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An Investigation of the Factors Determining Creep Strength and Ductility in Grade 92 Steel

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Summary
The ECCC creep rupture data sets, initially collated for design strength assessment, can also provide a valuable resource for wider investigations. Thus, variations in properties between different casts or batches with different chemical composition, heat treatment, manufacturing route and product form can be analysed. This can be an effective means of determining how alloy composition and heat treatment may be optimised.

This paper describes an investigation into the causes of the large cast-to-cast variations in long term creep ductility observed in the ECCC Grade 92 data set. The analysis shows that a major factor, accounting for the bulk of the variability, is the normalising and tempering heat treatment. Under-normalised batches, especially with normalising times below 30-60 minutes, show inferior long term ductility. Light tempering may exacerbate these effects.

Under-normalising is also well correlated with high ambient temperature tensile strength and with high creep rupture strength under short term test conditions, when virtually all test failures are highly ductile. In longer term uniaxial creep testing, when the under-normalised batches show much reduced ductility, their relative uniaxial creep rupture strength values tend to be somewhat poorer, though not exceptional. The potentially greater harmful influence of low creep ductility on performance under multiaxial loading and cyclic operation merits further study.

Long term uniaxial creep rupture strength tends to be relatively poor when batches are either severely over-normalised and tempered (hence weak and ductile), or severely under-normalised and tempered (hence strong but brittle). However, there is also substantial scatter, no doubt due to compositional variations. Hence, whilst intermediate levels of manufacturing heat treatment broadly appear to be optimal, some batches with intermediate heat treatment levels do also appear amongst those with the poorest long term creep performance.

Key Words
Creep rupture, creep ductility, Grade 92, effects of manufacturing heat treatment, ECCC creep data assessment.

Introduction
The influence of creep ductility on the performance of materials at high temperatures is still rather controversial. Design methodologies for pressure parts operating at high temperature are generally based solely on uniaxial creep rupture strength data, but it is often argued that low creep ductility should also be recognized as a specific hazard. Parker [1] pointed out that low ductility materials may fail prematurely in notched creep tests, and could also be at risk when components are multiaxially loaded. However, Spindler [2] has argued that creep ductility is only a substantial concern for relatively brittle materials with uniaxial ductility values below ≈ 10-30%.

Grade 92 steel, found to be stronger than Grade 91 in uniaxial but not in notched creep tests [1], has been identified as a specific material of concern. Ductility concerns have consequently limited the deployment of a stronger material which could potentially enable higher operating temperatures, efficiency gains and CO2 emission reductions.

In previous work initially undertaken at E.ON UK (now Uniper Technologies), this author sought to analyse the ECCC creep data sets on Grades 91 and 92 to throw light on these questions [3, 4]. It was shown that some casts, especially in Grade 92, do exhibit low uniaxial creep ductility values in longer term tests. However, different material casts showed considerable differences in ductility, with many Grade 92 and most Grade 91 casts exhibiting good ductility values throughout the experimental test range.
A new UK collaborative development project “IMPULSE”, on the industrial-scale pipe manufacture, welding and long term performance assessment of the UK MARBN steel type IBN1 developed in the preceding IMPACT [5] project, has now prompted further work on creep ductility. IBN1, and parallel martensitic alloys such as the Japanese SAVE12AD and the Chinese G115 broadly related to the 9Cr3W3CoBN “MARBN” concept put forward by Abe [6], have demonstrated considerable further advances in high temperature strength beyond that of Grade 92. However, creep ductility could again be a concern, and one which is less readily assessed, due to the limited availability of long term creep and creep ductility data.

IMPULSE has therefore chosen to undertake further work on the ECCC Grade 92 data set, with the aim of deriving a broader understanding of the causes of poor creep ductility in this well-tested material, and thereby obtaining insights which can be carried forward into the analysis of creep behaviour in IBN1. The current work concentrates on analysing why different casts of Grade 92 should show such major differences in creep ductility, alongside differences in rupture strength and other mechanical properties, with particular emphasis on the effects of the manufacturing heat treatment.

Analysis
This and the previous [3,4] work was based on the ECCC Grade 92 data set as assessed in 2005 to obtain the master equation predicting mean rupture strength values as provided in the current ECCC Data Sheet [7]. All-data plots show that short term tests usually fail with high ductility, but an increasing proportion of longer term tests exhibit much lower ductility, Figures 1 and 2.

