considers failure of a specimen in a punch test to occur in a tensile mode over an annular region do not produce realistic failure loads, i.e., a load to failure that is consistent with experimental data.
5.2.3 – Shear punch-tensile correlation for ductility

The ductility correlation provides a means for estimating the tensile uniform elongation from shear punch data. Figs. 4.22-25 show the various stages in the development of the ductility correlation described in Section 2.5 for the $^{59}$Ni series alloys. Fig. 4.26 shows a plot of true uniform elongation versus $n_t$ for the irradiated 316 variations and unirradiated 316 stainless steels. The data for the 316 material variations show less scatter than the equivalent plot for the $^{59}$Ni alloys (Fig. 4.27). This may be a reflection of the quality of specimens available, and the number of tested specimens contributing to each data point, i.e., there was greater redundancy in the 316 stainless steel specimen matrix. The result may also reflect refinements in the practices employed during shear punch testing, in particular the maintenance of the sharpness of the punch and die.

A similar slope was obtained in the plots of true uniform elongation vs. $n_t$ for both the $^{59}$Ni and the 316 variations. The two data sets are combined in Fig. 4.27 where it is clear that all the data falls on a single line which has a regression slope of 2.35 and an $x$-axis intercept of 0.05. The corresponding result in the study by Toloczko et al. [93] gave a slope of 2.26 and $x$-axis intercept of 0.066 (Fig. 3.19). In the work by Toloczko, the ductility correlation was formed using a variety of materials having a number of crystalline structures, i.e. face centred cubic, body centred cubic and hexagonal close packed. The data from this study supports the result from the original work that a single linear correlation exists between $\varepsilon_u$ and $n_t$ for a variety of materials, crystalline structures, thermomechanical starting states and irradiation-induced microstructures.
5.3 – *Discussion of results from mechanical testing and microscopy*

The following discussion pertains to some of the interesting materials properties and anomalies that were uncovered during the course of this study. Where appropriate the potential impact of some of these materials properties on the success of the correlations are discussed.

5.3.1 – Mechanical and microstructural analysis of $^{59}$Ni alloys

The $^{59}$Ni alloys were the first of the available materials for the correlation to be punch tested. These disks were originally intended for microscopy examination and density change measurements. Garner et al. [66] reported the tensile results for the $^{59}$Ni series in a previous experiment. This experiment showed in general that all the $^{59}$Ni alloys approached saturation levels of strength and ductility that were independent of He/dpa ratio and starting condition, but that were sensitive to the irradiation temperature and dose rate.

5.3.1.1 – An apparent effect of helium on model austenitic alloys in the $^{59}$Ni series

As might be expected, the shear punch test results replicate the trends seen in the original miniature tensile study. However, the one result which significantly differs from that of the original miniature tensile study is that, in the shear punch test, there appears to be an effect of helium on the evolution of the yield properties in the Fe-15Cr-25Ni-0.04P and especially the Fe-15Cr-45Ni alloys during the early stages (14-29 dpa) of the isothermal irradiation at 495°C (see Fig. 4.6). The tensile test experiment [66] did not include any of the materials in the cold worked condition irradiated at 495°C, and the tensile data for the solution annealed material did not show any obvious or consistent difference between the $^{59}$Ni doped and the undoped alloys (Fig. 4.8).

Helium gas is produced by nuclear transmutation reactions of nickel and boron (Section 2.2.1.2). The overall effects of helium on the mechanical austenitic 316 stainless steel are still not clear, but it is thought that it plays a role in high temperature embrittlement and in the nucleation of voids (Section 2.3.3.4) [120, 121, 66]. It is important that the effects of helium are studied as it is anticipated that first-wall materials in fusion reactor will experience helium generation levels of up to 10 appm He/dpa [122].

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Helium is insoluble in austenitic and ferritic alloys and so tends to accumulate at grain boundaries or precipitate interfaces. Austenitic alloys containing high levels of helium (after irradiation) exhibit a significant reduction in ductility above 550°C due to helium bubbles collecting at grain boundaries: at higher temperatures, grain boundary sliding is increasingly dominant over slip mechanisms as the mode of deformation and helium bubbles at the grain boundary reduce the ductility of the material. Helium will also accumulate in voids and in work by Mansur and Coghlan [120] it was shown that the effect of helium in voids is to lower the critical radius for bias driven cavity growth which can result in an acceleration of the transient regime of void swelling. This might also be reflected in a higher number density of smaller voids that develop in a material subject to a high He/dpa generation rate.

In an effort to explain some of the trends seen in some of the irradiated conditions with and without helium content (Fig. 4.6), a dispersed-barrier hardening calculation was conducted based on microstructural data from a study by Stubbins and Garner [70] of the microstructure of some of the materials irradiated at 495°C to 14 dpa (Tables 4.1 and 4.2). In these examples, the tensile data show a possible strengthening effect of helium, although it should be pointed out that in some cases, a reversal in the trend was seen in the tensile properties of Fe-15Cr-25Ni in the study by Garner [66] (Fig. 4.8 – 2nd sequence between 14 and 29 dpa).

The barrier model predictions that were presented in Section 5.1.4 are consistently lower than the tensile results by ~150 MPa for the Fe-15Cr-25Ni-0.04P alloy and ~70 MPa for the Fe-15Cr-45Ni alloy. The model predicts a difference of ~80 MPa between the yield strengths of the high and low helium content alloys in Table 4.2 and the miniature tensile test results show a corresponding difference of ~50 MPa for the conditions chosen. Assuming a valid model was selected, this suggests that there was an effect of helium on the evolution of the microstructure and also that some component of hardening was not accounted for in the model. Stubbins reported that no precipitation was observed in these materials [70], and so either the barrier strength of the voids, faulted loops and network dislocations has been underestimated, or there is some other strengthening mechanism has been overlooked.
On studying the microstructural data (from the same $^{59}\text{Ni}$ materials) by Stubbins in Table 4.1, it can be seen in general that the void number density is larger for the $^{59}\text{Ni}$ doped (helium producing) alloys, and that the corresponding average diameter was smaller. For these materials, the voids generally account for most of the barrier hardening in the microstructure. The high number density of voids results in additional hardening, which explains why the helium-containing alloys have a slightly higher yield strength as observed both in theory and experimentally.

Stubbins and many other authors that worked on the same materials [71, 123, 66, 124] commented that although the influence of helium was seen to increase network dislocation density and void number density of the $^{59}\text{Ni}$ materials after irradiation, the effect was secondary to that of phosphorous content, recent irradiation temperature and composition.

A similar divergence between barrier hardness model predictions and tensile measurements for Fe-15Cr-XNi ($X=25-45$) was observed in an earlier experiment by Brager and Garner [113]. Fig. 5.8 shows the disparity observed between the measured and the predicted radiation-induced changes in the yield strength of the alloy system as a function of the nickel content.

![Figure 5.8 - Disparity observed between measured and predicted radiation-induced changes in yield strength. G is the shear modulus, b is the burgers vector, $\alpha = 0.2$, $\beta = 1.0$, $N_d$ and $N_{FL}$ are the line lengths of dislocations and Frank loops, $\rho_v$ is the density of voids and $d_v$ their mean diameter, after Brager and Garner [113].](image-url)
The barrier hardening calculation used by Brager [113] is of a similar form to that used in this study, although some of the coefficients used differ\(^1\), but the trends are nevertheless quite clear. In the absence of any discreet phases or precipitates in the microstructure, the difference between the measured yield strength and the yield strength predicted from the barrier hardness calculation was attributed to radiation-induced spinodal-like decomposition of the matrix that was absent in alloys with 25% nickel but increased strongly in alloys with the range 35-45% Ni [113, 125]. Spinodal decomposition may occur in any alloy that is quenched. If the total free energy of the system can be reduced by forming two separate phases, the as alloy will slowly separate into the two phases with a characteristic wavelength between peak concentrations of each phase [126].

The decomposition of the high nickel alloys observed by Brager and Garner [113] resulted in alternating regions of enhanced nickel and reduced chromium and regions of enhanced chromium and reduced nickel that exhibit a period of 200-400 nm. Garner et al. [125] showed the compositional fluctuations, which were determined by Energy Dispersive X-ray (EDX) analysis, in a later paper. In the extreme case, the decomposition leads to near stoichiometric zones of Fe\(_3\)Cr and FeNi. Garner was able to observe the compositional fluctuations in the material by electropolishing techniques that resulted in selective chemical attack of the nickel-depleted areas. It was postulated that the phenomenon was an expression of a thermodynamic instability, as a result of the high nickel content, that was accelerated by radiation-enhanced diffusion rather than an effect of radiation-induced segregation.

It is possible that the additional hardening, which was not accounted for in either the barrier hardening models by Brager [113] or this work, was due to the interaction of dislocations with the internal stress field of the spinodal microstructure and also variations in the stacking fault energy of the areas of different composition.

\(^1\) The factor \(m\) that relates the shear stresses on a slip plane in a single crystal to the applied tensile stress is equal to 3.1 for a f.c.c. material, but in the study by Brager and Garner, \(m\) was taken to be \(\sqrt{3}\).
5.3.2 – Mechanical and microstructural analysis of 316 stainless steels

The tendency of the yield strength of AISI 316 to reach a saturation state dependent on the irradiation temperature is consistent with the behaviour observed in larger specimens of 20% cold worked AISI 316 irradiated in the EBR-II fast reactor [103]. The effective shear yield stress also follows the same behaviour.

5.3.2.1 – Microstructural analysis of irradiated 316 stainless steels

The barrier hardening calculation performed using the microstructural information for the CN13 material evaluated in this work (Table 4.3) is reasonably accurate for the specimens irradiated to 88 dpa at 430°C. The microstructural data for CN13 irradiated at 430°C to 17.5 and 88 dpa shows that the dislocation density has saturated at $\sim 4 \times 10^{15} \text{ m}^{-2}$ and that by 88 dpa, extensive voiding has occurred as indicated by the density swelling measurements in Fig. 4.12. It would appear from Fig. 4.12 that the incubation period of the void swelling in CN13 irradiated at 430°C finished soon after 17.5 dpa.

It is known from the tensile testing from this and other experiments [9, 66, and 103] that AISI 316 reaches a saturation state dependent on the irradiation temperature after as little as 10 dpa. Contrary to the results of miniature tensile and shear punch tests in this work, the barrier hardness calculation does not indicate that the yield strength of the material has saturated after 17.5 dpa (Figs. 4.10 and 4.11). In fact the yield strength predicted by the barrier hardness calculation from the microstructure of the material irradiated to 17.5 dpa is considerably less than that of the material irradiated to 88 dpa and it would appear from this calculation that the yield strength of the 316 stainless steel is still increasing at 17.5 dpa. On this evidence, it is concluded that the barrier hardness model is deficient in this case or that some element of hardening has not been accounted for.

It is not possible that black spot damage (small faulted interstitial loops) were present in the microstructure at 17.5 dpa. Maziasz [12] wrote that that black spot damage in the microstructure disappears above 300°C in 25% CW PCA² (as seen in Fig. 5.9). The measured faulted loop number density in this work for the CN13 stainless steel that was
irradiated at 430°C to 17.5 dpa ($1.8 \times 10^{21}$ m$^{-2}$) also agrees with the data summarised in Fig. 5.9. It is possible that small cavities in the microstructure that were not measured could account for the discrepancy between the barrier hardness calculation and the measured yield strength. As indicated above, at 17.5 dpa the material is still in the incubation period of void swelling, and so presumably many cavities exist that are below the critical size for bias driven void swelling. It is thought that if these cavities, which were to small to resolve in the microstructural analysis, were included in a barrier hardness calculation for the material irradiated at 430°C to 17.5 dpa, then the predicted yield strength might be more representative of the yield strength measured by tensile testing.

The barrier hardness calculations for the CN13 that was irradiated at 430°C to 88 dpa appears to be reasonably accurate, as does the microstructural calculation for the material irradiated at 550°C to 60 dpa. Although these two conditions exhibited the same swelling level, their strengths were not comparable. As can be seen by the Figs

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2 PCA – Prime Candidate Alloy is a 316 type stainless steel that was proposed for a first wall material in the International Thermonuclear Experimental Reactor (ITER).
4.14 and 4.15, the microstructure of the material irradiated at 550°C has fewer, but much larger voids. The total volume of cavities in each case is quite similar as indicated by both the cavity void swelling and density swelling measurements in Table 4.3. At 550°C, the reduced number of barriers available to impede dislocations accounts for its lower yield strength (especially the voids).

5.3.2.2 – Temperature sensitivity of the effective shear maximum strength of 304-type austenitic stainless steel alloys

It was shown in Section 4.5.3 that the effective shear maximum strength of solution annealed 304 stainless steel changes as a function of temperature, for tests conducted between room temperature and 100°C, whereas the effective shear yield strength at each test temperature remains the same (Figs. 4.44 and 4.45). Yield point phenomena [127] can immediately be ruled out in this case since the temperature changes affected only the effective maximum shear strength, and not the effective shear yield strength.

In an effort to find out whether the observed phenomenon was indeed a material effect or whether the change in temperature had some adverse effect on the test fixture or machine, a parametric study was initiated to establish the effect of a wide range of test parameters on the results of a test. A chronological account of the parameters varied in this study follows:

Routine checks were made on the equipment for calibration in case the anomalies that were observed at the three temperatures were machine induced. Since the temperature of the load cell was regulated at all test temperatures by a circulating coolant, temperature sensitive effects on the electronic load measurement could be eliminated as a potential source of error. During a typical test, the specimen temperature is controlled with a thermocouple attached to the lower fixture half. The specimen temperature was calibrated against the control thermocouple with a wire thermocouple that was spot-welded onto a dummy specimen in the fixture. The temperature of the wire thermocouple followed the specimen temperature to within a couple of degrees centigrade during heat up. Both thermocouples were made in-house specifically for the testing and they had been fully calibrated to the American National Institute for
Standards (NIST) requirements for thermocouples before the test program began and so the specimen temperature was known to be accurate.

Having eliminated the test equipment as being at fault, it was theorised that differential thermal expansion between the fixture, bore and punch might introduce either an element of misalignment or would increase the resistance of the punch passing through the specimen. Tests were carried out at room temperature and 50°C with various levels of misalignment introduced intentionally between the upper and lower test fixture halves and also with slightly different diameter punches to simulate a change in the clearance between the punch and bore. However, no combination of alignment or clearance changes between the punch and bore affected the shear maximum strength to the same degree as was observed in the solution annealed 304 stainless steel specimens at the different test temperatures. In any case, the total resistance would have to decrease with increasing temperature in order to agree with the experimentally observed trends, which is intuitively wrong.

In order to eliminate test variables external to the shear punch test fixture, tests were conducted first in an argon atmosphere at 50°C and secondly in a different test frame and oven assembly. The second test machine was a servo-hydraulic machine that also allowed the punch test to be conducted at a speed that was an order of magnitude lower than usual to establish if the 304 stainless steel specimen is strain rate sensitive at 50 and 100°C. Both experiments revealed identical trends in the effective shear maximum strength of the 304 stainless steel specimens as a function of temperature. These tests eliminated any variable external to the shear punch test fixture as being responsible for the apparent temperature effect.

Parameters associated with the fixture were changed to see if they had any effect on the outcome of the test that might account for the apparent temperature effect. Tests were conducted with varying amounts of torque applied to the bolts that hold the two halves of the fixture together. The theory was that if differential thermal expansion were to introduce a compressive force on the specimen at the higher temperature then the load required on the punch to initiate failure of the material might be reduced. However, little difference was observed in the measured effective shear maximum strength with either tight bolts at room temperature or loose bolts at 50°C. Finally, a new fixture was
fabricated and tests were repeated at room temperature and 50°C. As was expected at this point, identical results were obtained.

It was thus concluded that no combination of varying machine or fixture parameters could account for the variations observed in the effective shear maximum strengths, at the various test temperatures and since the yield strength is the same for specimens tested at each temperature (see Fig 4.45), it was concluded that there must be some deformation-induced change occurring in the specimens.

5.3.2.3 – A temperature-sensitive, deformation-induced phase transformation

The shear punch testing results showed that the effective shear maximum strength of solution annealed 304 stainless steel changes as a function of temperature between room temperature and 100°C, although the effective shear yield strength remains the same. It is postulated that this phenomenon is due a strain-induced phase transformation from austenite to martensite in the 304 stainless steel. The diffusionless phase transformation occurs in the deformation zone of the specimen during a punch test, resulting in additional strengthening of the austenite matrix by the formation of martensite in the process zone of the austenitic stainless steel specimen under test. The following discussion presents the evidence for this case, starting with a summary of the literature associated with this phenomenon.

