Aluminium–silicon carbide whisker-cast composites: some observations on the distribution, bonding and properties developed

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Errata Sheet

1) Page 12, equation (11), 1st part.
   \[
   \frac{y_0}{C_f}
   \]
   to be read as
   \[
   \frac{C_0}{2}\gamma
   \]

2) Page 14, Para. 3, line 15.
   "in plain strain"  ---  "plane"

3) Page 24, Para. 3, line 5.
   "summarized"  ---  "summarized"

4) Page 25, Para. 2, line 3.
   "summarized"  ---  "summarized"

   "fabrication"  ---  "fabrication"

   "acetylene"  ---  "acetylene"

7) Page 29, Table 4.
   "acetylene"  ---  "acetylene"

   \[
   133.3 \, \text{MN/m}^2
   \]
   ---  \[
   133.3 \, \text{N/m}^2
   \]

9) Page 41, line 1.
   (i) "supplement"  ---  "supplement"
   (ii) 4.5.2, line 3
   "ensure"  ---  "ensure"
10) Page 43, 4.5.4., line 5

1.5 m — 1.5 pm

11) Page 50, Para. 4, line 1

"Summarizes" — Summarizes

12) Page 51, 6.4 (1), line 3

"naked down" — necked-down
ALUMINIUM-SILICON CARBIDE WHISKER CAST COMPOSITES: SOME OBSERVATIONS ON THE DISTRIBUTION, BONDING AND PROPERTIES DEVELOPED

by

SAMAR CHATTERJEE, B.Met., M.Sc., A.I.M.

A thesis submitted for the degree of Doctor of Philosophy of the Loughborough University of Technology May, 1973


Department of Engineering Production
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SUMMARY
Summary

It is often said that whisker/fibre reinforcement would work, if perfect unidirectional alignment could be achieved. But this is not always true; for most structural materials, perfect unidirectional orientation is neither necessary nor desirable. In most cases, materials with isotropic properties are preferred, and, therefore, reinforcement of composites in three dimensional random orientation will be necessary.

With this view in mind, an exploratory investigation was carried out to study some aspects of whisker distribution, whisker-matrix bond and the composite tensile strength with the system SiC - Al-4.5% Cu alloy.

A common casting process, permanent mould, was considered as the composite fabrication method. A technique, to introduce and distribute the whiskers in the molten metal bath, has been proposed, which can be adopted to produce composites, reinforced in three dimensional random orientation. As evidenced from the metallographic examinations, uniform distribution of the whiskers could be achieved in the composites, when cast by this method.

Although, diffusion bonding is probable, the exact nature of the whisker-matrix interface could not be established during this investigation.

Metallographic studies, optical and electron microscopic, indicated that good wetting and bonding existed between the composite component phases.

Addition of the mixed sized whiskers was found to improve the tensile strength of the experimental matrix. The
observed improvements in tensile strength did not show any linear relationship with the volume fraction of the reinforcing phase.

Composite tensile strength could be further increased by the application of pressure during solidification.
CHAPTER I

INTRODUCTION
CHAPTER I.

Introduction

It has been recognised that no single material is capable of retaining its functional stability in applications involving both high stresses and high temperatures. Metals are eminently suited for many applications, being tough and easily fabricated, but undesirable deformation in service often limits the temperature at which they can be used. Ceramics, on the other hand, are stiff and resistant to deformation even at high temperatures. Ceramic materials are, therefore, potentially capable of withstanding high stresses over wide ranges of temperatures, but, due to their brittle nature, catastrophic failure usually occurs by rapid crack propagation, before high stresses can be attained.

A great deal of effort has been spent in attempts to combine the desirable properties of metals and ceramics in two-phase materials, i.e., 'Cermets'. To a large extent, these efforts have been unsuccessful, since the properties of the materials produced, were essentially those of the continuous phase. They were tough and deformable, or, brittle and stiff, depending on whether the metallic or the ceramic phase was continuous; one notable exception being the hard metal carbides bonded with metals which combine hardness and toughness and find extensive use as cutting tools.

There is currently a renewed interest in materials which combine the attributes of metals and ceramics. Advanced technology, particularly in the aerospace field, is demanding materials which are tough, strong, stiff and resistant to high temperatures and have low densities. Ceramic materials, in single crystal form, can withstand high tensile stresses; however, they lack toughness, i.e., the ability to absorb energy without fracture, particularly under conditions of 'shock'. It is generally accepted that these requirements can be met by composite materials in which metals are used to
confer toughness and small ceramic crystals are used to provide stiffness, refractoriness and lightness.

The development of the present generation of composite materials is based on a sound theoretical background which has been supported by a large volume of experimental work. This class of materials consists essentially of ceramic filaments, dispersed in a metal, and it is the filamentary shape of these fibres/whiskers which imparts the attractive properties to the composites. For example, when the filaments are all aligned in one direction, and the combination is stressed in that direction, both phases are essentially continuous in the direction of the stress; the ceramic component thus provides stiffness and strength while the metal provides toughness and protects the ceramics from surface damage.

A variety of whisker reinforced metal matrix systems have been studied and they have demonstrated excellent potentialities; although, the development of manufacturing techniques for successful production of large quantities of such materials are yet to be fully realized. In this context, reinforcement of aluminium matrices with long filaments is currently receiving much attention, but the whisker reinforcement of this metal matrix has been largely neglected. This is because, much of the research on whisker reinforced systems is aimed at improving the strength of metal matrices at temperatures, far in excess of the melting temperature of aluminium. This, coupled with the fact, that whiskers were not commercially available in quantity, until very recently, precluded their application in most aluminium alloy systems.

1.1. Reasons for selecting the project

The principle behind the production of suitable materials, for engineering applications, represents a delicate balance between the various useful properties like, elasticity,
strength, toughness, etc. The vast effort, already expended on the research and development of composite materials, indicates the potential economic advantages of the use of such materials, particularly, where strength/weight ratio is important. Thus design of structures for least weight has found certain outstanding applications, e.g., aircrafts, ground transportations, space activities. Of course, in addition to these, it has further implications. As for instance, for static structures like buildings, weight is normally considered to be unimportant, but, designing a least weight structure is often found to be desirable and economic.

With these applications in mind, it was considered worthwhile to undertake an investigation on the reinforcing capabilities of ceramic whiskers in an aluminium matrix. Contrary to the popular belief, that, the fibre/whisker reinforcement would not possibly work if perfect unidirectional alignment could not be achieved, the present work was started with a view that in real composites, unidirectional strengthening is neither necessary nor desirable. For example, sheets and cylinders are bidirectionally stressed and thick castings are tridirectionally stressed. In these cases, and particularly in those, where thermal stresses are induced, unidirectional orientation might be undesirable and random orientation is the solution.

Secondly, it was considered that the production of materials with high specific strengths does not necessarily mean the maximum realization of the strengthening potentialities of the whiskers. For most structural applications, some strengthening capabilities of the whiskers (tensile strengths of the order of $3 \times 10^3 \text{ MPa}$) can possibly be sacrificed in order to achieve the useful and isotropic properties in the composites.
1.2. Scope of the present investigation

Generally there are two types of reinforcement to consider:

i) Continuous filament, where the filaments completely support the load and the matrix acts as a binder, and,

ii) Discontinuous filaments, where the reinforcing material is of very short length and is well dispersed throughout the matrix (in either random or oriented manner) and serve to improve the strength and useful temperature capabilities of the matrix.

The present study is concerned with the latter method, discontinuous fibres (single crystal — whiskers) in a random orientation, for reinforcement of metals, because of its anticipated adaptability to commercial production processes and the inherent economic advantages.
CHAPTER II

BACKGROUND
CHAPTER II

Background

Reinforcement of a metal by the addition of high strength fibres (whiskers) is one of the several methods to improve the strength of metals. Metals, in their bulk state, are generally weak. It has been recognized that this weakness is due to the presence of dislocations and motion of these dislocations under stress. The apparent impossibility and the obvious impracticality of producing useful shapes that are free of dislocations (and stay free during use) have led to a number of compromises.

The general approach has been—assuming the matrix to be the load bearing phase—to impede the motion of dislocations by providing obstacles within the matrix. One effective obstacle is a grain boundary, and strengths have been increased by grain refinement. Another strengthening source is the dislocations themselves. Their interactions, when produced in high concentrations by cold working, result in considerable strengthening and hardening of the matrix. A third effective method is to disperse or precipitate a second phase to impede dislocation motion. Reduction of dislocation mobility by these techniques is utilized in commercial strengthening methods. However, elevated service temperature permits diffusion and enhances dislocation motion, by introducing the possibility of removal of these obstacles by recrystallization of the matrix or dissolution of the dispersed phases.