To provide a simple measure of creep ductility for each material cast, the ductility (reduction of area, RA%) data were averaged for all tests on that cast, subdivided into the following categories:
Short term tests – Predicted life < 1Kh
Medium term tests – Predicted life 1 – 10 Kh
Long term tests – Predicted life > 10Kh

To provide parallel information on creep rupture strength, the ECCC master equation was used to convert each data point into an evaluation of the relative creep rupture strength of the specific cast tested. Thus, when (for example) cast X tested at 105 MPa achieves a certain creep life, while the predicted mean Grade 92 rupture strength corresponding to that creep life is 100 MPa, the selected data point on cast X represents a rupture strength of 1.05 relative to the mean prediction. An overall average relative rupture strength for cast X may then be obtained by averaging all the data points on that cast. Again, separate average relative rupture strength values were also determined for each cast within the short, medium and long term test ranges defined above.

Correlation of Creep and Tensile Properties
Plots of creep rupture ductility and creep rupture strength against the as-manufactured ambient temperature ultimate tensile strength (UTS) revealed striking correlations. Whilst short term rupture ductility was uniformly high for all casts, medium term and long term creep ductility values decreased as the as-manufactured tensile strength increased, markedly in the case of the long-term data, Figure 3. Relative creep rupture strength values, while exhibiting rather more scatter between the different casts, also showed significant albeit more complex correlations with tensile strength, Figure 4. Short term creep rupture strength showed a strong positive correlation with tensile strength, but medium term rupture strength was more weakly correlated. For long term rupture strength, the linear correlation became inverse, while a polynomial trendline (dashed) suggested a more complex correlation. Optimal
creep properties were found at intermediate tensile strength values, with poorer overall creep rupture behaviour at both the lowest and the highest as-manufactured tensile strength.

A simple interpretation of these results is that high as-manufactured tensile strength is correlated with high creep deformation strength, but also with low longer term creep ductility. In short term high-stress tests which fail by ductile creep deformation, therefore, rupture strength is simply correlated with tensile strength. In medium term tests, ductility has some influence on rupture life, and acts to weaken the positive correlation between creep rupture strength and as-manufactured tensile strength. In longer term uniaxial tests, it is apparent that the markedly poorer ductilities of the high-UTS casts do act to reduce their uniaxial rupture strength, albeit not greatly. Indeed, the casts with the lowest long term uniaxial rupture strength shown in Figure 4 are weak and ductile, rather than strong and brittle. However, low ductility might become a greater concern in the very long term and/or under multiaxial loading [3].

Influence of Manufacturing Heat Treatment – Tensile Properties

Tensile and creep properties depend on alloy chemistry, processing route and product form, and the final manufacturing “quality” normalising and tempering heat treatment. Figures 3 and 4 indicate that long term creep ductility is quite highly correlated with as-manufactured tensile strength, but that other factors such as alloy chemistry also have a substantial influence on creep rupture strength.

The influence of manufacturing heat treatment on as-manufactured tensile properties was investigated by plotting the tensile data against the heat treatment Larsen-Miller parameter, defined as:

\[ LMP = T (20 + \log_{10} t) \]

where \( T \) is tempering (or normalising) temperature in °K, and \( t \) is soak time in hours

The results show, as expected, that lower tempering times and temperatures cause less softening and lead to higher tensile properties, Figure 5. Rather more surprisingly, lower normalising times and temperatures are also associated with higher tensile properties, and appear to have a greater influence than the tempering conditions, Figure 6, especially with respect to 0.2% proof stress (PS).

Separate plots of the tensile data against normalising time and normalising temperature, Figures 7 and 8, clarify these findings. Whilst the normalising LMP can be used as an indicative correlation parameter, it is normalising time rather than temperature which primarily governs tensile properties. Many casts were normalised for times of less than an hour, in some cases as short a time as ten minutes, and it is these which principally show raised tensile strength values.

A combined plot of the tensile data against the “normalising and tempering LMP”, determined simply by summing the normalising LMP and the tempering LMP, is shown in Figure 9.
As indicated by the $R^2$ values shown on the plots, the combined plot of Figure 9 provides the best overall correlation between tensile properties and heat treatment conditions. Thus, whilst short normalising time emerges as the most important factor causing high tensile strength, lighter tempering also appears to be associated with somewhat higher tensile properties.

Figure 9 also demonstrates that the relationship is far from linear. Whilst increasing the normalising and tempering LMP up to an approximately median value of about 48,000 clearly acts to reduce tensile strength, any further increase in LMP has little effect. Separate trendlines fitted only to the “high LMP” data above 48,000 are almost
flat, indicating that UTS then falls to a lower mean “plateau” value of the order of 680-700 MPa. This is quite close to the value which corresponds to maximum creep rupture strength, Figure 2.