It has been previously observed that austenite can undergo a phase transformation to α'-martensite at temperatures well above the martensite start temperature ($M_s$) during extensive plastic deformation [128, 129, 130 and 114] by a mechanism that competes with the stress required for slip. The $M_s$ temperature is the temperature at which the face centred cubic austenite phase transforms to body centred cubic martensite under equilibrium cooling conditions. The $M_s$ temperature for plain carbon steels is 723°C. In austenitic stainless steels, however, nickel is added to stabilise the face centred cubic austenite phase at temperatures well below room temperature. $M_s$ can be approximated from an empirical expression relating $M_s$ to the alloy composition in Eqn. 5.1, after Eichelmann and Hall [131].

$$M_s \ (°C) = 1305 - (61.1Ni) - (41.7Cr) - (33.3Mn) - (27.8Si) - [1667(C + N)]$$

(5.1)
CHAPTER 5 - Discussion

Using Eqn. 5.1, the value of \( M_s \) for Fe-18Cr-8Ni (304) stainless steel is calculated as being between \(-150\) and \(-180^\circ C\), depending on the content of minor alloying elements.

The deformation-induced austenite to martensite transformation occurs above \( M_s \) and is activated especially by shear stresses within a material. The amount of shear stress required for the transformation decreases with decreasing temperature, becoming zero at the \( M_s \) temperature, where it occurs spontaneously [130]. At temperatures above \( M_s \), plastic deformation occurs by normal slip mechanisms as the resistance to the deformation-induced transformation increases. This type of strain-induced transformation is often observed in 304 stainless steel during forming operations, especially in wire drawing and deep drawing [114].

The \( M_d \) point (\( M_d > M_s \)) is defined as the temperature above which no strain-induced transformation occurs, whatever the amount of strain, i.e., the extent of the phase transformation reduces with increasing temperature [114]. \( M_d \) can be thought of as an upper value of \( M_s \) when strain is applied. In order to cold work austenitic stainless steel without forming martensite, the operation must be carried out at a temperature that is above \( M_d \). The \( M_d \) temperature can be approximated by an empirical expression relating it to the alloy composition [132].

\[
M_d (\alpha') (20/5) ^\circ C = 552 - (29.7Ni) - (13.8Cr) - (20.6Mn) + (16.9Si) - [416(C + N)]
\] (5.2)

\( M_d (\alpha') (20/5) \) is the temperature at which 5% \( \alpha' \) martensite is formed after a true strain of 20% in compression, i.e., a cold rolling operation. Using Eqn. 5.2, the value of \( M_d (\alpha') (20/5) \) for Fe-18Cr-8Ni (304) stainless steel is calculated as being between 0 and 30\(^\circ C\), depending on the content of minor alloying elements. The amount of true strain in the deformation region of the specimen during a shear punch test is, however, estimated as being greater than 20%. It might therefore be expected that the absolute value of \( M_d \) that applies to the conditions during a test would be higher than 50\(^\circ C\). It can be deduced that the deformation-induced phase transformation occurs more extensively during a punch test on 304 stainless steel at room temperature than it does in a test at 50\(^\circ C\). This would explain the observed reduction in effective shear maximum
strength for those specimens tested at the higher temperatures seen in Figs. 4.44 and 4.45. Further shear punch tests on the 304 stainless steel control specimens would be required at temperatures above 100°C to establish the value of \( M_d \) for the solution annealed 304 stainless steel, which would be marked by a cessation in the temperature dependence of the effective shear maximum strength.

The first evidence that a martensitic transformation was occurring in the solution annealed 304 stainless steel during a punch test was indicated by the fact that punched specimens and their blanks of this material became slightly attracted to a strong magnet after testing. The relative degree and amount of magnetism was not detectable using a standard calibrated ferrite scope, which required a specimen much larger than a TEM disk. The martensite phase was, however, revealed in Fig. 5.10, which shows a specimen that was partially punched at room temperature and then sectioned parallel to the flat face of the disk as shown in the schematic. The surface of the sectioned specimen was electronically etched with 10% oxalic acid and is shown at \( \times300 \) and \( \times750 \) using differential interference contrast to reveal the martensite grains in the austenite structure. The transformation tends to occur in whole grains and is characterised by a lath structure seen in some of the grains in the deformation zone of the punched specimen as can be seen in Fig. 5.10.

In punch tests on a 316 stainless steel, there was some evidence that the martensite transformation also occurred, but to a lesser extent, i.e., the punched 316 stainless steel specimens were not as magnetic as the punched 304 stainless steel specimens and the change in effective maximum shear strength between tests conducted at room temperature and 50°C was less. \( M_s \) and \( M_d \) were estimated using Eqns. 5.1 and 5.2 to be \(-300\) and \(-100°C\), respectively, for AISI 316 stainless steel. The higher nickel content in 316 type stainless steel results in it having lower \( M_s \) and \( M_d \) temperatures and so it is likely that 316 stainless steel is less susceptible to the martensite transformations at room temperature.
Figure 5.10 - A 304 stainless steel specimen that was partially tested at room temperature and then sectioned parallel to the face of the disk. The specimen shows evidence of strain-induced austenite-martensite phase transformation in some of the grains in the deformation zone, which appear as a lath structure. The specimen was electronically etched in 10% oxalic acid and is shown using differential interference contrast at ×300 and ×750 to reveal the martensite grains.
For a comparison, shear punch tests were carried at room temperature and 50°C on non-irradiated, nickel-based Inconel 718 (Fig. 5.11). The $M_d$ temperature for Inconel 718 was calculated by Eqn. 6.2 as being below absolute zero, i.e., the austenite to martensite transformation would not be expected at any test temperature. As can be seen from Fig. 5.11, the effective shear maximum strength of the Inconel 718 is not changing as a function of test temperature over the same temperature range that the solution annealed 304 stainless steel was tested.

![Figure 5.11 - Shear punch tests at room temperature and 50°C on Inconel 718 specimens (Fe-53.58Ni-18.13Cr-3.06Mo-1.03Ti-0.48Al-0.13Mn-0.11Si-0.08Cu-0.04C-0.008P).](image)

5.3.2.4 - The consequences of the deformation-induced austenite to martensite phase transformations on the correlations

It should be pointed out that the deformation-induced phase transformations from austenite to martensite that have been observed will only affect correlations developed for nickel-stabilised austenitic materials where tests are conducted at different test temperatures below $M_d$. For test temperatures above $M_d$, it is assumed that in the absence of any strain-induced transformation occurring, data from different test temperatures can be used to construct correlations for strength and ductility.

The above points lead to a conclusion that for nickel-stabilised austenitic materials that exhibit strain-sensitive phase transformations of this type, separate shear punch-tensile correlations for maximum strength and uniform elongation, will have to be developed for individual test temperatures below $M_d$. Since the deformation-induced austenite-martensite phase transformation is a post-yield phenomenon, the shear punch-tensile
correlation for yield strength should be insensitive to test temperatures above or below $M_d$.

Should martensite or other phases be formed as the result of an irradiation-induced phase transformation, assuming for the moment that $M_d$ for material under test is below the test temperature, it is expected that the correlations would be insensitive to their presence in the microstructure, as in both the miniature tensile and shear punch tests the correlations have already been shown to be valid over a wide range of thermomechanically-evolved and irradiation-induced microstructures.

It has been shown in several instances, however, that the $M_s$ temperature, and presumably the $M_d$ temperature, are sensitive to the compositional changes that can occur during irradiation. For instance, the formation of irradiation-induced $\gamma'$ (Ni$_3$Si) and carbide (Cr$_{23}$C$_6$) phases [133, 9] has been observed to increase the $M_s$ temperature of austenitic stainless steel [134, 135]. In another case, the $M_s$ temperature of austenitic stainless steel was increased by virtue of nickel depletion at grain boundaries and voids due to radiation induced segregation [136, 137, 9].

Following an example by Mazey et al. [135], a calculation was performed to calculate the potential change in the $M_s$ temperature and more importantly the change in the $M_d$ temperature for CN13 (one of the three alloys from the 300 series alloys) and a typical Fe-18Cr-8.5Ni 304 stainless steel as function of the amount of irradiation-induced phase Ni$_3$Si ($\gamma'$) and also considering the effect of Cr$_{23}$C$_6$ carbide phase formation. It is assumed here that the irradiation-induced formation of Ni$_3$Si directly results in an effective depletion of the amount of nickel and silicon in the composition of the matrix. The effective matrix composition was calculated and then applied in Eqns. 5.1 and 5.2 to calculate $M_s$ and $M_d$ as a function of Ni$_3$Si content for CN13 as shown in Fig. 5.12a. The effect of the complete formation of the carbide phase Cr$_{23}$C$_6$ is also shown. It can be seen that $M_s$ and $M_d$ are both increasing towards room temperature.

Assuming that Equations 5.1 and 5.2 are reasonably accurate, it may be concluded that the $M_d$ temperature for the 316 type alloys tested in the 300 series experiment would

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1 N.B. Irradiation-induced phases are those that would not normally occur during thermal ageing (Section 3.3.3.3).
have been below room temperature, despite any \( \gamma' \) precipitation and/or segregation of nickel. Therefore it is not expected that the correlations developed in this work would have been affected by this phenomenon.

However, it is concluded from Fig. 5.12b that the value of \( M_d \) for lower nickel 304-type stainless steels will be sensitive to the amount of irradiation-induced precipitation and hence the dose that the material was exposed to. This fact can lead to the possibility that a range of \( M_d \) values, proportional to the accumulated dose, will be obtained for a given material irradiated to a number of different dpa levels. If this were the case, then for a given test temperature, some of the high dose specimens might be exhibiting martensite transformation while the low dose specimens are not. This would not affect the yield strength correlations since it is known from this work that the martensite transformation is a post-yield phenomenon in the shear punch test, but could potentially change the slope of the maximum strength correlation and hence the ductility correlation would also be affected.
Figure 5.12 – The potential effect of radiation-induced Ni$_3$Si and Cr$_{23}$C$_6$ precipitation on the $M_s$ and $M_d$ temperatures for (a) 316 stainless steel (CN13) and (b) a typical (18Cr-8.5Ni) 304 stainless steel as calculated using Equations 5.1 and 5.2.
5.4 – Application of shear punch-miniature tensile correlations

One of the most significant results obtained during the course of this work is seen in Figs. 4.19 and 4.21 where it is seen that the same slope and offset parameter can be defined for the yield and ultimate strength correlation of the unirradiated 316 stainless steels in different thermomechanical conditions as for the corresponding unirradiated controls and the three fast neutron-irradiated 316 stainless steel variations. Furthermore, it was seen in all other correlations developed that the data points corresponding to miniature tensile and shear punch tests on unirradiated control specimens fitted on the correlations developed from the irradiated materials.

The significance of this result is that the slope and offset parameter of a given alloy set can be evaluated using unirradiated miniature tensile and shear punch test specimens and then these coefficients can be applied to predict mechanical properties of the same alloy class from shear punch tests on irradiated shear punch test specimens. This valuable result was used in the predictions of the 316 and 304 stainless steels that were irradiated in a commercial boiling water reactor.

5.4.1 – Predicting properties of materials irradiated in a boiling water reactor

The correlations developed for austenitic stainless steels were applied to predict tensile properties of materials that were irradiated in a commercial boiling water reactor, in the first practical application of the shear punch test technique.

The benchmark tests conducted on unirradiated 316 stainless steel (Figs. 4.35) showed in general that the predictions made were very accurate. A mechanical property prediction exercise was then carried out on two 304 stainless steel heats and two 316 stainless steel heats that were irradiated in a boiling water reactor (Figs. 4.36-39). With the exception of the unirradiated condition, no miniature tensile specimens were available. Strength and ductility properties that would otherwise not have been possible to measure were evaluated using the correlations developed.
5.4.2 – Application of shear punch – tensile correlations to irradiated materials

It is clear from this discussion that for future applications of shear punch-tensile correlations to predict yield strength, ultimate strength and uniform elongation of irradiated materials, knowledge of the $\tau_0$ offset is required for the materials under test. This task is made simpler by the fact that this work has shown that the correlation slope and $\tau_0$ offset for a particular alloy set can first be established by conducting shear punch and miniature or full size tensile tests on the unirradiated material. The correlation constants established for a particular alloy system from the unirradiated material condition can then be confidently applied to predict the properties of irradiated materials from the results of shear punch tests alone. The shear punch technique can be used to predict tensile properties of highly radioactivated material as long as a good quality TEM disk can be produced from the irradiated material. The shear punch technique can be applied to situations where steep neutron flux or gamma heating gradients, insufficient irradiated material or insufficient reactor volume prevent the use of even small tensile specimens.

The strain-induced transformation from austenite to martensite that was observed to have an effect the effective shear maximum strength in shear punch tests on nickel-stabilised austenitic stainless steels is the result of a unique combination of material and physical factors, i.e., the strain-induced phase transformation in the austenitic stainless steels was activated by the strain conditions during a shear punch test, but it was not activated (or at least not to the same extent) by the strain conditions during a tensile test.

It is not expected that other materials will exhibit this behaviour, but nevertheless the possibility should be a consideration in future applications of the shear punch – tensile correlations to irradiated materials. Attention should be paid in particular to materials that might be expected to undergo irradiation-induced precipitation / segregation.

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4 A range of thermomechanical conditions (obtained by either cold working or ageing) will be required to obtain the slope and $\tau_0$ offset for a given alloy.
6.0 – Conclusions

6.1 – Small specimen test techniques

The success of the shear punch – tensile correlations for strength and ductility that were developed for irradiated materials in this study depend on the reliability and accuracy of tests using miniature tensile specimens. In this and other work, it has been shown that accurate tensile data can be obtained from miniature tensile specimens that are consistent with the results from full size specimens, provided that due care is taken during specimen fabrication, measurement and testing.

In the early part of this work, the shear punch test was shown to be a powerful tool for producing qualitative trends in the mechanical properties of a set of isotopically tailored Fe-12Cr-1.5Ni ferritic alloys, where no tensile specimens were available. The shear punch tests that were carried out on these alloys showed that helium levels up to 75 appm have little, if any, effect on the effective shear yield and maximum shear strengths on the ferritic alloys tested. Shear punch data also confirm the general trends in model austenitic alloys that were observed in earlier tensile data derived from the $^{59}$Ni isotopic doping experiment. There is a convergence to a common saturation level of yield strength that depends on alloy composition, displacement rate and recent irradiation temperature but not on the thermomechanical starting condition.

Aside from specimen condition (EDM fabricated vs. punched) and punch/bore sharpness, the shear punch test is remarkably tolerant of small variations in the experimental set-up, i.e., punch to bore concentricity, punch diameter and test machine. It has been shown though repeated testing that the effective shear yield and maximum strengths of duplicate specimens typically exhibit a standard deviation of 15 and 8 MPa. In the worst case a standard deviation of 10 MPa for the effective shear maximum strength and 30 MPa for the effective shear yield strength was recorded during a parametric test designed to evaluate the effect of various deleterious effects on the experimental set-up of a punch test.
6.2 – Mechanical properties of materials tested in this study

A small effect of helium was observed in shear punch tests on a model austenitic alloy (Fe-15Cr-45Ni) irradiated at 495°C with a high inherent helium generation level (by $^{59}$Ni content). Dispersed barrier hardness calculations conducted using microstructural data available from a previous experiment showed there to be a small effect of helium on the calculated yield strength that was attributed to a higher void number density of smaller voids in the high helium containing alloy. This apparent effect of helium was unique to the particular alloy and irradiation temperature and was seen to be secondary to the effects of phosphorous content, recent irradiation temperature and composition.

A disparity between the experimentally measured change in yield strength and that predicted by barrier hardness models in the same alloy condition was observed. In this and another study on similar materials, the barrier hardness calculation estimated the yield strength as being less than that measured experimentally. The strengthening mechanism that was not accounted for in the barrier hardness calculations is postulated by other authors to be related to a decomposition of the matrix into alternate nickel-rich and nickel-poor regions that cause complex internal stress and strain fields within the material, that affect dislocation movement.

The tendency of the yield strength of three AISI 316-alloy variations to reach a saturation state dependent on the irradiation temperature is consistent with the behaviour observed in many other experiments. The effective shear yield stress also follows the same behaviour as expected. Swelling as a function of dose for all alloys starts to increase rapidly above ~ 20 dpa, indicating the end of the void incubation period.

The yield strength of the alloys that were irradiated at 430°C was higher than that of alloys irradiated at 550°C. This is a reflection of the microstructural development at the two different irradiation temperatures: a reduced network dislocation line density (from the 20% CW starting state) and a low number density of large voids account for the reduced strength of the alloy irradiated at 550°C and a higher network line density and high number density of small voids account for the strength increase of the alloy irradiated at 430°C.
6.3 – **Shear punch - tensile correlations for strength and ductility**

Empirical yield strength correlations, spanning a wide range of material strengths, have been validated for the first time. It has been possible to derive a more simple and consistent approach for producing correlations for strength and ductility than previous methods, where an appropriate $\tau_0$ offset was chosen for each material.