The mechanism of fibre reinforcement is, fundamentally, different from the metallurgical approaches, just mentioned. The matrix does not carry the load, as such; it transfers the load between the fibres. The fibres, in orders of magnitude larger than typical precipitate
particles, do not interfere, fundamentally, with dislocation motion within the matrix.

2.1. Fundamentals of Fibre Reinforcement

The theory of fibre reinforcement (1) is based mainly on the observed behaviour of composites of tungsten wires in copper (1,2), and silver (3) and of stainless steel wires in aluminium (4). Isolated data on the behaviour of other composites, such as, molybdenum wires in copper (3), silica fibres in aluminium (5), and silica fibres in copper (3), support the conclusions of the theory.

The simplest approach to a mathematical description of fibre reinforcement is to apply the theory of combined action. Experimentally, a model based on the assumption of uniform strain in the matrix and the fibre is found to be applicable. For a given deformation, the fibres develop higher stresses than the matrix within which they are contained. When both the fibre and the matrix deform elastically, the following relationship is found to hold:

\[ \sigma_c = \sigma_f \cdot V_f + \sigma_m \cdot V_m \] ..................................(1)

where, \( \sigma \) refers to the stress developed in the composite (c), fibre (f) or matrix (m), and 'V' denotes the volume fraction. It is seen that the elastic modulus (E) of the composite will be described by a similar relationship, where 'E' replaces '\( \sigma \)',

\[ E_c = E_f \cdot V_f + E_m \cdot V_m \] ..................................(2)

Further application of a load can cause plastic deformation of the matrix, so that the fibres will be strained in tension (if, \( E_f > E_m \)). The ultimate tensile strength of the composite, \( S_c \), is then given by

\[ S_c = S_f \cdot V_f + \sigma_m^* \cdot V_m \quad (V_f > V_{\text{min}}) \] ..................................(3)
where $\sigma_m$ is the stress in the matrix at the strain at which the ultimate tensile stress of the fibres, $S_f$, is reached. It has been shown that for an accurate prediction of $S_c$, the stress-strain curves for both the fibres and the matrix are required (Fig. 1).

There are numerous limitations, and, therefore, modifications are required before equation (3) can be used to predict the ultimate strength of a composite. As for instance, $S_c$ can not be less than the ultimate tensile strength of the pure matrix ($S_m$) multiplied by $V_m$. This defines a minimum $V_f$:

$$V_{\text{min}} = \frac{S_m - \sigma_m}{S_f + S_m - \sigma_m} \quad \ldots \ldots \ldots \ldots \ldots \ldots (4)$$

Similarly, for strengthening to occur, i.e., $S_c > S_m$, a critical $V_f$ must be exceeded,

$$V_{\text{crit}} = \frac{S_m - \sigma_m}{S_f - \sigma_m} \quad \ldots \ldots \ldots \ldots \ldots \ldots (5)$$

As for example, if $S_f = 2756 \text{ MNm}^{-2}$, $S_m = 115 \text{ MNm}^{-2}$ and $\sigma_m = 63.25 \text{ MNm}^{-2}$, as would be the case for an Al-5% Cu alloy reinforced with SiC whiskers, then according to the equation (5), $V_f$ must exceed 0.019% before any strengthening effect can be observed (for unidirectional reinforcement).

The stress on the composite is applied to the fibres via shear stresses at the fibre-matrix interface. Assuming again, that the fibre axis is oriented parallel to the direction of the applied stress, one can equate the mean shear load on half of the fibre surface, $\frac{\pi d (1/2) \gamma}{4}$, to the maximum load, the fibre can withstand $\frac{\sigma_f (\pi d^2/4)}{4}$.
Fig. 1. Theoretical tensile behaviour of composites.
A critical length \( l_c \) or aspect ratio \( (l/d)_{\text{crit}} \) is thus defined:

\[
(l/d)_{\text{crit}} = \frac{\sigma_f}{2\gamma} \quad \text{(6)}
\]

Thus, the \( (l/d) \) ratio of the fibre should exceed the value — ultimate fibre stress divided by twice the shear stress of the matrix. If the fibres are shorter than the critical value, the composite will fail by plastic flow in the matrix. If, they are longer, the composite should fail by fracture of the fibres.

The relationship, as shown in equation (6), indicates that a critical aspect ratio of 21.7 should be exceeded in the experimental composite system to load the whiskers, up to their ultimate fracture stress.

2.1.1. Discontinuous Fibres

Real composites, during use, even if made from continuous fibres, contain many broken ones. If whiskers are incorporated, the composite would, obviously, become similar to the one containing discontinuous (broken/chopped) fibres. Under these conditions, the load in a fibre is transferred to it via the matrix. The average tensile stress in a fibre, in such cases, is always less than that found for continuous fibres. Therefore, the strength of the composite is also found to be less than that for the continuous fibres. Fig. 2. shows that at a given volume fraction and fibre diameter, the strength increases with the length of the fibre. For very long fibres, the strength may approach that for continuous fibres, so that within experimental error, the strength might appear the same as that for continuous fibres.

When a composite, containing uniaxially aligned fibres, is stressed in a direction parallel to the fibres, then due to
Fig. 2. Tensile strength versus volume fraction of 0.2 mm diameter tungsten wires in copper tested at 600 °C. Ratio of length to diameter of the wires is given by the values of 'l/d'. (3)
the differences in elastic moduli, the axial displacement in the fibre and the matrix will be different. Shear stresses are produced on planes, parallel to the axis of the fibres and in the same direction of this axis. These shear stresses are the mechanism by which the tensile loads supported by the fibre and the matrix are distributed between these two components. The tensile stress is produced in the fibre by shear stresses at the fibre-matrix interface. As shown in Fig. 3, for a fibre of radius \( r_o \), we will have,

\[
\frac{dP}{dz} = 2\pi r_o \gamma_{rz} \quad r = r_o \quad \text{..................................(7)}
\]

where, \( P \) is the tensile load in the fibre \( (P = \sigma_{zz} A, \) where \( A \) is the area of the fibre and \( \sigma_{zz} \) is the tensile stress along fibre axis, assumed constant over the fibre cross-section \) and \( \gamma_{rz} \) is a function of both \( r \) and \( z \).

Now, considering the case of a ductile metal matrix composite, i.e., the case where the matrix is allowed to flow plastically, as the load is applied to the composite, different displacements would take place in the fibre and the matrix. Flow in the matrix means that \( \gamma_{rz} \) in the equation (7) never rises above \( \gamma \), the flow stress in shear of the matrix. When the composite is subjected to a strain, greater than the plastic yield strain of the matrix, the whole of the matrix is brought to its yield stress. \( \gamma_{rz} = r_o \) can then be set equal to \( \gamma \). If work hardening effects are small, it can be further assumed that \( \gamma_{rz} = r_o \) is independent of \( z \).

Then from equation (7), we get,

\[
P = 2\pi r_o \gamma \quad \text{.................................(6)}
\]

This means that the load on the fibre builds up linearly from its ends and can be shown as in Fig. 4. Also, since, \( P = \pi r_o^2 \sigma_f' \), where \( \sigma_f' \) is the stress in the fibre, we have from equation (6),

\[
\sigma_f' = \left( \frac{2\gamma}{r_o} \right) z \quad \text{.................................(9)}
\]
Fig. 3. The distribution of stresses of a uniaxially loaded fibre in a matrix. (3)
Now, the strain in the fibre cannot exceed the strain in the composite, 'e', so $\sigma_f'$ will build up to the value $\sigma_f e$, provided that the fibre is long enough. Since the fibre is loaded from both the ends, a fibre of length 'l' will be long enough to be strained to the strain of the composite, provided,

$$ l \geq r_0 \frac{E_f}{\gamma} \quad \ldots \ldots \ldots \ldots (10) $$

Thus, we find, that the stress builds up linearly in the fibre and is elastic. The matrix flows round the ends of the fibre, as well as, parallel to it. The strain in the matrix is mainly plastic. It will, therefore, be possible to break a fibre by plastic flow of the matrix, provided that the stress in it builds up to the fracture stress, i.e., $\sigma_f'$ in equation (9) reaches $\sigma_f$. If, 'lc' is the critical fibre length for this to occur, we have from equation (9),

$$ \frac{1}{2} \frac{r_0 \cdot \sigma_f'}{d} \quad \text{or} \quad \frac{1}{2} \frac{1}{d} = \frac{\sigma_f}{2 \gamma} \quad \ldots \ldots \ldots \ldots (11) $$

where, 'd' is the fibre diameter.