Influence of Manufacturing Heat Treatment – Creep Ductility

A plot of long term creep ductility (RA) against the combined normalising and tempering LMP parameter, Figure 10, shows a remarkably strong correlation. Eleven casts have average long term RA values below 25%, and all eleven of these casts have combined LMP values falling below the approximate median LMP value of 48,000. As with tensile properties, Figure 9, long term RA is strongly dependent on LMP within the range up to 48,000, but becomes much less dependent on LMP at higher values. Thus, for LMP > 48,000, long term RA averages about 50% and shows little dependence on LMP, though values are scattered within an approximate range of 30% - 80%. Arguably, this scatter is unimportant, because all Grade 92 casts heat treated within this range demonstrate very acceptable creep ductility values. For LMP < 48,000, apart from two relatively ductile outlier casts, the data show a very strong correlation between ductility and LMP – with four creep brittle casts, all with very low LMP values, showing ductility values around or below 10%.

Separate plots of long term creep ductility against normalising LMP, Figure 11, and tempering LMP, Figure 12, throw more light on how heat treatment affects creep ductility. There is a clear correlation with normalising LMP alone, with all casts exhibiting ductility values below 30% falling in a normalising LMP range below 26700. By contrast, whilst the great majority of casts with tempering LMP values above 21,000 show very little correlation between ductility and tempering LMP, the five casts with tempering LMP values below 21,000 show strikingly different behaviour. Four of these casts, ringed on both plots below, are those with poorest ductility. These casts do have fairly low normalising LMP values, but also show particularly low tempering LMP values.

It thus appears that both of the manufacturing heat treatment operations influence long term ductility, but in different ways. First, if adequate normalising (approximately, with LMP > 26700) is carried out, then good ductility
in a range exceeding 25-30% can be guaranteed. However, if the material has been under-normalised, then long term ductility is highly likely to fall below 25%.

Secondly, it appears that tempering may also significantly affect long term ductility, though only when the material has been under-normalised. In that case, the avoidance of under-tempering (i.e. tempering LMP > 21000) appears to ensure at least moderate long term ductility, of the order of 20-25%. However, when the material is under-tempered (tempering LMP < 21000) as well as under-normalised, then Figure 12 suggests that long term ductility is likely to fall well below 20% - and for two of the casts in the data set, to 5% or below.

Material thickness was also identified as a significant variable, Figure 13. Of the eleven casts which had been normalised for soak times below 1 hour, the five products with wall thickness ≤ 12mm all showed fairly reasonable long term ductility, with RA values in the range 23-58%. However, the six products with wall thicknesses ranging from 27mm up to 180mm (solid bar) showed much poorer ductility levels, with long term RA values ranging from 21% down to 3%. It thus appears that the failure of the thicker section products to achieve their intended normalising temperatures within a short soak period was probably a key factor involved in producing a strong but creep-brittle microstructure.

Unfortunately, Figure 14 reveals a cause of uncertainty. Presumably by pure coincidence, all but one of these six thick casts (two of which plot identically) also happen to have a tempering LMP below the “critical” value of 21,000, while all the five relatively thin casts have a tempering LMP above 21,000. Hence, it is difficult to identify which factor/s truly act to exacerbate under-normalising (soak time below 1 hour) – low <21,000 tempering LMP, high (>12mm) material thickness, or indeed both. The single “thick” cast of Figure 14 with a relatively high tempering LMP does also have a relatively moderate long term RA value, 21%. Most probably, therefore, both factors play a role.

It should be noted that the “critical” tempering LMP value of 21,000 proposed here equates to 1 hour at 777°C or 3 hours at 753°C, and that the majority of the materials in the ECCC data set were in fact tempered at quite high temperatures within the specification range. Hence, whilst achieving the “critical” tempering LMP value of 21,000 in manufacture would seem a sensible provision, there is probably little need to achieve much higher values. By contrast, the avoidance of under-normalising, especially for thicker sections, emerges as a clear priority.

The four most brittle casts, with long term ductility values in the range 3-12%, had normalising times in the range 0.2-0.6 hours. The next seven fairly brittle casts, all with long term ductility values in the range 18-24%, had normalising times of 0.2-1.0 hours.