When the results of the $^{59}$Ni and AISI 316 series are combined, it can be seen that the mechanical property measurements obtained from both miniature tensile specimens and TEM shear punch tests are very consistent. The derived property-property correlations describing their relationship are independent of minor compositional changes, thermomechanical starting state, irradiation temperature, dose and dose rate, helium to dpa ratio and details of irradiation history, including the absence of irradiation. The tensile-shear punch correlation is effectively independent of starting state and irradiation condition over a very wide range of microstructures whether they are induced either by thermomechanical treatment and/or irradiation.

A separate empirical correlation, with different slope and offset parameters can be defined for ultimate tensile strength. The offset parameter for the ultimate strength correlation is generally larger than that for the yield strength. As with the yield strength correlation, the slope and offset for a particular alloy system in the irradiated condition is unchanged from that of the same alloys in the unirradiated condition. It is not yet completely clear why such similarities are observed for the maximum strength correlation.

With knowledge of the relevant offset parameter obtained using unirradiated materials, the current correlation allows tensile yield and ultimate strengths of irradiated materials to be predicted from effective shear maximum and effective shear yield strengths with a standard deviation of 50-60 MPa. The correlation becomes more accurate as materials become harder.

The slope of the yield strength correlation has been experimentally determined to be $\sim 2.1$ for 316 stainless steel. Consideration of the von Mises yield criterion in a model where the tensile test specimen experiences pure uniaxial tension and the process zone of a shear punch test specimen experiences a state of pure shear leads to an idealised
correlation, which has a slope of $\sqrt{3}$ and passes through the origin. However, a finite element model has shown that a compressive stress component in the deformation zone of a specimen during a shear punch test contributes significantly to the deviatoric stress that causes deformation in the specimen at yield. A correlation between tensile data and the finite element model evaluated effective shear yield strength for unirradiated 316 stainless steel has a slope similar to the experimentally determined value of 2.1.

The origin of the $\tau_0$ offset in the experimentally determined yield and ultimate strength correlations is not yet resolved. The offset appears to be material-dependent and for a particular set of irradiated materials, and it has been shown to be unchanged from that of the same alloys in the unirradiated condition. The $\tau_0$ offset is not observed in the yield strength correlation obtained using shear punch data generated by a finite element model and tensile test data, i.e. the regression line passes through the origin of the correlation. In the FEM-tensile correlation for yield strength friction had no effect on the value of $\tau_0$. Assuming that the model is not deficient in some way it suggests that there is some systematic error introduced when determining the value of effective shear stress experimentally that is responsible for the $\tau_0$ offset.

A ductility correlation produced from data on irradiated material is consistent with that obtained in earlier work from data on unirradiated materials data. A single slope and intercept can be defined for a range of different materials in both the irradiated and unirradiated condition.

6.4 – Application of developed shear punch - tensile correlations

In the absence of available tensile specimens, the correlations developed in this work were successfully applied to evaluate tensile yield strength, ultimate tensile strength and uniform elongation of two 304 stainless steel heats and two 316 stainless steel heats that were irradiated in a boiling water reactor.

The advantages of the shear punch test technique are that tensile properties of highly radioactivated material can be evaluated using the smallest amount of material, as long as a TEM disk can be produced from the irradiated materials. The shear punch test technique can be conducted in an out-of-cell facility that enables a quick turnaround. The single limitation in using the correlations developed for yield and ultimate strength
is that before predictions can be made, the $\tau_0$ offset for a particular alloy must first be evaluated from shear punch and tensile tests on unirradiated material.

In a phenomenon that is limited to nickel-stabilised austenitic stainless steels, the post-yield stress state in a shear punch test specimen can, at certain temperatures, result in a strain-induced phase transformation from austenite to martensite. This transformation does not occur (at least does not occur as extensively) in a tensile test and this difference can lead to the correlations for ultimate tensile strength and uniform elongation being sensitive to test temperatures below $M_d$ (the transition temperature above which no such phase transformation occurs). The $M_d$ transition temperature can be related to the alloy composition and hence it is expected that it is sensitive to depletion of elements in the matrix by irradiation-induced precipitation. In the worst case, this could lead to the slope and $\tau_0$ offset of the maximum strength correlation evaluated from irradiated materials, being different to that evaluated from unirradiated materials. It is stressed that only a unique combination of alloy composition, test temperature and irradiation conditions will lead to this discrepancy and that for the vast majority of irradiated materials, the correlations will not be affected.
7.0 – Future work

The value of the $\tau_0$ offset in yield and ultimate strength correlations had been shown to be a constant for alloys that have similar compositions. Future work in the development of the shear punch test might include the formation of strength and ductility correlations for a wider variety of materials to study the variability of the $\tau_0$ offset and whether the value of $\tau_0$ shows any dependency on the crystalline structure, i.e., body centred cubic, face centred cubic or hexagonal close packed.

The manner in which the effective shear yield strength is determined experimentally should be compared to that determined by the acoustic methods that were recently presented by Kasiviswanathan et al. [117]. This exercise might demonstrate whether or not the $\tau_0$ offset is a product of the current method for determining the effective shear yield strength and whether the $\tau_0$ offset can be eliminated by experimental technique.

Although the empirical correlations for yield strength has been investigated in some detail in this work, further work is required to aid the complete understanding of the empirical correlations that have been formed for predicting ultimate tensile strength from shear punch test data. A finite element model capable of simulating specimen failure might reveal more complete understanding the success of the maximum strength correlations. The effect of friction on the punch test should be evaluated for the post yield portion of the shear punch test and whether it is this factor that is responsible for the $\tau_0$ offset being, in general, larger for the ultimate strength correlations.

A three-dimensional finite element model of the shear punch test might improve the understanding of parameters such as punch to bore concentricity and angle of attack, which could not be resolved using the two-dimensional axisymmetric model presented in this work.

With knowledge gained from future work, it might ultimately be possible to replace the empirical relationships between shear punch data and tensile data by an analytical expression that relates mechanical properties from the two test types. This might eliminate the need to evaluate the $\tau_0$ offset for each new material under test.
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## Appendix 1 - Safe Operating Procedure for the Shear Punch Test

| PNL Operating Procedure | Org. Code: D9E33  
Procedure No.: SPT1  
Rev. No.: 10 December 97 |
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<tr>
<td>Author: GL Hankin</td>
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Supersedes Date: 1 July 1998 |
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X Physical Hazards  
X Hazardous Environment  
X Other: |
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| Are One-Time Modifications Allowed to this Procedure?  
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No |
| Signatures:  
GL Hankin, Author P8-15  
(Signature)  
(Date)  
ML Hamilton, Technical Reviewer P8-15  
(Signature)  
(Date)  
RH Jones, Line Manager P8-15  
(Signature)  
(Date)  
RD Sharp, Building Manager P8-55  
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SR Bivins, Radiological Control P8-55  
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**APPENDIX I**
# Shear Punch Testing of Irradiated Metallic TEM Discs

## Purpose/scope
The purpose of this procedure is to provide a generic procedure for the testing of metallic TEM size specimens.

## Applicability
This PNNL Operating Procedure (SOP) is applicable to shear punch testing of metallic TEM disc sized specimens. Tests will be performed in the tensile test frame contained in the furnace fume hood in room 5A of 326 building in the 300 area. The procedure applies to tests for irradiated and unirradiated material at both ambient and elevated temperatures.

## Definitions
- **Peanut vial** – A small, transparent vial commonly used for storing individual or multiple TEM specimens.
- **Pencil vial** – A 1/4" diameter and 4" long solid steel pencil with a screw-top end containing a single irradiated TEM specimen.
- **Temporary storage area** – A temporary storage area for peanut vials or pencil vials in lab 5A. The storage area consists of two lead wells which are ~6 in. deep. Number 1 well is adapted for use in unloading the pencil vials, and number 2 well is used for peanut vial storage.
- **Mobile cave** – A shielded storage unit on a moveable cart for use in transporting the specimens in their pencil vials from the holding area to lab 5A. The mobile cave is also used for pencil vial storage during testing (see Figure 2).
- **Cave insert** – A cylindrical lead block with holes drilled in it for holding pencil vials.
- **Extended manipulation tweezers** – A set of 6" tweezers joined in series with a 10" set of tweezers to enable remote handling of irradiated TEM discs.
- **Vial holding device** – A copper block with two holes drilled in it, used to hold the pencil vials or peanut vials during unloading.
- **Fixture holding device** – A steel plate designed to be clamped on to the working area which prevents the test fixture from rotating when tightening the Allen bolts of the test fixture.
- **Pencil forceps** – A set of long forceps adapted to grip the pencil vial so that it can be remotely handled during loading and unloading operations. Note that the grip at the end of the forceps can grip the pencil in two ways: either with the pencil gripped at 90° to the forceps or with the pencil held between the two notches on the grip. In this second position, the pencil remains parallel to the forceps.
Pencil grip – A device attached to the bottom of the temporary storage area, used to prevent the pencil vial from twisting so that its lid can be removed.

Pencil lid remover – A device used to grip the pencil lid to enable its removal from the pencil vial.

Pencil funnel – A guide used to locate the pencil vial in the pencil grip.

Specimen funnel – A funnel used to guide the tested specimen and blank into the pencil vial.

Hazard Assessment Summary

Staff members performing work in 5A / 326 Test Lab may encounter exposure to ionizing radiation from the test specimens. The dose taken will be minimal so long as the correct protective apparel is worn, the correct operating procedure is followed and ALARA recommendations are adhered to. Industrial hazards, such as positioning heavy loads, are not likely to be encountered. In an endeavor to reduce accidents involving the movement of the loaded test fixture, a tray will be employed to carry the parts from the work area to test machine. It is not anticipated that any staff will be subjected to hazardous chemicals during this procedure.

Hazard Mitigation Summary

Personal Protective Equipment - Anti contamination clothing required will be specified in the applicable RWP. Equipment that is required for this work will be specified in this SOP. Other items of required safety equipment such as safety glasses are posted at the entrance of the lab.

Emergency Response

In case of emergency, contact Battelle Emergency (375-2400)

Work shall be stopped and Radiation Control shall be notified should any known or suspected abnormal radiation exposure have occurred, or any equipment malfunction that might result in unusual personal radiation exposure have occurred.

Prerequisites

Staff members who will perform work in accordance with this work will require Radiological Worker II training and be trained in the safe handling of Hazardous Materials. In addition, lab hood safety training (SAF-IS-005) and radiation work in hoods training (SAF-IS-005RW and SAF-IS-005RWOJT) are required.
Precautions and Limitations

Working with radioactive materials/sources can cause large doses of radiation to one’s skin and extremities. Care should be taken to identify these materials. Avoid close contact with the specimens by using the remote handling tools such as described in the work instructions.

Radiological Hold Points

Intermittent radiological control will be required in accordance to the requirements of RCP-3.4.04 (Radiological work in fume cupboards and bench tops) for the safe practice of this experimental procedure. Contamination control surveys will be conducted before, periodically during and after the completion of the radiation work on the bench top within the contamination area as defined in the procedure.

Work instructions

1.0 Objective

The primary objective of the tests is to obtain load versus displacement data arising from the shear deformation of TEM sized disc specimens. The uniaxial strength and ductility of the material can then be calculated from an empirical correlation.

2.0 Specimen and Materials

The TEM specimens are usually identified by a four-digit code etched around the circumference of one surface. This code references the material and irradiation conditions each specimen was exposed to. Each specimen will have been identified and placed in a sealed and labeled peanut vial or pencil vial prior to transfer to lab 5A (the pencil vials have their own code engraved on the lid and body of each). The specimen ID code will be recorded on all data sheets and other records to enable the specimens to be correctly identified during dimensioning, testing and post-test evaluations. All data sheets and reports will be directly traceable to the specimens via this specimen code.

3.0 Measurement and Test Equipment

3.1 Measurement and test equipment used in the punch tests will be calibrated over the intended range of use.

3.2 A fixture (see Figure 1) has been constructed specifically to hold the specimens firmly while a punch is pushed through at a constant rate. Load will be measured with a standard load cell. Crosshead displacement of the test machine is assumed to be equal to punch displacement.
3.3 A data acquisition system will be used to record the load and displacement. The system will be capable of constructing load vs. displacement plots and determining the yield and maximum load obtained in the test.

3.4 Testing will be conducted on a tensile test frame, contained within a furnace. Tests will be conducted at room temperature and at elevated temperatures. The furnace temperature will be calibrated against the specimen temperature inside the closed fixture.

3.5 A specially designed containment hood, which completely encloses the test frame, will be used when testing radioactive specimens.

Figure 1. Photograph of miniature shear punch test fixtures.

4.0 Specimen acquisition and transport to temporary storage in 5A/326

4.1 Ordinarily, specimens will be contained in a labeled plastic peanut vial, but some special shipments will arrive with specimens contained in individual pencil vials, which are shielded by a lead case. A similar lead case can be inserted into the mobile cave for use in transporting the specimens from the holding area to the test area (see figure 2). An RCT will supervise specimen transfer from the holding area to lab 5A by one of two methods.

*either*

In the instance that specimens are contained in **peanut vials**, they will be transported from the holding area to lab 5A in a transport cask (pig). Load the vials one at a time into well number 2 of the temporary storage area using 10" tweezers. An RCT is then to perform a removable contamination survey and a radiation survey of the working and storage areas after
transferring specimens from the transport cask to temporary storage in room 5A.

or

In the instance that the specimens are contained in pencil vials and the mobile cave is being employed (figure 2), an RCT will supervise the loading of the mobile cave and escort the cave from the holding area to lab 5A. The dose rate of the mobile cave must not exceed 100 mR on the surface.

Figure 2. The mobile cave and lead insert

4.2 If specimens are in pencil vials, ensure that the temporary storage is secure: the brake of the mobile cave should be engaged. If the specimens are in peanut vials, then the cover pieces (shielding) should be in position over each of the two wells in the temporary storage area.
5.0 Procedure

5.1 Dimensioning of Specimens

The RCT is to observe the first specimen measurement and to perform a removable contamination survey of the Federal micrometer deck, equipment used and work area after the measurements have been taken.

The dimensions of each irradiated specimen will be measured prior to loading in the test fixture according to the following procedure in order to minimize dose taken in accordance with ALARA. The thickness of each specimen will be measured to the nearest 0.0001" prior to testing.

The following equipment will be required:

- Work tray
- Vial holding device (copper block)
- Federal micrometer
- Long handled Allen wrench
- 10" tweezers for vial handling
- Extended manipulation tweezers (6" and 10" tweezers connected in series)
- Pencil forceps
- Pencil lid remover
- Pencil funnel
- Specimen funnel

Dress and dosimetry requirements will be addressed in the RWP.

All handling of specimens, inside or outside of their individual peanut vials or pencil vials, should be carried out while maintaining at least a six inch distance from all extremities and whole body to comply with safe working dose levels. The working area should be entirely covered with disposable plastic or paper sheet. A piece of masking tape may be used on the bench top on which to temporarily record the dimensions of the specimens. The tape may not be removed from the area and must be disposed of as radiological waste.

5.1.1 Collect the required equipment and place together on top of the tray adjacent to the temporary storage area. Put on the gloves, lab coat and finger rings as required by the RWP. Contact the RCT if dose rates are unknown prior to handling or transferring to furnace fume hood.

N.B. The technician may test the specimens without the RCT present only if the specimen(s) dose rates are known and do not exceed the limiting conditions of the RWP, and the specimens do not exceed 100 mR at 30 cm and will not change the postings of the area to be transferred to.

**RADIOLOGICAL CONTROL HOLD POINT:** The RCT will perform a radiological survey of specimens and work area as required by RWP.
5.1.2 Transfer of TEM specimen from storage to the Federal micrometer for dimensioning. The TEM specimen will either be held in a peanut vial in well number 2 of the temporary storage area, or in a pencil vial in the mobile cave. The details for the transfer in each case are as follows:

\textbf{either}

In the instance that the TEM disks are held in \textbf{peanut vials}, use the 10" tweezers to transfer the specimen in its vial from the temporary storage to the vial holding device (copper block) such that the copper shields the lower portion of the vial. Using the same tweezers, remove the lid of the vial and set it on the work tray and then, using the 10" tweezers, pick up the open vial and carefully tip the TEM disc specimen on to the deck of the Federal micrometer.

\textbf{or}

In the instance that \textbf{pencil vials} are being used to hold the TEM disks, the following steps should be followed to remove the required pencil from the mobile cave, to transfer it to the unloading area and to unload the specimen for thickness measurement. Refer to figure 3 for the assembly of parts in well number 1.