This 'lc' is defined as the "Critical Length", 'lc/2' the "Transfer Length" and the 'lc/d' the "Critical Transfer Length".

For a plastic matrix, which does not work harden, $\gamma$ is constant. If the interface fails, $\gamma$ is equal to the frictional force per unit area, which the matrix exerts on the fibre as it slides over the fibre. For work hardening materials, the value of $\gamma$ is to be substituted in equation (11) to evaluate 'lc', depending upon the plastic strain of matrix. For long fibres of length $\geq l_c$, the average strain in the matrix is equal to 'e', the strain of the composite.
Kelly and Tyson (1) have shown that $\gamma$ depends upon $\epsilon$, and the measured values of $l_c$ indicated that for work-hardening matrices $\gamma$ is to be identified with the ultimate shear strength of the matrix. Since, the average strain in the matrix is small, the non-homogeneous flow of the matrix near the ends of the fibres gives rise to marked work hardening.

In case of the discontinuous fibre reinforced composites, the tensile stress in the fibres is not uniform but builds up from the ends. The average stress in the fibres is then less than the breaking stress $\sigma_f$ when the fibre is extended to failure. The average stress is

$$\frac{1}{\ell} \int_0^1 \sigma_f' \, dz.$$  

If $\gamma$ in equation (9) remains constant, the stress builds up linearly from the ends as shown in Fig. 4. The average stress in a fibre, stretched almost to its breaking strain over the critical portion is then given by

$$\bar{\sigma} = \sigma_f \left( 1 - \frac{l_c}{\ell} \right) + \sigma_f \cdot \frac{l_c}{2} \left( \frac{1}{\ell} - \frac{l_c}{\ell} \right)$$

$$\bar{\sigma} = \sigma_f \left( 1 - \frac{l_c}{2\ell} \right)$$

$$\quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \}
Fig. 4. Expected variation of stress along a fibre within a plastic metal matrix. (3)
arranged perfectly aligned, even if they are so desired. To-date, no comprehensive experimental investigation has been reported on the variation of composite strength with the orientation of fibres in a metal matrix. There are, however some isolated observations on the effect of fibre orientation on the fracture behaviour of composites.

It has been observed that if a composite, containing unidirectionally aligned fibres, is stressed at any angle to the fibre axis, the strength can no longer be directly predicted from the classical rule of mixtures. In these cases, the other possible modes of failure are also to be considered.

When $\phi$ is the angle between the fibre axis and the direction of tensile stress, $\sigma$, as shown in Fig. 5, failure can occur by one of the following modes:

i) Flow of the matrix parallel to the fibres until the fibres fail. The stress to produce failure is then,

$$\sigma_c = \sigma \cos^2 \phi$$

.........(14)

ii) Failure of matrix in shear on a plane parallel to the fibres. The stress to cause failure will then be,

$$\gamma' = \sigma \sin \phi \cos \phi$$

.........(15)

where $\gamma'$ is the ultimate shear stress of the matrix.

iii) Failure of the matrix by flow transverse to the fibre. The necessary condition to cause failure, will become,

$$\sigma' = \sigma \sin^2 \phi$$

.........(16)

where, $\sigma'$ is the ultimate tensile strength of the matrix in plain strain, provided the fibres are spaced far enough apart not to alter the properties of the matrix by contributing a dispersion strengthening effect.

Experimental studies of the tensile anisotropy of fibre composites have been made with chromium whisker reinforced copper (6), Al$_3$Ni whisker reinforced aluminium (7) and with a few other systems.
Fig. 5. Schematic representation of a composite containing aligned fibres oriented at an angle $\phi$ to the tensile axis.
Although chromium whisker content (1.6 %) was insufficient to significantly affect the strength of copper, Hertzberg (6) was able to delineate different fracture behaviour for three ranges of fibre orientations. When the composites were tested parallel to the direction of whisker alignment, the whiskers were found to fail in tension followed by ductile opening and link-up of the voids, resulting from each fractured whisker. At 45 degrees, failure occurred by shear in the matrix parallel to the whiskers. At 90 degrees, the fracture surface assumed an elliptical shape with the major axis parallel to the whiskers.

George et al. (7) concluded that the presence of whiskers (Al$_3$Ni) provided considerable strengthening of the aluminium matrix, even when oriented perpendicular to or at a large angle to the tensile axis. Although, deformation of the matrix was primarily by shear, the whiskers were considered to interfere with most of the active slip systems.

2.2. Interface

From the foregoing discussion, it is apparent that the behaviour of a composite is dependent upon, and, in many cases can be predicted from the properties of the constituent phases — the fibre/whisker and the matrix. A third entity, the interface, although vanishingly small in size in most composites, normally plays an important role in the behaviour of the material during service.

For the purpose of this discussion, the 'interface' will be defined as the region separating the fibre/whisker from the matrix. This interface can be formed by a mechanical locking or a chemical bond between the phases. It can consist of a reaction layer and, therefore, contain one or more phases. It can also consist of more than one interface, as in the case of composites containing coated fibres. Any
detailed picture of these interfaces is beyond the scope of the present discussion, since this would constitute a subject for research on its own. However, a less magnified view of the interfaces has been considered to explain their role in the behaviour of fibre composites.

2.2.1. Function of the Interface (Mechanical)

The rule of mixtures, to a first approximation, describes the modulus and the tensile strength of unidirectional fibrous composites, in the direction of fibre alignment. Although not explicit in equations (1) and (2), the reinforcing mechanism implied by the rule of mixtures assumes certain properties for the interface, the absence of which negates the validity of these relationships. It is assumed that when the composite is loaded in tension, parallel to the fibre-axis, the fibre and the matrix strain together in a uniform manner. However, this can only be accomplished if the interfacial bond is adequate to allow load transfer, by shear, at the interface. In such cases, the matrix can be envisioned as gripping the fibre along its interface near the ends, thus applying an axial load to the fibres.

The chief function of the interface is, therefore, that of transferring the load from the matrix to the fibres.

2.2.2. Chemical Function of the Interface

Since the major purpose of the interface is one of load transfer, it is important that the matrix is well bonded to the fibres, over the entire surface. The bond should be sufficiently strong to prevent rupture when the matrix deforms plastically around the fibres or when thermal and other stresses arise. In case of incomplete bonding over the surface of the fibre, due to poor contact between the
fibre and the matrix, the effective transfer length would obviously be reduced. Thus, a longer fibre will be required to compensate for the regions along the fibre surface, where contact with and bonding to the matrix is poor.

In order to maximize the bonding between the fibres and the matrix, it is first necessary to have the two mating surfaces in intimate contact. This means that the fibres must be effectively wetted by the matrix. However, it should be noted, that wetting is not the only criterion for bonding. To achieve a good bonding, the necessary conditions for wetting are to be fulfilled. For example, some metals that wet ceramics at elevated temperature, do not adhere to them at room temperature (8). The lack of adhesion at room temperature is usually due to the fact that the resulting thermal stresses are greater than the strength of the interfacial bonds. Other studies (9) have shown that sessile drops of liquid metals do not adhere to the ceramic substrate when interfacial reactions are absent. However, it was also noted, during these studies, that pronounced adherence was normally characterized by interfacial reactions. These were, presumably, due to the formation of interfacial bonds that were stronger at room temperature than the residual stresses at the interface.

It appears that both wetting and chemical reaction, at the interface, may be necessary to achieve the desired bond strength; however, the reaction must not be so severe as to weaken the fibres. Thus the chemical function of the interface is to provide a proper balance between, (i) the wetting and (ii) the chemical reaction between the fibre and the matrix to render adequate bonding.
2.2.3. Wetting and Fibre-Matrix Contact

For a good bond to develop, the surface of the fibres should be wetted by the matrix. Wetting implies a mutual attraction between the atoms of the solid (fibre/whisker) and the liquid (matrix) phase. When these mutual attractions are greater than the cohesive forces within the liquid, wetting of solid will occur. The degree of wetting depends upon the relative values of the surface energies of the solid-liquid ($\gamma_{SL}$), the solid-vapour ($\gamma_{SV}$) and the liquid-vapour ($\gamma_{LV}$), as shown in Fig. 6. The degree of wetting can be determined by the contact angle ($\theta$) between a drop of liquid resting in static equilibrium on a solid surface.