Finally, it should be noted that whilst under-normalising using an inadequate short soak time is identified as a major concern, producing undesirably strong but brittle Grade 92, the tensile properties of the under-normalised materials in the ECCC Grade 92 data set are well within the normal specification range, Figure 9. The ASTM Grade 92 specification provides only a minimum UTS value, 620 MPa, but the EN specification for Grade 91, 630 – 830 MPa, provides an indication of the normal range. Thus, all the low-ductility casts in the ECCC Grade 92 data set conform with current tensile property specifications.
Influence of Manufacturing Heat Treatment – Creep Rupture Strength

The direct relationships between creep rupture strength and manufacturing heat treatment conditions are shown in Figures 15 and 16. The all-data plot of Figure 15, dominated by short and medium term data, shows a consistent trend toward higher strength at low LMP values, with the “problem” under-normalised and under-tempered casts with very low LMPs amongst the strongest in short term creep. However, the long term data plot of Figure 16 shows quite different behaviour, with a tendency toward best performance at intermediate LMP. These correlations parallel the observed correlations with tensile properties, Figure 4, and also with creep ductility, as shown in Figure 17. The correlation with creep ductility shows least scatter, and this indicates that the two parallel results of the creep test, i.e. its life and its local strain to failure, are closely linked. Very high ductility is associated with low creep deformation strength, while very low ductility implies early failure and hence reduced creep rupture strength.

Discussion

Manufacturing Heat Treatment

In Grade 92, the steel manufacturing heat treatment clearly has a major influence on ambient temperature tensile strength, short term creep deformation strength, and long term creep ductility. Low normalising and tempering temperatures and timescales produce stronger, more brittle materials. However, these effects are not simple. Within the normal heat treatment ranges included in the ECCC data set, severe (high total manufacturing LMP) heat treatments did not generally produce much weaker or more brittle materials than did mid-range heat treatments. However, under-normalising, especially reductions in normalising time to 30-60 minutes or below, caused substantial strengthening, in particular when applied to thick section products, together with considerable reductions in long term creep ductility.

Tensile and Short Term Creep Strength

Because virtually all the short term creep tests failed with similarly high ductility, short term creep rupture strength in this data set is effectively a measure of creep deformation strength. Hence, it is not surprising that this high temperature strength parameter is well correlated with ambient temperature tensile strength, see the <1Kh creep life data points in Figure 4. Whilst the high temperature and low temperature “strength” parameters are each similarly correlated with the manufacturing heat treatment parameters, Figures 9 and 15, both these plots also show considerable scatter. This no doubt arises because the “strength” parameters are substantially dependent on alloy chemical composition, which varies from cast to cast, as well as on manufacturing heat treatment.

Long Term Creep Ductility

The parameter which is most strongly correlated with manufacturing heat treatment, however, is the long term uniaxial creep ductility, Figure 11. This shows rather less scatter, as compared with the overall data trend, than does Figure 9 or Figure 15. Further, some of the scatter observed in Figure 11 results from the effect of material thickness as shown in Figure 13. It can thus be concluded that, whilst under-normalising does substantially increase the high temperature and low temperature “strength” parameters, its effects on long term creep ductility are more pronounced, while compositional differences appear to have rather less influence on ductility.
Most importantly, however, this work shows that only a minority of Grade 92 casts, those which have been subjected to specific heat treatment parameters, exhibit a serious embrittlement problem. It cannot be adequate, therefore, simply to carry out tests on one or two selected Grade 92 materials and seek to draw conclusions applicable to Grade 92 as a whole.

Long Term Creep Rupture Strength
The fourth materials property parameter analysed here, longer term creep "rupture strength", is the most complex. Whilst the term "rupture strength" is conventionally used, this parameter actually depends on the interplay between creep deformation strength and creep ductility. Thus, as indicated in Figure 4, the high-deformation-strength under-normalised materials show superior creep rupture performance in short term tests, a lesser superiority in medium term tests when their poorer ductility begins to be a disadvantage, and (in some cases) inferior rupture performance in long term tests, when ductility becomes more important.

Longer term creep rupture strength again shows substantial scatter between different casts, outweighing the identified trend variations with manufacturing heat treatment, Figure 16. Consequently, whilst the lowest LMP casts (brittle) and the highest LMP casts (ductile) do both tend to show poor rupture strength, other relatively poorly performing casts in terms of creep rupture strength can be found at any manufacturing LMP level. To put this into context, it should also be noted that the Grade 92 data set shows a comparatively tight long term rupture strength scatter band, Figures 16 and 17, with virtually all casts within +/- 10% of the mean.