1. Remove the lid of well number 1 of the temporary storage area.
2. Assuming there is nothing in well number 1 except the pencil grip at the bottom, insert the pencil funnel so that it will guide the pencil into the grip. The funnel will be left in place throughout the remainder of the test sequence involving specimens contained in pencil vials.
3. Unlock and remove the restraining bar of the mobile cave and remove the opening brick of the mobile cave. Store them on the cart of the mobile cave.
4. Identify the required pencil and remove it using the pencil forceps.
5. If necessary, use the copper block to hold the pencil vial so that you can change the grip on the pencil to get ready for lowering it into the pencil funnel in the well. Close the lid of the mobile cave.
6. Lower the pencil (lid side up) into the funnel, which will direct it into the grip at the bottom.
7. The pencil will now be standing upright in the shielded well, with the lid facing up. The specimen will remain with the body of the pencil once the lid is removed.
8. Push the pencil lid remover on to the pencil lid. Rotate the handle clockwise until the flat faces of the top and bottom ends of the pencil engage with the grip at the bottom of the shielded well and pencil lid remover. As each side engages, you will feel the lid remover drop \(\sim \frac{1}{4}\)".
Figure 3 – Assembly of parts in well number 1 of the temporary storage area for pencil vial unloading.

9. Unscrew the pencil lid (counter clockwise) using the handle on the remover. The pencil lid will be attached to the lid remover once it is unscrewed. The pencil will stay in the holder at the bottom of the storage well. Lift out the lid and holder and set the assembly by on the bench top within the posted area.

10. The pencil forceps will be used to lift the opened pencil from the shielded well and to deposit the specimen onto the deck of the Federal micrometer for dimensioning.
11. Lower the forceps into the well and grip the end of the pencil between the two notches. Lift the pencil out and set it in one of the holes of the copper holding block ensuring that the pencil remains upright. Change the grip on the pencil such that it is now held at 90° to the forceps (this makes it more practical for tipping the specimen out of the pencil).

12. Remove the pencil vial from the copper block and carefully tip the TEM disk specimen on to the deck of the Federal micrometer. Replace the empty pencil back in the pencil grip (open-end up) at the bottom of well number 1, making sure to position it securely against the set screw.

13. Put the specimen funnel on top of the pencil. A guide on the underside of the specimen funnel will ensure that the funnel locates with the open-end of the pencil.

5.1.3 The TEM disc can be moved around on the deck using the extended manipulation tweezers. In order to lift the tip of the micrometer above the deck a lever must be depressed. This can be operated using the ordinary 10" tweezers with the other hand. Using the micrometer, measure the specimen thickness in at least three different areas where there are no identification markings (i.e., near the middle) and record the measurements on the data sheet. It is advisable to measure both sides of the specimen since a lip may be present on one side of the disc in the case of punch-fabricated discs. Make a note whether the specimen thickness is that measured with the specimen code facing up or down. If the specimen was made by electric discharge machining (EDM), then it is unlikely that there will be a lip on either side of the disk, in which case test and measure it code side up. If the measurements are not consistent with each other, make additional measurements until a consistent set of thickness data or a valid thickness profile is obtained. Note the specimen thickness on a piece of tape somewhere in the posted area. The data can be transferred to the corresponding specimen worksheet once protective clothing has been doffed.

5.1.4 Leave the specimen on the micrometer deck while the fixture is prepared for receiving the specimen. The shielding around the micrometer will reduce the personal dose received for the short time that it takes to prepare the fixture for receiving the specimen in section 5.2. If there is any problem with the test fixture, or a delay in operations, the specimen should be stored in a shielded area. Either return the specimen to its vial (and store the vial in the copper block) or return the specimen to the pencil (see instructions in section 5.4.8).

5.2 Loading of Test Specimens

The RCT will observe the initial loading procedure for shear punch testing and perform a removable contamination and radiation survey of the equipment used and work area after completion of the loading operation. Subsequent surveys
will be conducted periodically throughout the test program, the frequency of which will be determined by the RCT.

Additional equipment required for this part of the procedure includes removable handles for the fixture halves, an Allen wrench with a screwdriver type handle and a jig to hold the fixture in place while the Allen bolts are tightened.

5.2.1 Make sure the base support plate, the push pins, and both halves of the fixture are completely free of debris and dust - use a disposable alcohol wipe but do not use canned air to get them clean as airborne contamination may result.

The most critical areas on the lower half of the fixture are:
- the bottom surface (where it rests on the base support plate),
- the bushing area on the top surface (where specimen is placed), and
- the recessed areas on the top surface (where the shims are set)

The critical area on the upper half of the fixture is:
- the guide hole area

5.2.2 Using a fixture handle, invert the lower half of the test fixture and cover the bore with a piece of scotch tape (for tests conducted at temperature, use copper tape). This will prevent the blank, which is formed by the shear punch test, from being separated from the fixture after testing. Position the lower half of the test fixture in the holding device on the specimen handling area.

5.2.3 Select a set of shims the thickness of which is equal to or just slightly (some fraction of a mil) less than the sum of the recess depth (0.010" for the newest fixture) and the greatest specimen thickness. For convenience, the specimen data sheet has a conversion matrix, which shows which shim should be used for a particular specimen thickness. Circle the size of the shims used on the data sheet. Using the extended manipulation tweezers, place the shims into the lower fixture recesses making sure that they are seated completely inside the recesses.

5.2.4 Retrieve the specimen from the deck of the Federal micrometer using the extended manipulation tweezers. Position it on the lower half of the fixture, centering the specimen over the blanking hole. The sanded (i.e., deburred) side of the specimen should face up to optimize the contact between the specimen and the bushing. The engraving codes can be either up or down as long as they are not in the region that will be punched. A note should be made on the specimen data sheet as to the orientation of the specimen code. If there is no sanded side, the code should be on the top side.
5.2.5 Care should be taken when positioning the TEM disc to ensure as near to exact central coverage of the punch hole as possible. These measures are taken to ensure test reproducibility but should not compromise ALARA measures to minimize dose taken.

5.2.6 Using the second fixture handle, place the upper half of the fixture on the lower half and position the Allen bolts using the 10" tweezers. Using the Allen bolt wrench, secure the bolts until almost stopped. The bores of the upper and lower fixture halves are aligned by applying a shear force between the two halves in the direction of the arrows drawn on the fixture. This can be done by the use of the fixture handles. Use the Allen wrench to tap the upper fixture half in order to bed in the shims and fixture halves. Tighten the Allen bolts with the Allen wrench in order that the shims form a good contact with both halves of the fixture. You should be able to "feel" through the Allen wrench that the two halves make contact as the Allen bolts are tightened. Remove the lower fixture handle for convenience.

N.B. Be aware at this time that there will be an effective 'window' of radiation above and below the test fixture, being emitted from the push rod holes. In addition radiation will also shine from between the two fixture halves.

5.2.7 The size of the pushpin that will be used is dependent on the specimen thickness. For convenience, a conversion matrix has been included on the specimen data sheet. It relates the measured specimen thickness to the punch diameter that should be used.

The method for selecting the pushpin is:

\[
\text{pin diameter} = 41.0 - 0.2 \times \text{(specimen thickness)}
\]

where both diameter and thickness are measured in mils. This provides a clearance between the pin and the blanking hole (which is 41 mils in diameter) that is approximately 10% of the specimen thickness.
The pin sizes currently available are (in inches):

0.0400 (-)
0.0395 (-)
0.0390 (-)
0.0385 (-)
0.0380 (-)
0.0375 (+)
0.0370 (+),

where (-) indicates a diametrical tolerance of (+0.0000 / -0.0002) inches and (+) indicates a diametrical tolerance of (+0.0002 / -0.0000) inches. If the material to be tested is considered soft (such as copper or aluminum), use the pin that is equal to or slightly larger than the calculated size to provide slightly less clearance. If the material is considered hard (such as cold worked 316), use the pin equal to or slightly smaller than the calculated size to provide slightly more clearance. A table has been included on the specimen data sheet for convenience. For consistency, it is a good idea to use the same pin diameter throughout a test series if the majority of the specimens are of a similar thickness.

5.2.8 Using the extended manipulation tweezers, place the pushpin into the guide hole. If it does not slide smoothly into the hole until it makes contact with the specimen, figure out why and fix it before proceeding.

5.2.9 Using the upper fixture handle, transfer the test fixture from the loading area on to the tray and carry the tray to the Instron cabinet. Load the test fixture onto the adapter base of the lower push rod mounted on the crosshead of the Instron. Remove the fixture handle and replace both the handle and tray on the work table.

5.3 Shear Punch Testing

The RCT will observe the initial shear punch test.

The physical testing will be carried out according to the technical work document (SPT-TWD) This document details the procedure for the shear punch testing of irradiated materials at a constant displacement rate.

5.4 Specimen retrieval after testing

5.4.1 After a test is complete, lower the machine crosshead to allow suitable clearance for removal of the test fixture.

5.4.2 Bring the fixture handle and tray back to the test frame and insert the fixture handle into the fixture and move the fixture directly onto the work tray. Transfer the tray and fixture directly to the work area, which is adjacent to the temporary storage area.
5.4.3 Position the test fixture in the holding device and loosen the Allen bolts with the Allen wrench. Do not completely remove the bolts.

N.B. Once the test fixture is split, any shielding that may have been provided by the 304 stainless steel fixture will no longer apply. A safe working distance of 6” must again be adopted from this point.

5.4.4 Remove the pushpin from the fixture with the extended manipulation tweezers and store it for future use.

5.4.5 Remove the upper half of the fixture using the fixture handle, leaving the Allen bolts in the upper half for convenience. It may be necessary to replace the lower fixture handle at this point in order to facilitate this step.

5.4.6 Working with the extended manipulation tweezers, lift the specimen from the fixture and store it in the receptacle from which it came.

**either**

Store the punched specimen in the peanut vial from which it came (which will be held in the copper block) and continue to section 5.4.7.

**or**

In the case that the specimen is to be returned to a specimen pencil, use the extended manipulation tweezers to drop the tested specimen into the specimen funnel in well number 1 of the temporary shielding area. The funnel will guide the specimen into the top of the pencil. Verify visually that this happened with a mirror.

5.4.7 At this time, the blank will be contained within the lower half of the fixture. Remove the tape from the underside of the lower fixture half. If the blank is attached to the tape, then carefully remove it using the extended manipulation tweezers. If the blank is still in the bore of the lower fixture half, pick up and invert the lower fixture half over a glass dish with the fixture handle and carefully push the blank from the die with the 40 mil pushpin. Manipulate the pushpin with the 10” tweezers until the blank falls out into the dish.

5.4.8 Collect the blank with the extended manipulation tweezers and store it in the vial or pencil from which it came as described in section 5.4.6.

*If the blank cannot be found, then contact an RCT immediately for assistance in locating the blank prior to restarting work.*

5.4.9 Once the blank and punched specimen are stored:
either
In the case that a peanut vial is being used, use 12” tweezers to
close the lid of the vial and return it to well number 2 of the
temporary shielding area.

or
In the case that the pencil vials are being used, use the following
guidelines to close and replace the pencil.

1. Remove the specimen funnel with the 10” tweezers. The blank
and punched specimen will remain in the top of the open pencil.
Set it upright on the paper next to the temporary storage area.
2. The pencil lid will still be gripped in the pencil lid remover.
Replace the lid by screwing the lid on tightly, being careful not
to cross thread it.
3. Remove the closed pencil with the lid remover and set it in the
copper block. Use the pencil forceps to hold the pencil and pull
the lid remover from the pencil.
4. Open the top of the mobile cave and insert the pencil in one of
the vacant holes so that the lid is inserted first.
5. Close the lid of the mobile cave while preparing for the next
test.

5.4.10 Repeat test procedure for all remaining specimens. Never work
with more than one specimen at a time. Lock the mobile cave at
the end of the day or when it is not in use.

RADIOLOGICAL CONTROL HOLD POINT: The RCT will perform removable
contamination and radiation surveys of the test fixture, test machine, work area
and equipment used at the end of the day or at the discretion of the RCT.
Appendix 2 – Technical Work Document for the Shear Punch Test

PROCEDURE FOR SHEAR PUNCH TESTING OF IRRADIATED MATERIALS

Technical Work Document (SPT-TWD Rev. #6- June 1998)

This Technical work document (TWD) is specific to the shear punch testing of irradiated materials using the test equipment housed within Lab 5A, Building 326, 300 Area, PNNL. The following sections provide instructions for a series of shear punch tests on irradiated materials. The corresponding Safe Operating Procedure (SOP-SPT-1) and the current Radiological Work Permit (RWP) should be used together with this document. The document is specific to the Instron machine and equipment in Lab 5A. Should the shear punch test be carried out at any other location or on any other equipment than that discussed, the TWD would require revision.

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376-0156
1.0 Before a test series

1.1 Prerequisites
Before commencing a test series, ensure that the set of punches which are used are sharp and that the bore in the lower fixture is in good condition. See Appendix 1 for punch and fixture maintenance. Also ensure that the correct size shims are available and are cleaned of dust.

1.2 Equipment
- Instron test machine with furnace in fume hood (Lab 5A, Building 326, 300 Area)
- Furnace control.
- Instron cross head control (crosshead rate / direction) hereafter referred to as the Machine Control (M/C). This unit controls the power and the crosshead rate and direction. The crosshead rate is controlled by pressing one of the buttons adjacent to the small door on the M/C. The button should become illuminated on its selection (some of the buttons do not light up, but will at least remain depressed). Only one button may be selected at a time. The label indicates that with the gear selector on the Instron in the 'up' position, the corresponding crosshead speed is given in the first column of numbers and with the gear selector in the 'down' position, the crosshead speed is given in the second column. The units are given in inches per minute.
- Instrument racking containing the load cell amplifier, digital voltmeter (DVM) and chart recorder equipment.
- Omega temperature data logger for thermocouples used in furnace control.
- Crosshead gear selector. The lever is situated below and to the right of the Instron hood. In order to select the high-speed ratio, move the lever as far up as it will go. To select the low speed ratio, move the lever as far down as it will go. During a test, the lever will be kept in the 'down' position.
- Digital Transducer Readout (DTR). This unit is used to give an indication of crosshead position. It is located on top of the heater and environment control unit which is situated to the left side of the Instron hood.
- 1000 lbf load cell and load cell amplifier.
- Digital Voltmeter (DVM). This is used to monitor the output from the load cell. It is situated on top of the Instron Instrument racking.
- Chart recorder. HP 7004B X-Y Recorder placed on the shelf of the instrument racking.
- Data acquisition program capable of recording load cell voltage versus time (LabVIEW version 3.1).
- Test fixture and associated remote handling tools specific to job (see SOP-SPT-1).
- Fixture adapter plate. The plate locates and supports the fixture on the push rod of the Instron crosshead.
- Temporary specimen storage unit follower card (see Appendix 2).
- Portable specimen storage cart.
- Specimen data sheets (see Appendix 3).

1.3 Calibration (which components and at what frequency).
- Load cell - annually
- Crosshead rate - beginning of a new series or per cognizant engineer
Load cell and amplifier - beginning of a new series or per cognizant engineer
Federal Micrometer - code No. 999-35-01-004 – annually
Calibrate furnace temperature against the specimen temperature in the fixture.

1.4 Initial preparation for shear punch testing
This section details operation necessary prior to a new series of shear punch testing.

If necessary, remove the strain gauge push rod from the Instron crosshead assembly, which is used in some tests to connect the test assembly to a strain gauge, situated on the crosshead. The push rod runs through the length of the ceramic tube, which is used to support the test fixture in the environmental chamber. If the push rod is present, remove it from the assembly and store it in the hood.

Position the test machine adapter plate on the lower push rod of the crosshead.

Post these instructions on the wall behind the temporary storage area for convenience.

Post the follower card for the temporary storage area on the wall of the Instron M/C adjacent to the temporary storage area (Appendix 2). The follower card will indicate which specimen is in use and which specimens have already been tested and on what date. The reason for such close control of the specimens is that should a test series be interrupted for any reason, then the exact location of all specimens can be determined on returning to the lab.

Collect all the required tooling and equipment as required by the SOP-SPT-1 and by the current RWP for the test.

The radiological worker should be familiar with the layout of the bench top layout. Figure 1 shows a plan of the area. A waste receptacle for the radiological waste is positioned beneath the desktop.

Figure 1 - Plan of work area
2.0 Intron setup

2.1 Power up Intron M/C.

- Move the switch toggle marked ‘Main Power’ into the up position. The light will turn on. The arrow marked on the console points downward to the 'off' position.
- Verify the switch toggle marked ‘Amplidyne’ is in the up position. The arrow marked on the console points downward to the 'off' position.
- Keep the small door that is situated adjacent to the rate control buttons on the M/C 'open' when not in use. This isolates the crosshead motor and prevents inadvertent movement of the crosshead.
- Set the gear lever below the Intron hood to the 'down' position for the low speed operation.