In order to have a liquid to wet (to spread) a solid surface the condition,

$$\gamma_{SV} > (\gamma_{LV} + \gamma_{SL})$$

should be satisfied. In ceramic-metal systems, many ceramics have low values of $\gamma_{SV}$ relative to the value $\gamma_{LV}$ for the metals. As a result, many ceramics are not wetted by molten metals. For the purpose of illustration, the system Ag - Al$_2$O$_3$ will be considered.

From the available data (9), it can be seen that $\gamma_{SV}$ (Al$_2$O$_3$) ($\sim 970 \times 10^{-3}$ Jm$^{-2}$) is close to that of $\gamma_{LV}$ (Ag) ($\sim 925 \times 10^{-3}$ Nm$^{-1}$). However, this in itself is not sufficient to predict wetting (non-wetting of Al$_2$O$_3$ by pure Ag was observed in vacuum at 1100 °C). The contact angle was found to be 120°. The composites, formed by pressure casting of silver into a mould containing bundles of parallel arrays of whiskers, showed reasonable penetration into the channels, but the actual contact between the whiskers and the matrix were very poor. The strength data indicated little or no reinforcement of silver as shown by the lower curve in Fig. 7. The
wetting, $\theta < 90^\circ$  
non-wetting, $\theta > 90^\circ$

$\gamma$ = Surface energy (Jm$^{-2}$)  
For liquids, surface tension (Nm$^{-1}$)

subscripts,  
S, L and V = solid, liquid and vapour  
$\theta$ = contact angle

Fig. 6. Surface energy relationship between a liquid and a solid in static equilibrium.
Fig. 7. Tensile strength of silver reinforced with coated and uncoated whiskers of Al₂O₃. (9)
1. whiskers coated (metallized)
2. whiskers uncoated
(whisker orientation parallel to tensile axis)
composite fracture was characterized by gross pull out of whiskers, with little or no silver adhering.

The wetting difficulties were overcome by vapour depositing a thin coating of nickel. The nickel coating provided the whiskers with a higher effective surface energy (\( \sim 1730 \times 10^{-3} \text{N} \cdot \text{m}^{-1} \) at 1470°C) which could be effectively wetted by molten silver.

2.2.4. Stability of Fibre-Matrix Interface

One of the most important factors which determines the stability of a fibre composite, particularly at elevated temperature, is the chemical and the physical nature of the interface. This is important during both fabrication and subsequent uses of the composite.

Interfacial instability can take the form of a chemical reaction between the two phases which may weaken the reinforcing phase or a physico-chemical reaction which results in spheroidization or agglomeration of the reinforcing phase (10). These two types of instability can be differentiated by the fact that the former can reduce the composite strength markedly without any noticeable change in the microstructure, whereas, the latter can result in a substantial microstructural change with no change in the composition or amount of the phases present. Of course, both these types of instability can occur in the same system, but for the purpose of this discussion, they can be dealt with separately.

Chemical interaction has been found to substantially reduce the fibre strength during the fabrication of tungsten wire reinforced copper alloys (11), silica fibre reinforced aluminium (5) and boron fibre reinforced aluminium (12). In
addition, serious interfacial chemical reaction, during elevated temperature exposure has been observed in boron-aluminium composites (13-15).

Chemical instability, during fabrication, can be minimized by choosing the fibre and the matrix materials which are compatible or by adjusting the fabrication parameters. During elevated temperature use, the interfacial reaction can be reduced by providing a diffusion barrier, between the fibre and the matrix. Materials, such as metal carbides, borides, nitrides, silicides and oxides are suitable for this application (16). A thin layer of silicon carbide has been reported (17) to be sufficient to protect boron filaments in aluminium and titanium matrices. This superior stability of silicon carbide over boron in contact with liquid aluminium has, in fact, led to the development of 'BORSIC', i.e., silicon carbide coated boron fibre.

Physico-chemical instability occurs in systems in which the phases are chemically inert with respect to each other; but, because of the large amount of energy associated with the large interfacial areas between the phases, spheroidization or agglomeration of the fibres might occur. This type of behaviour has been reported (18) to occur, relatively rapidly, even at moderate temperatures in nickel and cobalt alloys, containing whiskers of silicon nitride, aluminium oxide and silicon carbide. It is due to the fact that separately grown fibres, when introduced in a matrix, will normally have arbitrary interfaces (and generally high energy) with the matrix. If one could arrange each fibre in the matrix such that there were good atomic matching across the interface, the specific interfacial energy would be low even though the interfacial area would still be large. This can, however, be accomplished in the unidirectionally solidified eutectic composites (10,19). It has been reported that the structure of the Al₃Ni whisker
reinforced aluminium composites remains unchanged even after 500 hours of exposure at 500 °C (about 85% of its melting temperature). At higher temperatures, the structure does coarsen, but the low energy interfaces persist, and the structure remains that of a whisker composite without any apparent degradation of tensile strength (10).
CHAPTER III

SURVEY OF LITERATURE
CHAPTER III
Survey of Literature

Data on whisker reinforced composites are rather limited at the time of this writing. This is due to the fact that the whiskers are fairly expensive and because the handling techniques and the methods for incorporation of whiskers into suitable matrices are still in the process of development. Furthermore, details of current and interesting developments are, in most cases, withheld from the engineering community for proprietary reasons.

However, mainly on the basis of the articles, of a review nature, it appears that considerable research has been carried out on model whisker composites and in systems where continuous filaments have been cut into short lengths to approximate the behaviour of whiskers.

Of all the fibre-reinforced systems, metals reinforced with ceramic whiskers (single crystals) have been subjected to, by far, the greatest degree of speculation regarding the properties that might be expected from the composite materials (20,21,22).

The initial efforts (23) were largely concerned with the fundamentals of wetting and bonding of single crystals in a variety of composite systems. Various fabrication techniques were explored and occasional tensile testing was carried out with the successfully fabricated composite specimens. Alumina, Silicon Nitride and Silicon Carbide whiskers were the most popular whiskers for these experimentations, both because of the ease of acquisition and because of their superior properties.

As a preliminary to fabrication studies, sessile-drop tests were carried out in $\text{Al}_2\text{O}_3 - \text{Ag}$ system (24). The contact angle and the surface tension of molten silver in contact
with alumina were used to assess the wettability of the system and the force necessary to shear the metal bead from the alumina disc was considered as a measure of the bond strength. These results indicated that a coating of a metal, such as platinum or nickel is necessary to promote wetting.

Although, hot pressing was considered as a method of composite manufacture, all the fabrications were carried out by using centrifugal force, or, vacuum or gas pressure to force molten metal through the whisker bundle (25). During the fabrication studies, it was recognized that the main factors affecting the quality of composites were:

i. mould length/diameter ratio
ii. volume of metal flowing through the whiskers
iii. metal flow rate and dynamics
iv. mould geometry
v. capillary shape and size of pores between the whiskers, prior to infiltration
vi. nature of fibre surface (wettability)
vii. composition of metal phase (flux, dross, etc.)
viii. mould and whisker temperature
ix. infiltration pressure (atmospheric or other)

Earlier investigations established the importance of metallizing before infiltration, as no strengthening of silver was obtained with upto ～16% uncoated whisker composites where all the fractures showed gross whisker pull out from the matrix. Fig.8, shows the effect of whisker concentration and size on the room temperature strength of the composites. The deviation from linearity of the plot for the 'mixed-sized' whisker composites at ～16% (vol.) was attributed to the difficulties encountered during the fabrication of higher vol.-% whisker composites, rather than to any inherent fundamental weakness in the reinforcement process (24).

Investigations, carried out at the Horizons Inc., Cleveland, Ohio, (24) were mainly directed towards the development of
Fig. 8. Room temperature tensile strengths of silver reinforced with alumina whiskers. (4)
high strength composites of nickel- and iron- base matrices with alumina whiskers, for potential uses in missiles, spacecrafts or aircrafts. Initial studies related to the applicability of conventional powder metallurgical techniques for incorporating and suitably aligning the whiskers into nichrome (80:20) were not successful to produce high strength composites. In this process, the whiskers were broken from the as-grown wool, and together with metal powder and a wetting agent were dispersed in water and filtered. The mass was then broken up, mixed with 0.1% methyl cellulose as a binder and 25% water and extruded at a ratio 16:1 to align the whiskers. After prolonged drying at 130°C, the extrusion product was sintered and finally cold rolled to the form of rods.