Implications – Existing Plant
These findings have important practical consequences for the monitoring of existing Grade 92 plant, which may contain under-normalised material. Previous work [3] has indicated that in Grade 91, all the casts with poorest long term rupture strength were weak and ductile, not strong and brittle. Hence, the monitoring of tensile, hardness and/or short term creep deformation strength (including impression creep) to identify creep-weak materials could be recommended as a valid and central approach toward the assurance of structural integrity in Grade 91 plant.

In Grade 92 plant, by contrast, neither the weakest nor the strongest casts within the normal range can necessarily be assumed to be those at greatest risk, while the tensile strength values of the brittle under-normalised casts are not clearly abnormally high. Monitoring techniques such as hardness checking do still have a place in detecting and eliminating "aberrant" Grade 92 materials, for example components subjected to gross heat treatment errors and hence exhibiting soft ferritic or hard untempered martensitic structures rather than the appropriate tempered martensite structure. However, it cannot otherwise be assumed that hardness or short-term creep strength data will reliably identify which Grade 92 components on plant are at greatest long term risk. Further, under-normalised components in service are not likely to be identifiable on the basis of exceptionally high hardness values, especially after in-service softening has taken place. Manufacturing records are likely to provide a more reliable guide.

Implications – New Materials
The findings offer a clear route toward the development of improved manufacturing procedures to eliminate creep brittleness and ensure the reliable performance of future Grade 92 components for future plant. For thicker section materials, the normalising time should be at least 1 hour, preferably higher. For thinner section materials, shorter normalising times may be allowable, subject to process development and assessment.

This work has been quite successful in identifying the critical factors, because a large number of casts with different heat treatments were included in the ECCC data set analysed. Nevertheless, the analysis of a collated data set inevitably has limitations. These should be addressed by direct experimental development, exploring systematic variations in heat treatment specifications on a single cast of Grade 92 material. As long term creep ductility is not simple to measure, it is suggested that tensile strength be used as a proxy. It should be possible to explore what minimum normalising time is required to bring tensile values down to a consistent moderate level, and to ensure that this moderate tensile (or hardness) level is uniformly achieved throughout the component thickness. This should enable more precise guidelines on minimum normalising time to be derived.
Previous work, which has identified inclusions such as BN as being responsible for creep cavity nucleation in Grade 92 [8], has been taken to suggest that Grade 92 ductility may be dependent on chemical composition. This has not yet been studied in this analysis, and it is reasonable to suppose that cavitation mechanisms could play a role. However, eleven out of the eleven casts assessed in this work with long term creep ductility (average RA) values below 25% also had heat treatment LMP values below the approximate mean LMP, 48,000, Figure 10. Hence, the effects of heat treatment are clearly dominant.

Under-Normalising and Microstructure

Sawada and co-workers [9] have identified parallel but rather different problems in Grade 91, for which materials normalised for less than 30 minutes were found to be abnormally weak in creep. Sawada et al found that the soft, weak delta ferrite phase had been formed. They also showed that this was due to inadequate homogenisation during the short normalising soak, and could hence be eliminated by a longer soak.

No metallographic evidence is available on the Grade 92 materials test data collated by ECCC, but it is evident that undernormalising has caused strengthening, not weakening, in the Grade 92 materials. The mechanism is not clear, but inadequate homogenisation and / or precipitate dissolution during the short normalising soak again seems likely to be implicated. Experimental investigation is clearly warranted.

Conclusions

1. Different Grade 92 casts show large differences in long term creep ductility, varying from high to very low.
2. Low creep ductility is primarily due to inadequate normalising during manufacture, in particular short soak times, especially in thicker components.
3. Inadequate normalising of Grade 92 also promotes high (though not exceptional) tensile and short term creep strength, but long term creep rupture strength tends to be somewhat low, because reduced ductility is associated with a reduction in creep life.
4. Amongst existing Grade 92 materials, both weak, ductile (severely normalised and tempered) casts and strong, brittle (under-normalised) casts tend to show poor long term rupture strength. However, poorly performing casts can be found at all levels of manufacturing heat treatment severity.
5. There is every prospect that the widespread current concerns with low creep ductility in Grade 92 will be eliminated by correct heat treatment specification.

Abbreviations

ECCC – European Creep Collaborative Committee
MARBN – Martensitic Boron – Nitrogen steel
IBN1 – “IMPACT” project Boron – Nitrogen steel – the UK variant of MARBN
LMP – Larsen-Miller parameter LMP = T (20 + log_{10}t): T is absolute temperature and t is time (hours)
RA – Reduction of area (%)