2.2 Setting a reference datum for crosshead movement.

This setup procedure is preformed before the start of a new test series and will establish the starting point for the crosshead. This will provide an efficient method to return the crosshead to a standard position for removal of the test specimen. (ALARA)

- Power up the M/C as described in section 2.1.
- Change the gear lever to the ‘up’ position for the fast ratio (see section 1.2 for location).
- Set the fastest speed on the M/C panel, i.e. 2” / min., by depressing the appropriate button on the Intron M/C.
- Close the door on the M/C panel to enable the crosshead.
- Using the up, stop and down controls on the M/C position the crosshead such that with the fixture adapter in position, the distance between the upper push rod of the Intron and the adapter plate rim is approximately 2.5 inches. This will allow adequate clearance for the positioning and removal of the test fixture.
- Fine-tune the crosshead position by selecting a lower crosshead speed (such as 0.2”/min.) and moving the crosshead up and down in the same manner.
- Note the reading of the DTR in order to provide a reference point for future testing. The DTR may be zeroed or may be used as the reference point prior to heat up for testing.
- Return the gear lever to the 'down' position.
3.0 Daily testing operations

3.1 Preparation for testing

- Start one data sheet for each specimen that is to be tested in the current session. Record specimen identity, the date and initial it. See Appendix 3 for a copy of the data sheet. If the specimen is to be tested at temperature, be sure to record the conditions in the relevant box.
- A separate, labeled vial is allocated for each specimen. It will be returned to the same vial after testing.
- Check that the Geiger-Mueller Counter (GM) is switched on and is operational. Conduct a battery check and test the meter against its source.
- Check that the airflow into the hood is adequate. A telltale is attached to the hood window as a visual aid. If the rate of airflow is insufficient, then stop work and call a Radiological Control Technician (RCT). Turn on the hood light. It is situated on the front panel of the hood and is labeled PNL YE CB 31.
- Cut a number of lengths of masking tape that can be used on the bench top to record the specimen dimensions. Place the tape near the bench top for use.
- Set the outer dial of the full-scale deflection on the Load Cell Amplifier to 100. This corresponds to a 1000 lbf load cell. The value is selected by turning the outer dial until the number desired (100) is visible above the black sector marked 'center zero' on the panel. The black sector will obscure the 200 mark on the dial.
- Set the inner dial of the full-scale deflection (FSD) of the Load Cell Amplifier to the position marked 20. This corresponds to a FSD of 200 lbf. Always ensure that the outer dial has been set prior to the inner, since the position of the former effects the reading of the latter.
- Select the chart speed of 20 sec/inch on the X-Y plotter.

3.2 Setting up test parameters, data acquisition and chart recorder

- If necessary, turn on computer and load LabVIEW version 3.1 for the Macintosh. This is the data-logging program.
- In the file menu of desktop finder, open the 'Shear Punch Test (load-time)' LabVIEW document file which is located at: Desktop / cobra 210i / users / fusion.
- Select the 'hand' icon for data input mode. It will be highlighted in black.
- Ensure the data logging status of the program is on 'no log', the load range is on 200 lbf full scale deflection, and the sampling frequency is on 0.1 seconds per point.
- The deflection range setting is not used. It need not be changed from the default value of 1.
- Fill in the test data in the 'run description' window. Record the information in the window by typing in the following exactly as shown:

Specimen ID = (A combination of Material/Specimen code-test number which is relevant to the current series) [e.g. Fe12Cr5N73-1]
Full scale load = 200 lbf
Chart speed = 20 sec/inch
Cross head speed = 0.005 in/min.
(Temperature / environment conditions) [e.g. Room Temperature (RT)]
(Date and tester initials)
(Hit ‘Enter’ for seventh line of data, no entry)
• Click on the start icon. The symbol for this is an arrow pointing to the right. The prompts ask for a file name to which to write the data.
• Select the relevant folder or create a new folder for the test series within the ‘shear punch data’ folder, which is found at Desktop/ Cobra/ users/ fusion/ shear punch data and create a new file name for logging the data. The file name will be the same as the specimen ID that was entered previously. No file extension is required in the name.
• Click O.K. on the following prompts and follow back to LabVIEW. The data logging is now primed for a test.

3.3 Loading the specimen and fixture for testing

In this section, an open bullet (o) indicates when the tester is working within the zone bound by the radiological restrictions imposed by the RWP. With the exception of the test fixture and its associated handling tool, no items may be removed from the Instron hood or loading area without obtaining a release survey preformed by an RCT.

- Don the required protective clothing and dosimetry per the RWP if you are not already wearing them from a previous test (i.e., in the event of a re-load). Rather than removing the protective clothing after each test, a second pair of gloves may be worn atop the first, taped, pair. After exiting the posted radiological material areas, remove the second pair of gloves and perform a personal contamination survey.
- Optional: If testing at room temperature, seal the hole of the underside of the lower fixture with scotch tape in order to prevent the punched blank from being separated from the test fixture. Make sure that the tab extends beyond the base of the fixture to facilitate its removal after the test is complete. Use the remote-handling tool for manipulating the fixture.
- Retrieve the specimen from the temporary storage according to the SOP instructions. See figure 2.
- Measure the specimen thickness (as outlined in the SOP) and record them on the masking tape located in the bench top radiological zone. Select the relevant shim size and punch pin diameter as will be indicated on the specimen data sheet.
- Load the specimen in the fixture per SOP–SPT-1.
Fig. 2. Bench top used for loading and preparing shear punch test fixture.

- Per SOP-SPT-1, use the remote handling tools and tray to position the loaded fixture on the adapter plate within the Instron environmental chamber. See figure 3a and 3b.

Fig. 3a. Instron hood and environmental chamber

Fig. 3b. Shear punch test fixture in position
APPENDIX 2

- Doff the outer pair of gloves and dispose of in the waste receptacle. Perform a radiological contamination control survey of hands, arms, and upper torso per the RWP.
- The worker is now free to move between the temperature controller, computer, chart recorder and Instron M/C while still wearing the lab coat and taped gloves.
- Record the change in the location of the specimen on the Temporary Storage Unit Follower Card. Use the date of the test to fill in the 'in use' column.

3.4 Fixture loading and positioning

- The door to the environmental chamber will need to be open in order to view the relative position of the fixture and the upper push rod of the Instron test rig.
- Throughout the test only the low gear of the Instron will be employed. For the approach to the specimen, ensure the gear lever is still in the 'down' position. This will eliminate any chance of overloading the specimen when trying to position it.
- Close the door on the front of the M/C in order to allow crosshead operation.
- Using the 'up' and 'stop' controls on the M/C, move the crosshead so that the push pin of the fixture assembly is almost in contact with the upper push rod of the test machine. A faster crosshead speed such as 0.2 " / min. may be used for this first stage (gear lever is down and top button on the M/C is depressed). If the specimen is to be tested at an elevated temperature, position the top of the push pin ~.1 from contact with the upper push rod.
- Turn on the chart recorder. Depress the Power, Servo, and Pen buttons.
- Place the chart paper on the chart recorder and press the chart button to secure the chart paper to the chart recorder.
- Record the specimen thickness measurements on the Shear Punch Data Test Sheet.

⇒ If the specimen is to be tested at an elevated temperature, proceed to section 3.5.

- On the chart recorder, set Y, to .1 volts per inch. This is done in order to aid in the 'positioning' of the specimen without over-loading the specimen.
- Set the crosshead speed control on the M/C to 0.05 " / min. so as not to load the specimen excessively on contact, i.e., second button down on the M/C panel is depressed and the gear lever is still in the down position.
- Advance the crosshead using the 'up' and 'stop' controls until the slightest load is registered on the load cell. Watch the pen on the chart and wait for it to start moving before stopping the crosshead.
- Back off the crosshead a fraction using the 'down' and then 'stop' in order to unload the specimen.
- Prepare the chart paper and chart recorder for testing.
  Record on the chart paper the following information replacing each blank with the appropriate information:
  Specimen ID: __________
  FSL = 200lb
  Crosshead speed = 0.005in/min
  Chart:
  \[X = \_\text{sec/in}\]
  \[Y = \_\text{volts/inch}\]
  (Temperature/environment conditions)
  (Date/ Initials)
If the specimen is to be tested at room temperature, skip to section 3.7.

3.5 Preparation for testing a specimen at temperature (≤ 200°C)

- If the specimen is to be tested at temperature, then it is necessary to leave a sufficient distance between the pushpin and crosshead to allow for the thermal expansion of the fixture and test machine during the heating up procedure. Set the crosshead speed control to 0.05 "/ min. The top of the push pin should be ~.1 to ~.2 inches away from contact with the upper push rod. Verify the position of the push pin and push rod.

- Put on a second pair of gloves so that the environmental chamber door can be shut.

  - In order to close the door, the front of the hood must first be moved on its hangings by pulling it backward from the top of the open window. This allows enough clearance for the environmental chamber door to be swung around and closed. Restraining guide rods ensure the door cannot swing around until it is clear of the furnace. Pull both sides of the door until it is clear of the guide rods and then swing the door through 180 degrees. The door can be then pushed backwards such that the opposite end of the same guide pins locate into the same receiving guide holes. Secure all eight door latches (see figure 4).

  - Doff the outer pair of gloves and dispose of them in the waste receptacle. Conduct a radiological contamination control survey of hands, arms, and the upper torso per the RWP. If desired, all protective clothing may be removed until after completion of the heating, testing, and cooldown of the specimen. Perform a whole body survey if leaving the vicinity of the test area.

- Continue with the heating procedure (section 3.6).

Figure 4 -- Furnace door.
Note: The specimen testing may be performed at elevated temperatures by reaching that test temperature by two different methods. The first method addressed in 3.6 to 3.7 is by temperature stabilization. The second method addressed in 3.8 to 3.9 is by testing the specimen as it is reaching the test temperature after setting the furnace temperature to a higher temperature set point. The cognizant engineer will determine the method of specimen testing at elevated temperatures.

### 3.6 Heating Procedure for shear punch test (stabilization)

**General Notes**

A. The furnace "CONTROL POWER" is always left on (see Fig. 5), however, it is briefly turned off for one step in the heating/cooling procedure. If the furnace controller is off, find out why before continuing.

B. No environmental control is included in this procedure which was designed for tests at temperatures of 200°C or less. If environmental control is needed for testing at higher temperatures, the procedure will require modification.

C. If anything unusual happens during the heating procedure, turn off the "FURNACE POWER" (see Fig. 5), and resolve the problem before continuing.

#### 3.6.1. Turn on the ethylene glycol (EG) cooling system.

A. Referring to Fig. 6, open the EG valves on the back wall behind the hood. These valves are designated C-1 and C-2.

B. Push the "ON" button on the back wall that activates the EG pump. The EG flow rate is monitored by a flow meter on the back wall. The flow rate must be greater than 15 GPM for testing to proceed.

C. Referring to Fig. 5, all the "ALARM CONDITION" lights on the furnace controller will turn off shortly after completion of steps 1A and 1B.
Figure 5 -- Main furnace controls.

- Overtemp. thermocouple keypad
- Upper display
- Lower display
- Alarm silence
- Control thermocouple keypad
- Furnace power
- Control power
3.6.2. Turn on the furnace power. Refer to Fig. 5.

A. Observe the display on the control thermocouple controller. Verify that the controller is in manual mode, and that it is also in hold mode. "MAN" on the lower display indicates manual mode, whereas "H" in the upper display indicates hold mode.

B. If the controller is not in manual mode, press the "MAN/AUTO" key.

C. If the controller is not in hold mode, press the "RUN/HOLD" key.
D. Change the Set Point to 20:
Press the “LOWR DISP” key until “SP” is shown on the lower display.
Use the orange keys to adjust the set point (SP) to 20 if not already on 20.

E. Change the Output to 0.0:
Press the “LOWR DISP” key until “OUTPUT” is shown on the lower display.

F. Turn on the "FURNACE POWER".

3.6.3 Setting the overtemperature
Set the overtemperature to 25°C above the "Set Point Temperature". See Table 1. Refer to the overtemperature thermocouple keypad shown in Fig. 5.
A. Press the blue "DISP" key until "SP" is displayed.
B. Use the orange arrow keys to adjust the overtemperature. To accelerate the temperature adjustment, press the arrow key that sends the temperature in the desired direction. While still holding that arrow key, press the other arrow key. This will cause the 10's digit to change rather than the 1’s digit. Pressing the arrow key again causes the 1000's digit to change.
C. To exit, press the "DISP" key again.

3.6.4 Setting the ramp time and thermocouple temperature
Set the ramp time and control thermocouple (TC) temperature. Refer to the control thermocouple keypad shown in Fig. 5. The control TC is used to control the temperature of the specimen, however the control TC measures the ambient temperature of the furnace. This temperature is not equal to the specimen temperature, but it is linearly related to the specimen temperature as shown in Table 1.
A. Press the blue "SET UP" key until "SP RAMP" shows on the upper display.
B. Press the blue "FUNC" key until "TIME MIN" shows on the lower display.
C. Set the ramp time to 20 minutes using the orange keys.
D. Press the blue "FUNC" key until "FINAL SP" shows on the lower display.
E. Use the orange keys to set the appropriate value, which will be determined by the desired test temperature and Table 1.
F. Press the blue "LOWR DISP" key. The digital display should, among other things, have "H" listed in the upper display and "MAN" listed in the lower display.
Table 1 - Relation between desired specimen temperature and required set point temperature.

<table>
<thead>
<tr>
<th>Desired Specimen Temp. [°C]</th>
<th>Target base Temp. [°C]</th>
<th>Approx. Set Point Temp. [°C]</th>
</tr>
</thead>
<tbody>
<tr>
<td>20</td>
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<td>26</td>
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<td>190</td>
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</tbody>
</table>

From calibrations performed 2/98. For shear punch testing in test frame located in 326/5A.

\[
T_{\text{set point}} = 0.758T_{\text{specimen}} + 10.5 \text{ [°C]}, \quad T_{\text{base}} = 0.94T_{\text{specimen}} + 1.905 \text{ [°C]}
\]

12/9/97 MBT
3.6.5 Preparing the temperature data logger

The data logger will be used to record the temperature of the control TC. This is channel 2 on the data logger.

A. Ensure there is sufficient paper left on the roll (requires ~25 in. per test).
B. If not already turned on, turn on the data logger and the multiplexor on which the data logger is sitting. When turned on, the data logger will print out the current settings. If it's already on, to see the current settings, press "arrow" key, and then "LIST". An audible beep should be heard after each key is pressed. No beep indicates the key was not pressed correctly. The datalogger should be set to read K type thermocouples and measure temperature in °C. The low limit should be less than room temperature and the high limit should be greater than 250°C.
C. Set the logging interval to five minute or per cognizant engineer. For example, press "5", "0", "0", "arrow" key, "INTERVAL". This sets the logging interval to 5 minutes and 0 seconds.
D. Set the data logger to read the temperature of the first four TCs. TC1 reads the coolant temperature, and TC2 goes to the controller, TC3 goes to the over-temperature controller and TC4 reads the test fixture base/adapter temperature. Press "4", "arrow" key, and then "CHANNELS". The data logger will now display the temperatures of TC1 to TC4 in five-second intervals.

3.6.6 Bringing the specimen up to temperature

Bring the specimen up to temperature using the control thermocouple keypad.
A. Press the blue "MAN/AUTO" key. "A" should be displayed in the lower display.
B. Press the blue "RUN/HOLD" key. "R" will be displayed on the upper display, and the controller will begin ramping the furnace up to the specified control TC temperature.

3.6.7 The specimen temperature will stabilize at the desired test temperature in no more than 2 hours after completing step 3.6.6. Testing may begin at that time. The user need not be present during this time, but it is advisable that the operator performs a progress check every half-hour.

⇒ Once the temperature is stabilized or if the test plan specifies otherwise, proceed to section 3.7 to conduct the test.

3.7 Running a test (Stabilized temperature)

After loading the fixture and following the heating procedure (when applicable), the test can now be completed. Transfer the specimen thickness information to the Shear Punch Test Data Sheet.

- Verify the load cell amplifier is set to 200 lbf full scale deflection.
Select a crosshead speed of 0.005 "/ min. in preparation for the test, i.e., the gear lever is down and the sixth button down on the M/C is depressed.

Zero the load cell voltage of the DVM, which displays the load cell voltage by adjusting the balance of the load cell amplifier. Do not use the zero control of the DVM, but instead use the 'fine' and, if necessary, the 'medium' balance control on the load cell amplifier, which is contained on the instrument racking.

For each subsequent test using the same pushpin, the DVM will almost always read zero when the Instron push rod is in contact with the pushpin. This can be used as a guide when 'locating' future specimens.

Prepare the chart recorder:

Select the Y range (for example solution annealed 304 samples, Y would be set on 1 volt/inch).

Remove the cap from the chart pen.

Adjust the X and Y zeros so that the pen is located in the bottom left corner of the chart.