As reinforcement was not reproducible, when fabricated by powder metallurgical methods, alternative techniques involving heating of the compacted mixture to temperatures above the melting point of the matrix were attempted (26). At this stage, the effects of liquid phase wetting agents were also examined. The addition of calcium chloride was proved to be a definite aid to improve wettability. The actual fabrication of test specimens, consisted of rapid melting of pre-extruded composites or mechanical mixtures using the halide salt as a flux. Composites, produced by this method exhibited tensile strengths of about 2067 MPa, a 25 - 30% increase over the blank control.

Sutton and Feingold (27) studied the effects of impurity atoms on the wetting and bonding between Al₂O₃ and nickel. Their observations on all the three active-metal doped specimens (Ni/Gr, Ni/Ti and Ni/Zr) were reported to be similar and are summarized as

i) The active metal (diluent) atoms segregated from the previously homogeneous solid solution and concentrated in the region of metal-sapphire interface.

ii) Chemical attack of the sapphire substrate in each case
was found to have occurred under the solidified drop (the solid-liquid region) and at the periphery of the drop (the vapour-solid region).

iii) New crystalline species were developed in the vicinity of the interface.

iv) Diffusion of the active metal into the sapphire plaque was observed.

The above steps in the active-metal doped Ni-Sapphire bonding can be shown schematically as in Fig. 9. In table 1, various properties of the bonded systems are summarized and compared.

Mehan (28) reported on some aspects of fabrication and evaluation of sapphire whisker reinforced aluminium composites. The composites, he prepared, were initially coated with a duplex coating of nickel and titanium, orienting the coated whisker bundle and infiltrating the bundle with liquid aluminium. Evaluation of the composites was primarily conducted by elevated temperature tensile testing at 500°C. Composite strengths, as high as 378.26 MNm⁻² at 500°C, with about 35% volume of whisker addition were reported.

Some interfacial studies were also conducted to assess the quality of the bond. It was reported that the electron microscopic examinations failed to indicate any presence of weak interfacial bond either between the whisker and the coating or between the coating and the matrix. The absence of any void at the interfaces was interpreted as an indication of good wetting. Good interfacial bonding was characterized by fibre fracture, when the composites were tested in tension at room temperature. No fibre pull out was observed during the study. However, it was reported that fibre pull out did occur, in cases of elevated temperature tensile testing, which was attributed to the insufficient fibre lengths and low matrix shear strengths. This is because (that even during these pull outs) the fibres were coated with metal indicating a strong whisker-coating bond.
Table 1.

Observed properties of Active metal doped Nickel/Sapphire Bonds (27)

<table>
<thead>
<tr>
<th>Property</th>
<th>Cr</th>
<th>Ti</th>
<th>Zr</th>
</tr>
</thead>
<tbody>
<tr>
<td>Contact Angle $\theta^{**}$, deg.</td>
<td>108</td>
<td>95</td>
<td>136</td>
</tr>
<tr>
<td>Interfacial Energy, Jm$^{-2}$</td>
<td>1470</td>
<td>1090</td>
<td>1900</td>
</tr>
<tr>
<td>Shear Strength$^{***}$, MNm$^{-2}$</td>
<td>172.25</td>
<td>33.072</td>
<td>-</td>
</tr>
<tr>
<td>Interfacial Adhesion</td>
<td>Medium</td>
<td>Very</td>
<td>Very</td>
</tr>
<tr>
<td>Strong</td>
<td>Strong</td>
<td>Strong</td>
<td>Weak</td>
</tr>
<tr>
<td>Interfacial Reaction</td>
<td>Weak</td>
<td>Very</td>
<td>Very</td>
</tr>
<tr>
<td>Weak</td>
<td>Very</td>
<td>Strong</td>
<td></td>
</tr>
<tr>
<td>New crystalline phases at the interface</td>
<td>Yes</td>
<td>Yes</td>
<td>Yes</td>
</tr>
<tr>
<td>(ZrO$_2$)</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

* Concentration of Cr, Ti and Zr diluent = 1 atomic percent.

** Sessile drop test conducted at 666.61 x 10$^{-6}$ Nm$^{-2}$ pressure, 1500 ± 20°C, 30 min.

*** Shear strength of solidified drop on Al$_2$O$_3$ plaque at room temperature.

**** 0.01 atomic percent.
Fig. 9. Steps in bonding active metal 'x' doped Nickel to Sapphire. (27)
As a preliminary step in the whisker reinforcement, Parratt (18,29) at the ERDE, used silicon nitride whiskers to reinforce silver. The following three steps were involved in producing the composites: (i) grading the whiskers, (ii) incorporation of a matrix, and (iii) moulding the mixture of the powdered matrix and whiskers to form a dense solid.

Experimental results were reported on composites, containing 15% whisker in a two dimensional random array, which on the basis of Cox's analysis (52) Parratt suggested to be equivalent to 5% aligned whiskers. The tensile testing results showed that although silver was strengthened from 172.25 MNm⁻² to 275.6 MNm⁻², the elastic limit was reduced somewhat. This reduction was attributed to the stress concentration at fibre ends producing premature plastic flow in these regions, at stresses lower than the overall macroscopic elastic limit of the matrix. The elevated temperature properties indicated that although the composite retained its strength to higher temperatures than the control matrix, the fall-off was more than could be accounted for, on the basis of the combined action theory. The explanation, put forward by Parratt, that the whiskers were of an aspect ratio lower than the critical at any temperature of test, was justified on the basis of fibre pull out observed in these cases. Attempts were also made to improve the elevated temperature properties by adding 1% silicon to the matrix, to improve liquid phase (eutectic AgSi) sintering at 630°-840°C. Although, some improvements in strengths were reported, no explanation was put forward to justify, whether the observed improvement was the result of better wetting or due to efficient fabrication.

Since 1966, considerable research (30) has been carried out at the Kelpar Inc., Falls Church, Virginia, on the silicon carbide whisker reinforced composite systems. Initial
investigations were concerned with the feasibility studies to establish the suitability of SiC whiskers to reinforce Al- and Ni- base alloys. Although, the aluminium base alloys were found to be chemically compatible with the whiskers, nickel appeared to degrade them at about 538°C.

The composites were prepared by vacuum sintering of whisker-metal powder cakes at temperatures 550 -650°C and at a pressure of 133.32 x10⁻⁶ Nm⁻² followed by hot rolling at about 550°C. Two aluminium base alloys were chosen during these studies — Al- 10.2%Si and Al- 5%Cu. Selection of the Al-Si alloy was made on the basis of their previous success with this alloy with Al₂O₃ whiskers. The 5%Cu-Al alloy was chosen on the basis of the published reports related to the improvements in wetting characteristics due to the presence of copper. The results, reported by them, have been summerized in table 2.

These results indicated that the Al- 5%Cu alloy exhibited no reinforcement. On the basis of the observed uniform whisker distribution, it was concluded that the high pore density was responsible for the lowering of the strength values. The limited strengthening, observed in case of the Al- 10.2%Si alloy was believed to be an effect of improved soundness, as indicated by the density values.

Reporting a separate programme of study, the same workers, Lare, Divecha and Hahn (31) mentioned that further studies indicated the compatibility of silicon carbide whiskers with several alloys of nickel, titanium and iron and with pure magnesium, at temperatures upto their respective solidus temperatures.

The compatibility studies of the AZ 31B Mg alloy (Al-3%, Zn-1%, Mn-0.2%, Mg-Bal.) and the Al-Si alloys with SiC whiskers were conducted directly on the fabricated composites. Microstructural evidence indicated the SiC whiskers to be
Table 2.

Properties of Aluminium base SiC whisker composites consolidated in air by induction hot pressing (\%)  

<table>
<thead>
<tr>
<th>Composition</th>
<th>Hot pressing temp.(°C)</th>
<th>Hot reduction temp.(°C)</th>
<th>Density theoretical (%)</th>
<th>U.T.S. (Kgfmm(^{-2}))</th>
</tr>
</thead>
<tbody>
<tr>
<td>Al-5%Cu Control</td>
<td>570-590</td>
<td>544-549</td>
<td>70</td>
<td>91.5</td>
</tr>
<tr>
<td>+ 10% SiC (vol)</td>
<td>&quot;</td>
<td>&quot;</td>
<td>&quot;</td>
<td>&quot;</td>
</tr>
<tr>
<td></td>
<td>&quot;</td>
<td>&quot;</td>
<td>68</td>
<td>91.4</td>
</tr>
<tr>
<td>Al-10.2%Si Control</td>
<td>585-595</td>
<td>370-390</td>
<td>70</td>
<td>99.2</td>
</tr>
<tr>
<td>+ 10% SiC (vol)</td>
<td>&quot;</td>
<td>&quot;</td>
<td>&quot;</td>
<td>&quot;</td>
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<td></td>
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</table>
stable in these alloys above their solidus temperatures.