Ensure the chart pen is in the down position and that the servo button is depressed.

Begin recording on the chart by depressing the X 'Start' button.

Set the data acquisition status to 'logging' - data is now being recorded. If data are being taken, the display will vary with the DVM readout. The DVM tends to vary up and down by a few milivolts with time.

Press 'up' on the Instron M/C panel.

Run the test until failure has occurred and the load has dropped and stabilized unless otherwise indicated in the test matrix. If it is obvious during a test that a load of 200 lbf is going to be exceeded, then change the scale of the chart recorder to 500 lbf full scale deflection by turning the inner dial of the FSD of the load cell amplifier to 50.

Stop the crosshead movement by pressing 'stop' on the Instron M/C.

Stop the data logging by setting the status to 'stop'.

Stop the chart recorder by pressing the 'reset' button.

Return the crosshead to the reference position by using the 'down' control on a high speed setting, such as 0.2"/min. The reference position is located where the DTR reads zero.

Open the door of the Instron M/C panel to prevent any further crosshead movement.

The LabVIEW program displays a prompt. Click O.K. on the dialogue box and then edit the data window to get ready for the next test if a subsequent test is desired. Only the specimen ID should need changing at this point. After editing the data window click on start again, follow the prompts and assign a new file name corresponding to the next specimen to be tested, click on the O.K. to get back to LabVIEW and set the data logging status to 'no log'.

Before unloading the tested specimen, note its intended storage location on the storage area unit follower card.
3.8 Heating Procedure for shear punch test (Transient)

General Notes
The following section provides an alternate procedure to reduce the time required to heat a specimen to test temperature without having to wait for the temperature in the furnace to stabilize. This section of the procedure will allow completion of a shear punch test after 30 to 85 minutes instead of the 2+ hours it would take for the specimen temperature to stabilize. This alternate method is done by setting the set point to a higher temperature than the test temperature and testing the specimen as it reaches/passes through the test temperature. The longer heat-up time required for the 164°C, is due because of a requirement to keep the furnace from exceeding 250°C while being operated in an air atmosphere. During heat up for testing at 164°C the over temperature thermocouple registers ~225°C with a set point of 180°C and a ramp time of 30 minutes.

A. The furnace "CONTROL POWER" is always left on (see Fig. 5, page 13), however, it is briefly turned off for one step in the heating/cooling procedure. If the furnace controller is off, find out why before continuing.

B. This procedure does not address environmental control or the testing of specimens at temperatures greater than 200°C. If environmental control or higher test temperatures are needed this procedure will require modification.

C. If anything unusual happens during operation of the furnace, turn off the "FURNACE POWER" (see Fig. 5, page 13), and resolve the problem before continuing.

3.8.1. Turn on the ethylene glycol (EG) cooling system.

A. Referring to Fig. 6 (page 14), open the EG valves on the back wall behind the hood. These valves are designated C-1 and C-2.

B. Push the "ON" button on the back wall that activates the EG pump. The EG flow rate is monitored by a flow meter on the back wall. The flow rate must be greater than 15 GPM for testing to proceed.

C. Referring to Fig. 5 (page 13), all the "ALARM CONDITION" lights on the furnace controller will turn off shortly after completion of steps 1A and 1B.

3.8.2. Turn on the furnace power. Refer to Fig. 5 (page 13).

A. Observe the display on the control thermocouple controller. Verify that the controller is in manual mode, and that it is also in hold mode. "MAN" on the lower display indicates manual mode, whereas "H" in the upper display indicates hold mode.

B. If the controller is not in manual mode, press the "MAN/AUTO" key.

C. If the controller is not in hold mode, press the "RUN/HOLD" key.

D. Change the Set Point to 20:
Press the "LOWR DISP" key until "SP" is shown on the lower display. Use the orange keys to adjust the set point (SP) to 20 if not already on 20.

E. Change the Output to 0.0:
Press the "LOWR DISP" key until "OUTPUT" is shown on the lower display.
Use the orange keys to adjust the output to 0.0.

F. Press the button "FURNACE POWER".

3.8.3 Setting the over-temperature
Set the over-temperature to 250°C. Refer to the over-temperature thermocouple keypad shown in Fig. 5.
A. Press the blue "DISP" key until "SP" is displayed.
B. Use the orange arrow keys to adjust the over-temperature. To accelerate the temperature adjustment, press the arrow key that sends the temperature in the desired direction. While still holding that arrow key, press the other arrow key. This will cause the 10's digit to change rather than the 1's digit. Pressing the arrow key again causes the 1000's digit to change.
C. To exit, press the "DISP" key again.

3.8.4 Setting the ramp time and thermocouple temperature
Set the ramp time and control thermocouple (TC) temperature. Refer to Table 2 (page 21) for parameters for testing for each goal temperature.
A. Press the blue "SET UP" key until "SP RAMP" shows on the upper display.
B. Press the blue "FUNC" key until "TIME MIN" shows on the lower display.
C. Set the ramp time as listed in Table 2 using the orange keys.
D. Press the blue "FUNC" key until "FINAL SP" shows on the lower display.
E. Use the orange keys to set the appropriate set point temperature as listed in Table 2 (page 21).
F. Press the blue "LOWR DISP" key. The digital display should, among other things, have "H" listed in the upper display and "MAN" listed in the lower display.
Table 2. Temperature Control for Tests at Elevated Temperature

<table>
<thead>
<tr>
<th>TEMPERATURE CONTROL SETTINGS AND PROCEDURES</th>
<th>ANTICIPATED TEMPERATURE HISTORY IMMEDIATELY BEFORE AND AFTER START OF TEST</th>
</tr>
</thead>
<tbody>
<tr>
<td>Goal Test Temperature (°C)</td>
<td>Temperature Ramp Time</td>
</tr>
<tr>
<td>---------------------------------------------</td>
<td>--------------------------</td>
</tr>
<tr>
<td>50°C</td>
<td>20 minutes</td>
</tr>
<tr>
<td>• Start to bring the crosshead up to contact the push pin when the specimen temperature is ~43° (reference temperature TC_{4}=~43°).</td>
<td></td>
</tr>
<tr>
<td>• Establish contact with specimen before 49° by pre-loading the specimen and then backing the crosshead off of the specimen.</td>
<td></td>
</tr>
<tr>
<td>• At TC_{4}=49° turn furnace off and start test.</td>
<td></td>
</tr>
<tr>
<td>110°C</td>
<td>30 minutes</td>
</tr>
<tr>
<td>• Start to bring the crosshead up to contact the push pin when the specimen temperature is ~97° (reference temperature TC_{4}=~97°).</td>
<td></td>
</tr>
<tr>
<td>• Establish contact with specimen at TC_{4}=107° by pre-loading the specimen and then backing the crosshead off of the specimen.</td>
<td></td>
</tr>
<tr>
<td>• At TC_{4}=107° turn furnace off and start test.</td>
<td></td>
</tr>
<tr>
<td>164°C</td>
<td>30 minutes</td>
</tr>
<tr>
<td>Furnace kept on</td>
<td></td>
</tr>
<tr>
<td>• Start to bring the crosshead up to contact the push pin when the specimen temperature is ~158° (reference temperature TC_{4}=~151°).</td>
<td></td>
</tr>
<tr>
<td>• Establish contact with specimen at TC_{4}=155° by pre-loading the specimen and then backing the crosshead off of the specimen.</td>
<td></td>
</tr>
<tr>
<td>• At TC_{4}=155° start test.</td>
<td></td>
</tr>
</tbody>
</table>

*Up to 10 minutes after test start
3.8.5 Preparing the temperature data logger

The data logger will be used to record the temperatures of the four TCs. Verify that TC₁ = Coolant, TC₂ = Control, TC₃ = Overtemp, and TC₄ = Reference by inspecting the connections found adjacent to the datalogger.

A. Ensure there is sufficient paper left on the roll (requires ~60 inches for testing at 50°C and 110°C and ~120 inches for testing at 164°C).

B. If not already turned on, turn on the data logger and the multiplexor on which the data logger is sitting. When turned on, the data logger will print out the current settings. If it’s already on, to see the current settings, press "arrow" key, and then "LIST". An audible beep should be heard after each key is pressed. No beep indicates the key was not pressed correctly. The datalogger should be set to read K type thermocouples and measure temperature in °C. The low limit should be less than room temperature and the high limit should be greater than 250°C.

C. Set the logging interval to half a minute. Press “0”, “3”, “0”, "arrow" key, "INTERVAL". This sets the logging interval to 30 seconds.

D. Set the data logger to read the temperature of the first four TCs. TC₁ reads the coolant temperature, and TC₂ goes to the controller, TC₃ goes to the over-temperature controller and TC₄ reads the test fixture base/adapter temperature. Press "4", "arrow" key, and then "CHANNELS". The data logger will now display the temperatures of TC₁ to TC₄ in five-second intervals.

3.8.6 Bring the specimen up to temperature and test as the specimen reaches the desired temperature.

Turn on the furnace and begin the heat cycle.

A. Press the blue “MAN/AUTO” key. “A” should be displayed in the lower display.

B. Press the blue “RUN/HOLD” key. “R” will be displayed on the upper display, and the controller will begin ramping the furnace temperature up to the specified control TC temperature. Record “Heat up start time” on the Shear Punch Test Data Sheet.

C. As the temperature approaches the goal temperature, move the crosshead up until a small load is detected on the specimen. (See Table 2 for instructions on when to start pre-loading.)

1. The set the crosshead speed on the M/C to .05”/min and the set the chart recorder Y position to .1v/cm.

2. Advance the crosshead using the “up” and “stop” controls until a small load is registered on the load cell. Watch the pen on the chart and wait for it to start moving before stopping the crosshead.

3. Lower the crosshead using the “down” and then “stop” in order to unload the specimen. Allow some small distance for expansion as the specimen continues to heat.
4. Mark the data logger tape with “I---” to note the time in which the adjustment to the crosshead was made.

3.9 Running a test (Transient Temperature)

After loading the fixture and following the heating procedure (when applicable), the test can now be performed. Transfer the specimen thickness information to the Shear Punch Test Data Sheet.

- Verify that the load cell amplifier is set to 200 lbf. full scale deflection.
- Select a crosshead speed of 0.005 ″/min. in preparation for the test, i.e., the gear lever is down and the sixth button down on the M/C is depressed.
- Zero the load cell voltage of the DVM, which displays the load cell voltage by adjusting the balance of the load cell amplifier. Do not use the zero control of the DVM, but instead use the 'fine' and, if necessary, the 'medium' balance control on the load cell amplifier, which is contained on the instrument racking.
- For each subsequent test using the same pushpin, the DVM will almost always read zero when the Instron push rod is in contact with the pushpin. This can be used as a guide when ‘locating’ future specimens.
- Prepare the chart recorder:
  - Select the Y range (for example solution annealed 304 samples, Y would be set on 1 volt/inch)
  - Adjust the X and Y zeros so that the pen is located in the bottom left corner of the chart. Follow the instructions of Table 2 (page 21); start the test at the reference temperature listed in instruction block and turn off the furnace as instructed.
  - Begin recording on the chart by depressing the X ‘Start’ button.
  - Ensure that X is set to 20 sec/in and Y is set to 1 volt/inch.
  - Set the data acquisition status to ‘logging’ - data is now being recorded. If data are being taken, the display will vary with the DVM readout. The DVM tends to vary up and down by a few millivolts with time.
  - Press ‘up’ on the Instron M/C panel.
  - Mark the data logger tape with “2---” to note the time at the start of the test.
  - Run the test until failure has occurred and the load has dropped and stabilized unless otherwise indicated in the test matrix. If it is obvious during a test that a load of 200 lbf is going to be exceeded, then change the scale of the chart recorder to 500 lbf full scale deflection by turning the inner dial of the FSD of the load cell amplifier to 50.
  - Stop the crosshead movement by pressing ‘stop’ on the Instron M/C.
  - Stop the data logging by setting the status to ‘stop’.
  - Stop the chart recorder by pressing the ‘reset’ and ‘servo’ buttons. Mark the data logger tape with “3---” to note the time at the end of the test.
  - Return the crosshead to the reference position by using the ‘down’ control on a high speed setting, such as 0.2″/min. The reference position is located where the DTR reads zero.
  - Open the door of the Instron M/C panel to prevent any further crosshead movement.
  - The LabVIEW program displays a prompt. Click O.K. on the dialogue box and then edit the data window to get ready for the next test if a subsequent test is desired. Only the specimen ID should need changing at this point. After editing the data window click on start again, follow the prompts and assign a new file name.
corresponding to the next specimen to be tested, click on the O.K. to get back to LabVIEW and set the data logging status to 'no log'.

- Before unloading the tested specimen, note its intended storage location on the storage area follower card.

⇒ If the specimen was tested at temperature, proceed to the cooling procedure (section 3.10).

⇒ If the specimen was tested at room temperature, proceed directly to section 3.11 for specimen unloading.

3.10 Cooling the specimen and fixture

- To bring the specimen back to room temperature after the test is completed:
  A. Place the furnace controller into manual mode by pressing the blue "MAN/AUTO" key.
  B. Place the furnace controller in hold mode by pressing the "RUN/HOLD" key.
  C. Press the "LOWR DISP" key until "SP" shows in the lower display. Using the orange keys, reduce the SP temperature to zero.
  D. Turn off the "FURNACE POWER".
  E. Stop the data logger by simply turning it off. (The data logger will remember its program if it is turned off.)
  F. Remove the paper strip from the data logger and record the time and temperature information of the test on the Shear Punch Test Data Sheet.
  G. Using the reference graphs in Appendix 4, record the specimen temperature details on the Shear Punch Test Data Sheet.

- To minimize dose, allow the fixture to cool in the furnace to a temperature less than 40°C before handling.
- Note: Generally after heating and testing the specimen, the operator may leave the zone and should follow proper doffing and surveying procedures. Upon completion of the cool down cycle, the operator shall again don the appropriate apparel and dosimeters per the RWP.

o In order to open the door, the front of the hood must first be moved on its hangings by pulling it backward from the top of the open window. This allows enough clearance for the environmental chamber door to be swung around and closed. Open all eight door latches (see figure 4). Restraining guide rods ensure the door cannot swing around until it is clear of the furnace. Pull both sides of the door until it is clear of the guide rods and then swing the door through 180 degrees. The door can be then pushed backwards such that the opposite end of the same guide pins locate into the same receiving guide holes and the door is held clear of the oven.

⇒ Continue with the unloading procedure (section 3.9). There is no need to doff the outer pair of gloves at this point since they will be required for the unloading procedure.

3.11 Unloading the test fixture and specimen

As previously discussed, an open bullet (o) indicates when the tester is wearing protective clothing. With the exception of the test fixture and its associated handling tools, no items may be removed from the Instron hood or loading area without consent
Items not resident to either the Instron hood or loading area may be brought into these areas, but cannot be removed without concurrence of an RCT.

- Record on the specimen storage follower card the date in the 'test complete' column. Any comments relevant to the test can be filled in at this point on the data sheet.
- Put on a second pair of gloves if you are not already wearing them after the cooling procedure.
- Retrieve the remote-handling tool, remembering to survey the tips of it for contamination before transferring it to the Instron hood and locating it in the fixture.
- Retrieve the fixture with the tray by the correct procedure as described by the SOP.
- For optional use of scotch tape at room temperature: Using the 12" tweezers, carefully remove the piece of scotch tape, which was used to seal the bore of the lower fixture. If the specimen blank is attached to the piece of tape, then carefully remove it with the extended manipulation tweezers and replace it in the correct vial. Dispose of the tape in the waste receptacle allocated for the tape and discarded gloves.
- Unload and store the tested specimen in accordance with the SOP in a labeled peanut vial or pencil vial and return the vial to the temporary storage area.

⇒ If another test is to be carried out, then proceed to instructions for loading the next specimen (Section 3.12).

⇒ If no additional tests will be performed, doff the outer pair of gloves and dispose of them in the waste receptacle. Conduct a radiological contamination control survey of hands, face and the front of the lab coat as directed by the RWP prior to exiting the radiological area and proceed to Section 4.0 and finish for the day.

3.12 Reloading for an additional test
This section should only be followed should time allow for a subsequent test. The tester will already be wearing the required protective clothing, i.e., having just completed the unloading procedure (Section 3.9). Note the use of open bullets to indicate when protective clothing is being worn.

- Before doffing protective clothing and after safely storing the previous tested specimen, the next specimen may be loaded. Go back and repeat the loading procedure from the beginning of section 3.3. When changes are made to the specimen storage follower card, both the unloaded specimen and the newly loaded specimen will have to be accounted for. The new specimen information will have already been entered into the LabVIEW window at the end of the last test (Section 3.7).

4.0 Finishing the test session
- Having completed the last test of the day/session, ensure that all specimens are in the correct vial or pencil and are stored safely.
- Check that all the shims and punch used in the previous test(s) is free of dirt and debris. If they are not then they must be cleaned prior to doffing the protective clothing and finishing for the day. Some wipes will be available in the work area.
Used wipes must be disposed of in the radiological waste receptacle along with the gloves etc.

- Tidy up the work area, then conduct a personal contamination survey.
- If the mobile cave has been used, make sure it is locked.
- Doff the outer pair of gloves and dispose of in the waste receptacle. Conduct a radiological contamination control survey of hands, face and the front of the lab coat as directed by the RWP prior to removing the remaining pair of gloves and lab coat. Conduct one final personal contamination control survey before exiting the radiological area.

- Note the changes to the location of the specimen last tested on the temporary storage follower card.
- Turn off the power to the Instron in the reverse order that it was powered up: Open the door on the M/C and move both the Amplidyne and main power toggle switches to their down positions.
- If the furnace has been used, it must be reset and left on for the next time.
  A. Turn off the "CONTROL POWER".
  B. Turn off the EG pump.
  C. Close coolant valves C-1 and C-2 (Fig. 6).
  D. Remember that the control power is always left on. Turn on the "CONTROL POWER" by simultaneously pressing "CONTROL POWER" and "ALARM SILENCE". Failing to press "ALARM SILENCE" will cause an alarm to sound. If the alarm goes off, press the "ALARM SILENCE" button.
- Turn off the hood light.
- Turn off the GM(β/γ) counter.
- Back up recorded data from the day’s testing on a disc and store it in a different location than Lab 5A.
- Leave the computer on for the next session.
TWD - Appendix 1 - Maintenance of the push pins.

Testing on controls has shown that maintaining a sharp punch edge and a square edge to the bore of the lower fixture half can reduce the scatter of the effective shear yield strength.

The punches used in the shear punch test were made from 2 in. stock gauge pins which are available from Vermont gage, Spokane, WA. The gauge pins are available for diameters ranging from 40 to 37 thousandths of an inch in increments of 0.5 thousandths of an inch. Two punches can be produced from each gauge pin. Each pin is to be no longer than 6/8 in. to prevent it from buckling under the applied load.

One end of the pin is polished in a two-stage operation to give a square edge for the punching operation. The top half of an old test fixture was used to hold the punch perpendicular to the abrasive surface. A small weight was applied to the top of the punch and then the punch and fixture were polished in a figure ‘eight’ motion on a glass surface with a 6 jim diamond paste for 5 minutes. The fixture and punch were cleansed thoroughly with acetone before the final polish was carried out with a 1 \( \mu \text{m} \) diamond paste for another five minutes.

Figure 7 shows the condition of the punch as received alongside a punch polished to a 1 \( \mu \text{m} \) finish. The radius of the sharpened punch was estimated at less than 0.5 of a thousandth of an inch when using a shadowgraph. The third picture shows the condition of the punch after approximately 30 tests. The edge of the punch is slightly rounded.

It is also important to ensure that the bore of the lower fixture half, which supports the TEM disc and receives the push pin, is kept in good condition. The best way to achieve this is to replace the bushing of the lower fixture half from time to time. After 300 - 400 tests is suffice, depending on the hardness of the material being tested.

First ensure that the lower fixture half is free of contamination. The old bushing may be removed while the fixture is still in the radiological materials area (RMA) of the bench top. With an RCT’s approval, take the cleaned fixture from the RMA and remove the three location dowels which are on the top face of the fixture. The new bushing should be pushed into the fixture until it butts up against the shoulder inside the fixture’s central bore. The bushing can be turned down on a lathe to within 0.005 in. of the
surface of the fixture. In order to obtain a clean, square and level edge of the bushing, the final 5 mil is removed by polishing the inverted fixture and bushing on a glass surface with diamond paste in the same way that the push pins were polished. Figure 5a and b show the condition of the bore before and after testing.

Figure 8a. Bore of lower fixture half after replacement and polishing.  
Figure 8b. Bore of lower fixture half after ~ 500 tests.
### Appendix 3 - Shear Punch Test Data Sheet

#### Shear Punch Test Data Sheet

<table>
<thead>
<tr>
<th>Material data</th>
<th>SHIM SIZE *</th>
</tr>
</thead>
<tbody>
<tr>
<td>Material type:</td>
<td>shim thickness, s (mil)</td>
</tr>
<tr>
<td>Thickness, t₁,₂,₃ (mil)</td>
<td>(s ≤ t_max + 8.0)**</td>
</tr>
<tr>
<td>Specimen ID:</td>
<td>Code Check:</td>
</tr>
<tr>
<td>Date:</td>
<td>Avg. t (mil)</td>
</tr>
<tr>
<td>Tester initials:</td>
<td>&lt; 8.0 mils – don’t test!</td>
</tr>
<tr>
<td>Shear lip? (y/ n)</td>
<td>8.0 &lt; t ≤ 9.0</td>
</tr>
<tr>
<td>9.0 &lt; t ≤ 10.0</td>
<td>19</td>
</tr>
<tr>
<td>Instron Settings</td>
<td>10.0 &lt; t ≤ 11.0</td>
</tr>
<tr>
<td>Load cell (lbfs) = 1000</td>
<td>11.0 &lt; t ≤ 12.0</td>
</tr>
<tr>
<td>Center zero reading (corresponds to size of load cell ) = 100</td>
<td>13.0 &lt; t ≤ 14.0</td>
</tr>
<tr>
<td>Full scale deflection (lbfs) = 200</td>
<td>PUSH PIN SIZE *</td>
</tr>
<tr>
<td>Cross head speed (in/min.) = 0.005</td>
<td>Pin diameter, p = 41.0 – 0.2 t</td>
</tr>
<tr>
<td>Chart speed (in/min.) = 20 sec/inch</td>
<td>Avg. t (mil)</td>
</tr>
<tr>
<td>6.25 - 8.75</td>
<td>39.5</td>
</tr>
<tr>
<td>8.75 - 11.25</td>
<td>39.0</td>
</tr>
<tr>
<td>11.25 - 13.75</td>
<td>38.5</td>
</tr>
<tr>
<td>13.75 - 16.25</td>
<td>38.0</td>
</tr>
<tr>
<td>Temperature details</td>
<td>Time log</td>
</tr>
<tr>
<td>(base thermocouple temperatures)</td>
<td>(specimen temperatures)</td>
</tr>
<tr>
<td>Target temperature:</td>
<td>Goal temperature:</td>
</tr>
<tr>
<td>Temp during crosshead</td>
<td>Temp during crosshead adjust:</td>
</tr>
<tr>
<td>adjust at start of test:</td>
<td>Time at test start:</td>
</tr>
<tr>
<td>Temp at start of test:</td>
<td>Temp at start of test:</td>
</tr>
<tr>
<td>Temp at end of test:</td>
<td>Temp at end of test:</td>
</tr>
<tr>
<td>Average specimen temperature:</td>
<td>Heat up start time:</td>
</tr>
<tr>
<td>°C</td>
<td>°C</td>
</tr>
<tr>
<td>°C</td>
<td>°C</td>
</tr>
<tr>
<td>°C</td>
<td>°C</td>
</tr>
</tbody>
</table>

#### Data calculations:

- Effective shear YS = Yield load / 2πr
- Effective maximum shear strength = Maximum load / 2πr
- 2r = average of push pin and bore diameters; t = specimen thickness; bore diameter = 0.0410 in

#### RESULTS:

<table>
<thead>
<tr>
<th>Yield Load / (lbfs)</th>
<th>Effective Shear Yield Stress</th>
</tr>
</thead>
<tbody>
<tr>
<td>Maximum Load / (lbfs)</td>
<td>Effective Maximum Shear Stress</td>
</tr>
</tbody>
</table>

#### Comments:

*Circle which push pin and shim were used

**t_max = largest recorded value of t
# Temporary Storage Unit Follower Card

## Test series:
Sheet number:
Date:
Initial:

<table>
<thead>
<tr>
<th>#</th>
<th>Specimen ID</th>
<th>Pencil ID (if applicable)</th>
<th>Initial location (cell number)</th>
<th>In use (record date)</th>
<th>Test complete (record date)</th>
<th>Final location (cell number)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td></td>
<td></td>
<td></td>
<td></td>
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<tr>
<td>2</td>
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</tr>
</tbody>
</table>

Remember to change punch after ~20 tests.
<table>
<thead>
<tr>
<th>Pencil Number</th>
<th>Specimen code</th>
<th>Dose Information</th>
<th>Test Temperature</th>
<th>Material</th>
<th>Location in 15/17A</th>
<th>Date shipped to 5A</th>
<th>Date tested in 5A</th>
<th>Date returned to 15/17A</th>
<th>Final location in 15/17A</th>
<th>Comments</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>Return to same</td>
<td>cell in lab 15/17A</td>
</tr>
</tbody>
</table>
Appendix 3 – Idealised shear punch-tensile correlation

Assume that a uniaxial stress state exists in the gauge length of a tensile specimen, i.e., \( \sigma_x = \sigma_y = 0 \)

\[ \sigma_{zz} = \sigma_y = 0 \]

...then using the von Mises yield criterion for this stress state yields:

\[
\sigma_0 = \frac{1}{\sqrt{2}} \left[ (\sigma_x - \sigma_y)^2 + (\sigma_y - \sigma_z)^2 + (\sigma_z - \sigma_x)^2 + 6(\tau_{xy} + \tau_{yz} + \tau_{xz}) \right]^{\frac{1}{2}}
\]

\[
\sigma_0 = \frac{1}{\sqrt{2}} \left[ (-\sigma_y)^2 + (\sigma_z)^2 \right]^{\frac{1}{2}}
\]

\[
\sigma_0 = \sigma_z
\]

And assuming that the stress state in the deformation zone of the specimen is that of pure shear, i.e., \( \tau_0 = \tau_{00} = 0 \)

...then using the von Mises yield criterion for this stress state yields:

\[
\sigma_0 = \frac{1}{\sqrt{2}} \left[ (\sigma_x - \sigma_y)^2 + (\sigma_y - \sigma_z)^2 + (\sigma_z - \sigma_x)^2 + 6(\tau_{x0} + \tau_{y0} + \tau_{z0}) \right]^{\frac{1}{2}}
\]

\[
\sigma_0 = \frac{1}{\sqrt{2}} \left[ 6(\tau_{x0} + \tau_{y0} + \tau_{z0}) \right]^{\frac{1}{2}}
\]

\[
\sigma_0 = \sqrt{3} \tau_{rz}
\]
The ratio of deviatoric stress in the tensile test to that in the shear punch test is given by the ratio of (i) to (ii):

$$\frac{\sigma_z}{\tau_{rz}} = \frac{1}{\sqrt{3}} = 1.73$$  \hspace{1cm} (iii)

The correlation that is expected by this theory between effective shear yield strength and tensile yield strength therefore has a slope of 1.73 and passes through the origin:
Appendix 4 – Derivation of uniform elongation correlation

Equation 3.3 was derived from the definition of power law strain hardening at yield and maximum strength:

\[ \sigma = K \varepsilon_{pl}^n \]

...where \( \varepsilon_{pl} \) is power law strain, \( n \) is the tensile strain hardening coefficient and \( K \) is a constant of proportionality.

Equation 3.3 was formed, assuming that \( \varepsilon_{pl} \) equals \( n \) at the onset of necking or maximum tensile strength:

\[ \sigma_{\text{max}} = K (n)^n \quad (-i-) \]

...and assuming that \( \varepsilon_{pl} \) equals 0.002 at the yield strength (standard offset):

\[ \sigma_{\text{yield}} = K (0.002)^n \quad (-\text{ii}-) \]

...taking the ratio of (i) / (ii) it follows that:

\[ \left( \frac{n \sigma}{0.002} \right)^n = \frac{\sigma_m}{\sigma_y} \]

(3.3)
Appendix 5 – A finite element model of the shear punch test

1. – Marc code listing with comments

2. – Model geometry

3. – Boundary conditions
1. Program listing [with comments in square brackets].

```plaintext
| Version : Mentat 3.1 |
| parameter names |
| all dimensions in mm |
| t   = Specimen thickness |
| rbl = Radius of specimen blank |
| rt  = Inner rad of Top Hold-down plate |
| ht  = Height of Top Hold-down plate |
| radt = Corner rad of Top Hold-down plate |
| rb  = Inner rad of Bottom Die |
| hb  = Height of Bottom Die |
| radb = Corner rad of Bottom Die |
| hp  = Height of punch |
| rp  = Radius of punch |
| radp = Corner radius of punch |
| nu  = Poisson's Ratio |
| emod = Modulus of Elasticity |
| mu  = Coeffic of Friction |

*new_model
yes
*reset
*define t
0.25
*define rbl
2.8/2
*define rt
1.0414/2
*define ht
0.5
*define radt
0.010
*define rb
1.0414/2
*define hb
0.5
*define radb
0.010
*define hp
0.5
*define rp
```

APPENDIX 5
0.9906/2
*define
radp
0.010
| define friction and matl props
*define
nu
0.28
*define
eemod
193e3
*define
syld
280.
*define
ehard
eemod/125
*define
eyld
syld/eemod
| friction coefficient, mu
*define
mu
0.204
| *****************************************
*add_points
*add_points
0,0,0
0,1.003*rp,0
0,1.0035*rp,0
0,1.11*rp,0
*add_points
0,rbl,0
*fill_view
*set_duplicate_translations
t,0,0
*duplicate_points
all_existing
*set_node_labels on
*set_point_labels on
*set_node_labels off
*redraw
*add_points
-t,0,0
-t,rp,0
-3*t,rp,0
-t,rt,0
-3*t,rt,0
-t,1.1*rbl,0
*fill_view
4*t,rb,0
2*t,rb,0

[geometry file - creates a grid to draw on, surfaces and fillets of model]
2*t, 1.1*rbl, 0

*add_curves
11
12
13
15
14
16
17
18
19

*set_curve_type_fillet
*add_curves
1
2
radp
3
4
radt
5
6
radb
*zoom_box
*zoom_box(1, 0.276644, 0.615776, 0.303855, 0.657761)
*fill_view
*set_curve_type_line
*add_surfaces
6
7
2
1
8
10
5
3

*convert_surfaces
*set_convert_divisions
50, 50
*set_convert_bias_factors
0.3
0

*convert_surfaces
1
# | End of List
*set_convert_bias_factors
-0.38, 0
*convert_surfaces
2
# | End of List
*redraw
*zoom_box(1,0.210884,0.867684,0.604308,0.975827)
*set_sweep_tolerance
0.0005
*sweep_all
*remove_unused_nodes
*renumber_all
*fill_view
*store_elements
sheet
all_existing
*select_sets
*zoom_box
*zoom_box(1,0.541950,0.627226,0.590703,0.681934)
*set_nodes_off
*set_node_labels_off
*redraw
*fill_view
*zoom_box
*zoom_box(1,0.410431,0.569975,0.589569,0.702290)
|**************************************************** [end of geometry]
|
*new_contact_body
*contact_body_name
mesh
*contact_deformable
*add_contact_body_elements
*fill_view
all_existing
# | End of List
*fill_view
*new_contact_body
*contact_body_name
punch
*contact_rigid
*contact_value initvx
0.05
*contact_value friction
mu
*contact_option rigid_desc: analytical
| *contact_value curve_div
| 10
*add_contact_body_curves
1 2 3
# | End of List
*new_contact_body
*contact_body_name
top_die
*contact_rigid
*contact_option control: velocity
*contact_value initvx

[surface contact definition - define rigid surfaces of dies and punch and defines elastic /
plastic surfaces of specimen and define friction]
0.05
*contact_value friction
mu
*contact_option rigid_desc:analytical
| *contact_value curve_div
| 10
*add_contact_body_curves
  4 5 6
# | End of List
*new_contact_body
*contact_body_name
bot_die
*contact_rigid
*contact_option control:velocity
*contact_value initvx
-0.05
*contact_value friction
mu
*contact_option rigid_desc:analytical
| *contact_value curve_div
| 10
*identify_contact *regen
*add_contact_body_curves
  7 8 9
# | End of List
*zoom_box
*zoom_box(1,0.648526,0.480916,0.785714,0.690840)
*zoom_box
  15
*zoom_box
*zoom_box(1,0.303855,0.641221,0.400227,0.745547)
*flip_curves
  7
  8
  9
# | End of List
*fill_view
*zoom_box
*zoom_box(1,0.302721,0.609415,0.332200,0.653944)
# | End of List
*fill_view
*new_contact_table
*contact_table_touch_entry 1 1
*contact_table_touch_entry 2 1
*contact_table_touch_entry 3 1
*contact_table_touch_entry 4 1
*contact_table_property $ctbody1 $ctbody2 friction
mu
*contact_table_property $ctbody1 $ctbody2 sep_for
lel
*apply_type fixed_displacement
*apply_name
fixx  
*apply_dof x  
*apply_value x  
0.00  
*zoom_box  
*zoom_box(1,0.545351,0.142494,0.596372,0.223919)  
*add_apply_nodes  
2651  
# | End of List  
*new_material  
*material_name  
mat11  
*material_type mechanical:isotropic  
*material_value isotropic:youngs_modulus  
emod  
*material_value isotropic:poissons_ratio  
nu  
*new_table  
*table_name  
elplastic  
*set_table_type  
plastic_strain  
*table_add  
0.000,280.0  
*table_add  
0.0192,340.0  
*table_add  
0.194,600.0  
*table_add  
0.394,820.0  
*table_add  
0.514,927.0  
*table_add  
0.900,930.0  
*table_fit  
*material_type mechanical:isotropic  
*material_type plasticity  
*material_value plasticity:yield_stress  
1.0  
*material_table plasticity:yield_stressO  
elplastic  
*add_material_elements  
all-existing  
*geometry_type mech_axisym_solid  
*add_geometry_elements  
all-existing  
*previous_contact_body  
*previous_contact_body  
*contact_rigid  
*contact_option control:velocity  
*contact_option control:velocity  
*contact_value vx  