During these studies, the agehardenable aluminium alloy matrix composites were also briefly examined. The consolidation characteristics were found to be excellent, although, failure of the matrix to respond to conventional ageing treatments was observed. However, no details of these investigations have yet been published.

It has been reported that at the MITRON Research & Development Corp. of Waltham, Mass., Rosenberg and his co-workers are actively engaged in the development of ceramic whisker reinforced cast metal matrix composites. The main attraction of their work is in the fact that, it is possibly the first effort towards the production of real composites. The investigations, undertaken by them, aim at producing cast composites, reinforced in three dimensions, by randomly oriented whiskers. The reported programmes are unique in a sense that:

i) It is the first step to bridge the gap between the making of high strength laboratory scale composites and the production of useful composites for engineering applications.

ii) To overcome the obvious limitations of the directionally reinforced composites, attempts are made to impart isotropic properties to useful composites, and,

iii) Simple fabrication process like, casting, is being explored to economically produce components of useful shapes and sizes.

MITRON has developed (32) a reinforcement technique which consists of (i) introducing ceramic whiskers into a molten metal alloy, (ii) distributing them in the melt, and, (iii) pouring the whisker containing molten alloy into a mould cavity to form a cast shape. However, no further details have been disclosed for proprietary reasons.

The reinforcement obtained for the Al- 4.5\%Cu alloy using
Al₂O₃, SiC and AlN-Al₂O₃ whiskers showed a positive deviation from the values, that could be predicted from the rule of mixtures. The addition of 0.043% by volume of Al₂O₃ whiskers (TKF Type 3B) doubled the as-cast yield strength. Addition of 0.047% vol. of SiC whiskers (TKF Type 5B) nearly doubled the as-cast yield strength, increased the tensile strength and produced a significant increase in ductility. AlN-Al₂O₃ whiskers (TKF Type 6A), when added in 0.063% vol. showed an increased yield and ultimate tensile strength, as well as, a near three-fold increase in elongation when compared to the control sample. Table 3, lists the results reported by them, which were obtained with various whisker materials, introduced into the experimental alloy.

Based on the optical microscopic examinations, it was concluded that the 'stitching' effect produced by the whiskers at the grain boundaries served as a reinforcing network among the primary grains. The reason for the unusually high ductility, observed during the investigation, could not, however, be justified by the authors.

In another programme (33), Rosenberg and Ryan evaluated the the previously reported technique for introducing ceramic whiskers into molten cobalt base alloys and studied the effects of composition variations on the properties of these composites. Tensile testing was performed to evaluate the high temperature reinforcing capabilities of SiC and Al₂O₃ whiskers. The test results (Table 4 and 5) showed an increase in strength. Maximum strengthening was available when the whiskers, coated with carbon by using an acetelene torch, were used.

During this investigation, electron probe microanalysis technique was tried to study the aspects of interfaces in the SiC whisker reinforced composites. Their inability to
### Table 3

Mechanical properties of the experimental Aluminium-4.5% Cu alloy (32)

<table>
<thead>
<tr>
<th></th>
<th>0.2% Yield (MN m⁻²)</th>
<th>U.T.S. (MN m⁻²)</th>
<th>Elon. %</th>
<th>% Whisker By vol.</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Control (as-cast in machined graphite mould)</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>1. Control</td>
<td>59.94</td>
<td>144.69</td>
<td>4</td>
<td>0</td>
</tr>
<tr>
<td>2. Al₂O₃ (a)</td>
<td>119.20</td>
<td>149.51</td>
<td>1</td>
<td>0.043</td>
</tr>
<tr>
<td>3. SiC (b)</td>
<td>104.73</td>
<td>167.43</td>
<td>7</td>
<td>0.047</td>
</tr>
<tr>
<td>4. AlN-Al₂O₃ (c)</td>
<td>66.13</td>
<td>184.75</td>
<td>11.6</td>
<td>0.063</td>
</tr>
</tbody>
</table>

(Values in solution treated and age-harden condition: 532 °C for 1 hour, Water quench, 149 °C for 3 hours.)

<table>
<thead>
<tr>
<th></th>
<th>0.2% Yield (MN m⁻²)</th>
<th>U.T.S. (MN m⁻²)</th>
<th>Elon. %</th>
<th>% Whisker By vol.</th>
</tr>
</thead>
<tbody>
<tr>
<td>1. Control</td>
<td>179.63</td>
<td>227.37</td>
<td>3.8</td>
<td>0</td>
</tr>
<tr>
<td>2. Al₂O₃</td>
<td>175.70</td>
<td>233.57</td>
<td>1.1</td>
<td>0.043</td>
</tr>
<tr>
<td>3. SiC</td>
<td>220.48</td>
<td>254.93</td>
<td>1.3</td>
<td>0.047</td>
</tr>
<tr>
<td>4. AlN-Al₂O₃</td>
<td>175.70</td>
<td>223.24</td>
<td>2.5</td>
<td>0.063</td>
</tr>
</tbody>
</table>

(a) Al₂O₃ whiskers, 10-30 μm diam. by 125-2500 μm length.
(b) SiC whiskers, 1-5 μm diam. by 20-1000 μm length.
(c) AlN-Al₂O₃ whiskers, 3-30 μm diam. by 30-600 μm length.
### Table 4

<table>
<thead>
<tr>
<th>Test Temperature, °C</th>
<th>U.T.S., kN/m²</th>
<th>Remarks</th>
</tr>
</thead>
<tbody>
<tr>
<td>816</td>
<td>19.53</td>
<td>Control.</td>
</tr>
<tr>
<td>816</td>
<td>28.94</td>
<td>5 gms. Al₂O₃ (1)</td>
</tr>
<tr>
<td>816</td>
<td>29.25</td>
<td>5 gms. SiC (1)</td>
</tr>
<tr>
<td>816</td>
<td>34.55</td>
<td>5 gms. Al₂O₃ (1)</td>
</tr>
<tr>
<td>982</td>
<td>7.92</td>
<td>Control</td>
</tr>
<tr>
<td>982</td>
<td>30.59</td>
<td>5 gms. Al₂O₃ (1)</td>
</tr>
<tr>
<td>982</td>
<td>16.56</td>
<td>5 gms. SiC (1)</td>
</tr>
<tr>
<td>982</td>
<td>56.84</td>
<td>5 gms. SiC (2)</td>
</tr>
<tr>
<td>982</td>
<td>6.11</td>
<td>10 gms. SiC (1)*</td>
</tr>
</tbody>
</table>

* High porosity

(1) Lamp Black film coating

(2) Carbon film coating with acetelene torch

SiC whiskers — 0.5 - 3 μm dia. and 100-750 μm length.

Al₂O₃ whiskers — 6 μm dia. and 60-1250 μm length.

Typical melt — 2.5 kg.
### Table 5.

<table>
<thead>
<tr>
<th>U.T.S.</th>
<th>Yield Strength</th>
<th>Elongation (%)</th>
<th>Remarks</th>
</tr>
</thead>
<tbody>
<tr>
<td>(MNm⁻²)</td>
<td>(MNm⁻²)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>125.4</td>
<td>91.64</td>
<td>12</td>
<td>10 gms. SiC (1)</td>
</tr>
<tr>
<td>101.28</td>
<td>79.92</td>
<td>3</td>
<td>&quot;</td>
</tr>
<tr>
<td>131.6</td>
<td>69.59</td>
<td>9</td>
<td>Control</td>
</tr>
<tr>
<td>117.82</td>
<td>66.83</td>
<td>7.5</td>
<td>10 gms. SiC</td>
</tr>
<tr>
<td>188.1</td>
<td>77.17</td>
<td>20</td>
<td>10 gms. SiC (1)</td>
</tr>
<tr>
<td>181.9</td>
<td>66.14</td>
<td>11.6</td>
<td>10 gms. Al₂O₃</td>
</tr>
<tr>
<td>156.4</td>
<td>79.24</td>
<td>8.3</td>
<td>10 gms. Al₂O₃ (1)</td>
</tr>
<tr>
<td>160.54</td>
<td>95.08</td>
<td>8.5</td>
<td>15 gms. SiC (1)</td>
</tr>
<tr>
<td>195.68</td>
<td>99.91</td>
<td>13.5</td>
<td>25 gms. SiC (1)</td>
</tr>
<tr>
<td>179.83</td>
<td>98.53</td>
<td>13.5</td>
<td>25 gms. SiC (1)</td>
</tr>
</tbody>
</table>

(1) Lamp Black film coating

SiC whiskers — 0.5 - 3 μm dia. and 100-750 μm length
Al₂O₃ whiskers — 6 μm dia and 60-1250 μm length.

Typical melt — 2.5 kg.
determine the presence of the whiskers with EPM, in the as cast structure implies the extreme experimental difficulties, these micron sized whiskers normally present. It is to be noted that a probe diameter of 1 \( \mu \text{m} \) was tried and the whiskers were of 0.5 - 3.0 \( \mu \text{m} \) in diameter and 100 - 750 \( \mu \text{m} \) in length. These aspects of the experimental difficulties, when experimenting with the commonly available electron optical instruments, will be discussed in some detail in chapter no VI.
CHAPTER IV

EXPERIMENTAL PROCEDURE
CHAPTER IV

Experimental Procedure

This research was undertaken with a view to studying the distribution of whiskers, nature of the whisker-metal interface and the properties of metal-matrix composites, reinforced with randomly oriented ceramic whiskers. An aluminium alloy was chosen as the experimental matrix, for the wide applicability of aluminium as a structural material. Selection of the whisker — SiC — was primarily dictated by commercial availability and cost considerations.

4.1. Composite System

4.1.1. Matrix

The aluminium-base alloy, LM 11, was selected as the matrix material. The choice of this alloy was primarily on the basis of the published data (23,30,34,35) that copper improves the wetting characteristics of silicon carbide with molten aluminium, without any apparent degradation of the whiskers.

LM 11, a copper bearing (4 - 5% Cu) commercial casting alloy, is normally used in the solution treated or age-hardened condition. The high shear strength and ductility of this alloy are useful in redistributing stress and arresting the leading edge of the propagating crack during plastic deformation. The certified chemical composition and some useful properties of this alloy are given in Table 6.
The table below provides the chemical composition of the experimental alloy (LN 11).

<table>
<thead>
<tr>
<th>Element</th>
<th>Cu</th>
<th>Mg</th>
<th>Si</th>
<th>Fe</th>
<th>Mn</th>
<th>Ni</th>
<th>Zn</th>
<th>Pb</th>
<th>Sn</th>
<th>Ti</th>
<th>Al</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>4.55</td>
<td>.01</td>
<td>.18</td>
<td>.23</td>
<td>.01</td>
<td>.01</td>
<td>.02</td>
<td>.01</td>
<td>.01</td>
<td>.13</td>
<td>Bal.</td>
</tr>
</tbody>
</table>

Table 6a. Chemical composition of the experimental alloy (LN 11).

<table>
<thead>
<tr>
<th>Condition</th>
<th>Alloy BS 1490 (1963)</th>
<th>LM 11(W)</th>
<th>LM 11(WP)</th>
<th>LM 11(M)*</th>
</tr>
</thead>
<tbody>
<tr>
<td>SAND</td>
<td>0.1% Proof Stress</td>
<td>154.4</td>
<td>200.8</td>
<td>59.94 (+)</td>
</tr>
<tr>
<td></td>
<td>Tensile Strength</td>
<td>262.6</td>
<td>283.4</td>
<td>144.7</td>
</tr>
<tr>
<td>CAST</td>
<td>Elongation (%)</td>
<td>10</td>
<td>5</td>
<td>4</td>
</tr>
<tr>
<td></td>
<td>Hardness (BHN)</td>
<td>75</td>
<td>95</td>
<td></td>
</tr>
<tr>
<td>CHILL</td>
<td>0.1% Proof Stress</td>
<td>154.4</td>
<td>200.8</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Tensile Strength</td>
<td>293.4</td>
<td>339.8</td>
<td></td>
</tr>
<tr>
<td>CAST</td>
<td>Elongation (%)</td>
<td>15</td>
<td>10</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Hardness (BHN)</td>
<td>85</td>
<td>100</td>
<td></td>
</tr>
</tbody>
</table>

* Ref. 32  (+) 0.2% Proof Stress

Table 6b. Some useful properties of the alloy, LN 11.
4.1.2 Whisker

Silicon Carbide whiskers were chosen for this experimental investigation. Although, from the available data, the mechanical properties of SiC whiskers do not appear to be well characterized, there is little doubt about their superior properties, when compared to the available high strength materials.

Some of the useful properties of these single crystal whiskers have been presented in Table. 7. The certified properties of the whiskers, used during investigation, supplied by Metals & Methods Ltd., Slough, Buck., are listed in Table. 7. Fig. 10 (a) & (b) are the representative photomicrographs of these whiskers.

4.2 Experimental Set-up

4.2.1 Mould Assembly

The mould assembly, designed, to carry out the casting operation has been shown in Fig. 11.

The die, previously used during the programme (36) on the solidification of aluminium alloys under pressure, was modified for making castings of a different shape. The actual shape of the casting is shown in Fig. 12. The choice of this particular shape of the casting was made for the following reasons:

i) to observe the effectiveness of pressure in consolidating metal in the enlarged section of the die cavity and at a distant part from the piston end.

ii) to provide suitable locations for specimens to be taken from a direction, transverse to the direction of the load, applied during solidification.
### Table 7

(A) Some useful properties of SiC Whiskers. (*)

<table>
<thead>
<tr>
<th>Property</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Maximum Tensile Strength</td>
<td>$20 \times 10^3$ MNm$^{-2}$</td>
</tr>
<tr>
<td>Young's Modulus</td>
<td>$68 \times 10^4$ MNm$^{-2}$</td>
</tr>
<tr>
<td>Specific Gravity</td>
<td>$3.2 \times 10^3$ kg.m$^{-3}$</td>
</tr>
<tr>
<td>Melting Temperature</td>
<td>$2600$ °C</td>
</tr>
</tbody>
</table>

(B) Certified properties of the whiskers, used during this investigation.

- **Supplier**: Metals & Methods Ltd., Langley, Slough, Buck., SL3 6EF.
- **Grade**: S-M 1
- **Size**
  - i) Diameter: $0.1 \mu m - 1 \mu m$ approx.
  - ii) Length: $5 \mu m - 50 \mu m$ approx.
- **Purity**: $99.5\%$
- **Method of inspection**
  - i) Size: Electron Microscope

Fig. 10. Photomicrographs (SEM) of the SiC Whiskers.

a. x 2000

b. x 1000
Fig. 11. Experimental Die Set-up.
Fig. 12. Line diagram of the experimental casting.
iii) to reuse the existing die by the necessary alterations, that could be carried out, fairly easily, with the facilities available within the department.

It should be noted, that, although no work was carried out to evaluate the critical size (length/diameter ratio) of the castings, from previous experience (36) it was known that the castings of the chosen configuration could be successfully consolidated.

The details of the die material, die-design and heat treatment schedule are given in Appendix I.

4.2.2 Control of Temperature

Die heating was carried out by four equispaced cartridge heaters. These heaters, having a maximum working temperature of 800 °C, were controlled by an energy controller to maintain the desired die temperature of 200 °C.

4.3. Composite Fabrication Technique

4.3.1 General Considerations

Previous investigation has already established (37,38) that the soundness of the cast products can be greatly improved by the application of pressure, on the liquid metal during solidification. This, coupled with the published evidence, that, in most cases the full strengthening capabilities of the high strength fibres could not be realized due to the presence of gross porosity - in the matrix and in some cases preferentially at the fibre-matrix interface, did indicate that solidification of whisker composites under pressure would undoubtedly be of additional advantage. Initially, it was also thought (37) that the application of pressure would
alter the whisker-metal surface energy relationships as well, and, therefore, might be of use in improving the wettability, even in the case of the less compatible systems. However, it was soon realized that there was no simple experimental procedure to verify the latter hypothesis.

4.1.2 Exploratory Attempts

The project was undertaken, on the anticipation, that some published information would be available on the fabrication aspects of cast composites, reinforced with randomly (three dimensional) oriented whiskers. However, it became obvious that no such process does exist, at least to the extent that could be used during this investigation.

As a preliminary attempt, an injection gun, similar to the one used by Badia and Rohatgi (39) was built with the object of injecting the whiskers into the molten metal bath, prior to casting. During the initial trials with various powdery materials, e.g., graphite, clay, metal powders, this injecting gun worked reasonably well. However, the actual difficulties were not encountered till the time, the whiskers could be tried with this gun. It was intended that the whiskers would be introduced in the liquid metal bath by means of a nitrogen gas stream. It was found that the gas pressure, sufficient to cause a steady bubbling action in the metal bath, induced a swirling motion to the whiskers inside the glass container. This, in effect, caused the whiskers to stick to the sides of the glass container due to the surface charges, resulting from the frictional forces. To overcome this problem, the following steps were taken, singly or jointly, but without any success.

i) Metallizing the inside surface of the glass container.
ii) Vibrating the glass container by mechanical and ultrasonic means.
iii) Trying different shaped containers to give the least possible resistance to the flow of the whiskers.