[sets a node on the centreline to be rigid in x-axis]  
[defines material]  
[sampled data from true stress versus true plastic strain curve for material defined]  
[sets velocity of punch]
1.0
*loadcase_type static
*loadcase_value maxrec 50
*loadcase_option nonpos: on
*loadcase_ctable
ctable1
punch
ctable1
*loadcase_type static
*loadcase_ctable
ctable1
*loadcase_value time 2e-3
*loadcase_value nsteps 40
*job_class mechanical
*add_job_loadcases lcase1
*job_option large: on
*job_option update: on
*clear_post_tensors
*job_param post
2
*job_option post: binary
*add_post_tensor stress
*add_post_tensor el_strain
*add_post_tensor pl_strain
*remove_post_tensor el_strain
*remove_post_tensor pl_strain
*add_post_tensor strain
*add_post_tensor pl_strain
*add_post_var von_mises
*add_post_var epl_strain
*add_post_var pe_energy
*job_param memory 10000000
*job_option solver: sparse
*job_option frictype: coulomb
*job_option dimen: axisym
*element_type 10
all_existing
*renumber_all
*check_job
cw0_bc
*save_as_model cw0_bc.mud yes
*update_job
*set_sweep_tolerance 0.0005
*sweep_all
*remove_unused_nodes
*renumber_all
*fill_view
2 – Screen shots of model geometry

Fig. 1 shows the finite element model for the shear punch test. The upper and lower die close on the specimen before a test and remain rigid in space as the punch is advanced on to the specimen. There are 50 elements across the thickness of the specimen and there is a reduction in the element size (in the y-axis) towards the region that is will be subjected to maximum distortion. Fig. 2 shows the detail of the elements at the point of interest. The clearance between the punch and die is 7.5% of the specimen thickness.

Figure 1 – Shear punch finite element model geometry before running.
Figure 2 – Detail of the shear punch finite element model, showing mesh size (note that approximately only a eighth of the punch diameter is actually shown). The clearance of the punch in the lower die, expressed, as a percentage of the specimen thickness is approximately 7.5% for the configuration shown.

3 – Finite element model boundary conditions

- The punch is modelled as a moving, rigid surface and the die and clamping surfaces are also modelled as being rigid.
- The effects of strain rate are ignored as the punch is moving at a slow strain rate (~0.127 mm.min⁻¹).
- The specimen material is isotropic and obeys the von Mises yield criterion.
- Gravity and temperature effects are ignored.
- The material properties of the specimen are input as a sampled set of co-ordinates taken from a true stress versus true plastic strain plot from a tensile test.
- There is a non-penetration constraint between the working piece, punch and die that is handled by a subroutine in the MARC code. The friction between the contacting bodies can be changed.
THE USE OF SHEAR PUNCH TESTING TO CLARIFY THE CONSEQUENCES OF HELIUM PRODUCTION IN THE DEFORMATION OF ISOTOPICALLY TAILORED FERRITIC ALLOYS


ABSTRACT: The shear punch test was used to evaluate the strengthening associated with the production of helium in model Fe-Cr-Ni alloys during irradiation in HFIR at 300-600°C. Four alloys were considered, a base alloy of composition Fe-12Cr and three alloys with 1.5% Ni, doped isotopically to contain 60Ni, natural nickel or 59Ni. The addition of nickel in any isotopic balance significantly strengthened the base alloy, and as expected, the strength of the alloys decreased with increasing irradiation temperature. Helium itself, however, up to the 75 appm produced over 7 dpa appeared to have little effect on the strength of the alloys.

KEYWORDS: shear punch, ferritic alloys, helium, isotopic tailoring

It is expected that the most significant difference between fission and fusion reactor environments is the high rate of transmutant helium generation resulting from fusion spectra [1]. In the worst case in a high nickel content alloy, the rate for helium generation in a fast reactor is ~0.5 appm He/dpa which compares with an expected level of ~10 appm He/dpa for iron-based alloys proposed for first wall applications in fusion reactors. Many of the previous experiments devised to study the effect of helium levels relevant to fusion reactor materials used complex alloys which had sometimes experienced different neutron irradiation spectra and flux levels in order to simulate the expected conditions. These experiments were not always successful since potential effects of the helium were masked by the more dominant effects of neutron flux and temperature history [2]. Helium production at a rate of 10 appm He/dpa can be achieved in HFIR by the addition of 1.5% 59Ni [3].

A recent study [4] reported on a simple, one variable experiment which was devised in order to study the effects of different helium levels on the microstructures that evolved in a set of isotopically tailored ferritic alloys, nominally of composition Fe-12Cr-1.5Ni. The rate of helium evolution was varied from 0.3 to 10.7 appm/dpa without changing the neutron spectrum or the atomic displacement rate, by varying the isotopic content of the nickel. The same composition and initial microstructure was maintained for the nickel containing alloys. The first Fe-12Cr-1.5Ni variant contained the 59Ni isotope which undergoes an (n, α) reaction to form helium. This idea had been tested and proven in a previous series of FFTF irradiations on Fe-Cr-Ni austenitic alloys [5]. The second alloy contained 60Ni, which produces very little helium (~2 appm He after 7 dpa), and the third alloy contained natural nickel, which produces an intermediate level of helium after the delayed development of 59Ni. The fourth alloy, which was included to clarify the role of nickel on the properties of these alloys, contained no nickel.

This paper reports on the shear punch testing of the same set of alloys. Of particular interest are changes in strength of these alloys as a function of helium content.

EXPERIMENTAL METHOD

Specimens were irradiated side-by-side to ~7 dpa in the HFIR-MFE-JP23 experiment at temperatures ranging from 300 to 600°C. Based on results from previous irradiation experiments, it is expected that sufficient dose to initiate void swelling in ferritic/martensitic alloys would have been experienced by this point [4].

The shear punch test is essentially a blanking operation which is common to sheet metal forming. A 1 mm diameter punch is driven at a constant rate of 0.127 mm/min. (0.005 in./min.) through

1 Senior research scientist and staff engineer, respectively, Pacific National Northwest Laboratory, Richland, WA 99352
2 Graduate student, IPTME, Loughborough University, LE11 3UT, England
a TEM-sized disk (nominally 0.25 mm thick and 2.8 mm in diameter). The load on the punch is measured as a function of punch travel, which is taken to be equivalent to the cross head displacement [6]. This assumes that the test machine and punch are completely stiff relative to the response of the test specimen. A plot of punch load versus punch displacement was obtained for each specimen.

With the exception of one set of irradiated aluminium alloys [7], shear punch testing has thus far only been carried out on unirradiated materials. A new test facility was set up and a detailed procedure was written to accommodate the shear punch testing of highly irradiated specimens. All tests were conducted under ambient conditions and the results of each test were recorded by computer and simultaneously recorded on a chart recorder.

The curve obtained from a shear punch test is of a similar form to that obtained from a tensile test. Initially a linear relationship exists between load and punch displacement during which no plastic deformation occurs. This is followed by a deviation from linearity or yield point when permanent penetration of the punch into the specimen occurs. Beyond the yield point, further deformation forms a shear process zone between the die and punch. Work hardening compensates for thinning until a maximum load is achieved [6]. The points of interest on the curve were the yield load and maximum load. Effective shear yield strength (τ_y) and an effective maximum shear strength (τ_m) can be evaluated from these values, respectively, by the following equation [8]:

$$τ_{sy, sm} = \frac{P}{2\pi rt}$$

where P is the appropriate load, r is the average of bore and punch radii and t is the specimen thickness. Previous work has shown that an empirical relationship can be developed between data from shear punch testing and that from tensile testing [7,8,9]. In this instance, however, no tensile data were available and the shear punch test was used only as a tool to identify trends in the mechanical properties that might occur as a result of differing helium levels.

RESULTS

Two tests per specimen condition were performed with good reproducibility in the data: effective shear strength typically varied by no more than 30 MPa between duplicate specimens. Figure 1 shows a summary of the average values of τ_y as a function of helium content. Figure 2 shows a similar plot for τ_m. Data from the unirradiated material is included in Figures 1 and 2 with the corresponding data from the irradiated material.

It is evident from the plots of τ_y and τ_m that the addition of nickel to the Fe-12Cr base alloy significantly increases the strength of both the unirradiated and the irradiated alloys, especially for irradiation temperatures of 300 and 400°C. The yield and maximum shear strengths are increased by ~100% for the unirradiated condition, a result which is independent of the nickel isotope balance used.

The strength of all alloys decreased with increasing irradiation temperature. The highest strength was observed for alloys irradiated at 300°C. The alloys irradiated at 500 and 600°C experienced an overall decrease in strength when compared to the unirradiated condition.

A small increase in both τ_y and τ_m is observed with increasing helium content in the irradiated alloys. Since the same trend is echoed in the data for the unirradiated material, however, it cannot be attributed to the helium level, but rather to another factor inherent to the alloys. Figures 3 and 4 show the change in τ_y and τ_m with respect to the unirradiated condition (Δτ_y and Δτ_m, respectively) versus helium.
Figure 1 - Effective shear yield strengths ($\tau_y$) in Fe-12Cr-1.5Ni as a function of helium content (an open symbol signifies the control alloy [Fe-12Cr] at the same condition as the corresponding filled symbol).

Figure 2 - Effective maximum shear strengths ($\tau_m$) in Fe-12Cr-1.5Ni as a function of helium content (an open symbol signifies the control alloy [Fe-12Cr] at the same condition as the corresponding filled symbol).
Figure 3 - Change in $\tau_y$ with respect to the unirradiated condition as a function of helium content.

Figure 4 - Change in $\tau_{um}$ with respect to the unirradiated condition as a function of helium content. While a shallow negative gradient is observed for the lower irradiation temperatures for $\Delta\tau_{op}$, the trend is reversed in Figure 4 for $\Delta\tau_{um}$. 
DISCUSSION

It is clear from Figures 1 and 2 that nickel additions significantly increase \( \tau_{\text{sy}} \) and \( \tau_{\text{sm}} \) of the alloys before irradiation. The strengthening effect persists following irradiation at all irradiation temperatures, which suggests that the strengthening action was present, at least in part, prior to irradiation and can probably be attributed primarily to solution strengthening/precipitation hardening due to the addition of nickel. The slight variability observed in the strength for each isotopic variation is currently unexplained, but may arise from variability in impurity levels associated with the isotopic additions.

Irradiation at 300 and 400°C both caused an increase in strength when compared to the unirradiated material with the greatest strengthening occurring at 300°C. This is in agreement with the microstructural analysis carried out by Gelles [4] on the same set of alloys. The microstructure of the material irradiated at 300°C exhibited a dense distribution of fine precipitates, small voids and small dislocation loops. Each of these features would be expected to produce an increase in the strength of the alloy. The material irradiated at 400°C showed a more coarse microstructure, with larger and fewer precipitates and fewer, but more developed, dislocation loops. The material irradiated at 500 and 600°C showed an overall reduction in strength when compared with the unirradiated condition. Although the microstructures have not been studied for the alloys irradiated at 500 and 600°C, it is expected that further coarsening and increased loop and precipitate growth will have occurred.

The plot of \( \Delta \tau_{\text{sy}} \) versus helium content (Fig. 3) shows a shallow positive gradient for materials irradiated at the lower temperatures. This trend is reversed, but has a slightly steeper gradient, in the plot of \( \Delta \tau_{\text{sm}} \) versus helium level (Figure 4). The variation in \( \Delta \tau_{\text{sy}} \) over the range of helium levels for irradiation at a given temperature is only ~30 MPa, which is essentially the same as the scatter in the data for a given test condition (~±20 MPa). It is therefore difficult to draw any conclusions, other than that helium levels up to 75 appm have no significant influence on the mechanical properties over the range of temperatures considered.

CONCLUSIONS

The shear punch testing of a series of isotopically tailored Fe-12Cr-1.5Ni ferritic alloys showed that helium levels up to 75 appm have little, if any, effect on the effective shear yield and maximum shear strengths. The strengthening effect of nickel was evident prior to irradiation and the strength of the irradiated Fe-12Cr-1.5Ni ferritic alloys shows a strong dependence on irradiation temperature, decreasing with increasing irradiation temperature.

FUTURE WORK

Chemical analysis of the unirradiated controls is planned to investigate the possible effect of variation in impurity levels.

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and Uniaxial Tensile Data”, Journal of Nuclear Materials, in press.
Appendix 7 – Manufacturing Drawings for the Shear Punch Test Fixture

1. – Upper fixture half
2. – Lower fixture half
3. – Fixture handling tools
4. – Fixture Shims
SHEAR PUNCH FIXTURE
UPPER HALF

INSERTION HOLES FOR
FIXTURE HANDLING TOOL

Material: 304 SS
Units in mm
Not to scale
Drawn by GL Hankin
g.l.hankin@lboro.ac.uk
August 17 1998

Ø70.0

3xF - HOLES FOR Ø7.0 mm
BOLTS

3xG - Ø7.0 mm ALIGNMENT PINS
PRESS FIT IN UPPER FIXTR. HALF
(British Standard 4500A hole size
H7, shaft size p6) OR SUGGEST
0.01-0.015 mm DIAMETRAL
INTERFERENCE [G+0.010 G] [J 0.005]

H - SUPPLIED PART (hard ferritic
tool steel bushing) - Ø5/32"
OUTER DIA., Ø0.0395" INNER DIA.
BUSH CUT TO 15.5 mm LONG.
PRESS FIT IN UPPER FIXTURE
HALF (British Standard 4500A
hole size H7, shaft size p6)
OR SUGGEST 0.01 - 0.015 mm
DIAMETRAL INTERFERENCE

Loughborough
University

SECTION A - A
SHEAR PUNCH FIXTURE
LOWER HALF

SHIM RECESS
0.250 mm DEEP

10.0

22.5

61.0

ø5.0

B

ø70.0

ø50.0

2.0

8.5

B

3х1 - THREADED ø7.0mm HOLES FOR 3xM7 FINE-PITCH BOLTS

4.0 mm WIDE
X 2.0 mm DEEP

3х1 - ø7.0 HOLE FOR SLIDE FIT (BS 4500A H7, h6 OR DIAMETRAL CLEARANCE<0.02mm) FOR ø7 mm ALIGNMENT PINS SET IN UPPER FIXTURE HALF [J+0.010] [J+0.005]

K - SUPPLIED BUSHING -ø5/32" O.D., ø0.0410" I.D. (ferritic tool steel) CUT TO 9.6 mm LONG. PRESS FIT (SAME FIT AS PART H) IN LOWER FIXTURE HALF, THEN POLISH DOWN TO FIXTURE SURFACE (see special instructions) [K+0.010] [K+H0.005]

Material: 304 SS
Units in mm
Not to scale
Drawn by GL Hankin
g.l.hankin@lboro.ac.uk
August 17 1998

Loughborough University

SECTION B - B
SHEAR PUNCH FIXTURE
SHIM SET

CUT 3 OF EACH SIZE FROM STANDARD GAUGE SHIMS.
THE RANGE OF SIZES REQUIRED IS BETWEEN 0.400 AND 0.600 mm IN INCREMENTS OF 0.025 mm

Material: 304 SS
Units in mm
Not to scale
Drawn by GL Hankin
Loughborough
University

17 August 1998
SHEAR PUNCH FIXTURE
FIXTURE HANDLING TOOL

2 EACH MADE FROM Ø5 mm TUBE — *LENGTH IS
APPROPRIATE FOR REMOTE HANDLING

Material: 304 SS
Units in mm
Not to scale
Drawn by GL Hankin
g.l.hankin@lboro.ac.uk
17 August 1998

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- PLAN -

- SIDE ELEVATION -

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