It was also noted, that the static surface charges, caused the whiskers not only to stick to the wall of the glass container, but to each others, as well, resulting in tiny clusters of whiskers. It was, therefore, realized that the separation of these whiskers, i.e., 'individualization', is of prime importance. In case of perfectly individualized whiskers, the above mentioned injection method should work satisfactorily, provided,

i) The whiskers are metallized, so that no surface static charge can be formed. (During this programme this possibility was ruled-out because of the anticipated cost involved in doing so.), and,

ii) By making necessary alterations of the injection gun (tip) and working with a melt of greater depth, to promote the transfer of the whiskers (individualized and metallized) from the gas bubble to the melt.

Although, not very relevant in this particular context, it is worth mentioning that the transference of particles from the gas bubbles to the melt mainly depends upon,

(a) the probability of their coming to the bubble-melt interface, during the rise of one such bubble, becomes more, smaller the diameter of the bubble, greater the depth of the metal bath and slower the rate of rise of these bubbles, and,

(b) the energy considerations to make the transfer favourable, i.e., choice of a coating material which is compatible with the molten metal.

However, it was considered at this stage that some other method, to introduce the whiskers in the molten metal bath, would be necessary to make the experimental composites.
Section 4.3.3 Modified Process

From the experience, gained so far, it was believed that a process could possibly be developed with the following objectives:

i) Whiskers are to be separated from each other.

ii) The density/specific gravity values of the silicon carbide whiskers (density $3.17 \times 10^3$ kg.m$^{-3}$) and molten aluminium alloy (density $2.9 \times 10^3$ kg.m$^{-3}$) are very close. Also, due to the very minute sizes, the apparent density of the whiskers would be somewhat lower than the above value. As a result, the settling of the whiskers in the molten metal bath would largely depend upon the available time. Therefore, by proper manipulation of the experimental condition, they can be kept in suspension in the metal bath for certain length of time, provided they are wetted by the molten alloy.

iii) The surface energy of SiC ($\gamma_{SV}$ at $1200 \degree C \sim 840 \times 10^{-3} J m^{-2}$) (9) and molten aluminium ($\gamma_{LV}$ at $800 \degree C \sim 665 \times 10^{-3} N m^{-1}$)(40) being very close, it might be possible to achieve wetting by allowing time to attain equilibrium at the appropriate temperature. Secondly, accepting the reported effect of copper in improving the wetting of SiC by molten aluminium, one must allow sufficient time, so that diffusion of copper from the melt to the surface of the whiskers could occur.

iv) Providing some means to keep the whiskers within the metal bath in a well separated and individualized condition.

A process was, therefore, developed which consists of the following steps:

i) Production of composite compacts from the whiskers and metal powders.

ii) Introduction of the compacts into the molten metal bath
iii) Pouring the metal, containing whiskers, into the mould cavity followed by the application of pressure, if and when desired.

Certain basic steps, which seem self-evident, are required in the conversion of bulk whisker material to the individualized condition, in a random mixture (mechanical) of solid metal powders.

4.3.4 Dispersion media and method

Separation of individual whiskers from the as-grown mass, in which they are randomly oriented, involves dispersing them in a suitable fluid so that the solid-solid forces of attraction between the whiskers become inoperative.

Wettability of SiC whiskers
Achievement of satisfactory dispersion of a given solid in a liquid is usually dependent upon finding those liquids which will wet the surface of the solid or, alternatively finding a surfactant which renders the solid and liquid interfaces compatible. If little or no wetting is present, then the liquid will not flow into the pores of the whisker-mass to displace the gas phase and to relieve the forces of attraction at the solid-solid contact points.

Silicon Carbide whiskers are readily wetted by water and can be satisfactorily dispersed simply by stirring in a beaker with a glass rod. No surfactant or modifying agents (ions) were required in the experiments with water dispersions.

Relative Densities of System Components
The density of silicon carbide is $3.17 \times 10^3$ kg.m$^{-3}$ at room temperature and hence there is a fairly large tendency for the whiskers to settle, when suspended in water or other low density liquids.
The dimension of these whiskers are large enough, in most cases, so that Brownian diffusion does not overcome the gravitational forces. Of course, there are not many liquids which have densities approaching that of silicon carbide. Those which do, are usually uncommon, and were not considered to be absolutely necessary during these experiments.

Dispersion of the Whiskers
Initially, the required amount of whiskers were weighed accurately. They were, then, dispersed in the preselected liquid medium. A mixture of iso-propyl alcohol and water (80:20) was used as the dispersing medium. To assure freedom from clumps and clusters, that exist in the as-received whisker-mass, the dispersion was performed in a variable speed laboratory mixer. The as-grown wools or mats of silicon carbide whiskers, in general, required severe agitation to break the clusters. In most cases, the stainless steel blade of the mixer was replaced with a simple stirrer made of hard rubber. This was to ensure that no whisker damage could occur to reduce the aspect ratio.

Co-dispersion of Metal Powders
Atomized aluminium powders of average size of 200 μm were used for this purpose. Following the dispersion of the whiskers, the metal powders were added and the entire mixing procedure was repeated.

4.3.5. Formation of Whisker-Metal Compacts

After the uniform co-dispersion of the whiskers and the metal powders, they were filtered through a Buchner funnel and under a reduced pressure. The whole idea was to complete the filtering operation, within the time, the suspension was in motion and before any differential settling or segregation could occur. The filtered mass was then thoroughly dried at room temperature followed by an oven
drying at 100 °C. The dried mass, comprised of whiskers, randomly oriented in a two dimensional array, within the powdered metal matrix.

The dried mixture was compacted into briquettes. Finally, these briquettes were degassed under pressure of 133.3 MNm^{-2} and at a temperature of approx 450 °C.

4.3.6 Melting and Casting

Commercial aluminium alloy, LM 11, of certified chemical composition, was used throughout this investigation. All test castings were made from the same metal stock and were cast under identical work environmental conditions. The procedure for casting test specimens was as follows:

i) Melting 0.75 kg. of alloy, LM 11, in a Galenkamp electric resistance furnace.

ii) Holding the metal at the treatment temperature, 750 - 800 °C.

iii) Degassing the metal with nitrogen gas.

iv) Addition of preformed briquettes, to give the desired whisker volume fraction.

v) Holding the compact, within the melt, until it dissolves completely. Normally, 35-45 mins. were found necessary during the experiments.

vi) Mechanical mixing of the metal bath, prior to pouring, to avoid segregation of the whiskers.

vii) Pouring the metal, containing whiskers in a random orientation, into the mould cavity, maintained at a preselected temperature of 200 °C.

viii) Application of the preselected load on the liquid metal, in case of castings solidified under pressures, higher than atmospheric.
ix) Repeating procedures (i) to (viii) for different casting conditions, on the basis of whisker volume fraction and/or solidification pressure.

A total time of 25 to 30 seconds was required to bring up the desired load on the liquid metal. Thermal analyses, during the initial experimentations, indicated that under the identical condition, load could be successfully applied on the liquid metal.

4.4. Selection of Tensile Specimens and different Metallurgical Tests

Two locations were chosen from the cast blanks for making tensile test specimens. Normally, two specimens were prepared from each casting — one from the longitudinal and the other from the transverse direction of the load, applied during solidification. The actual locations of the test specimens in the cast blanks are shown in Fig. 13.

Metallographic examination of the polished specimens, taken from the different locations of the cast blanks, were carried out in the unetched and etched conditions. Normal Optical Microscopic and Scanning Electron Microscopic (SEM) observations were made on the distribution of the whiskers in the as cast structure, and the nature of the whisker-matrix interface.

Studies of the composite tensile fractures were carried out with the SEM to correlate the observed features related to the whisker distribution and their failure behaviour to the nature of the interface and their reinforcing capabilities.

The chemical nature of the whisker-matrix interface was examined by Electron Probe Microanalyser (EPM) and Energy Dispersive Spectrometer (EDS). Some X-ray diffraction
Fig. 13. Location of the tensile test specimens.
(powder) work was also undertaken to supplement the EPM/EDS observations, related to the nature of the interface and the extent to which chemical reactions could alter, if any, the stability of the whiskers.

A limited number of thin foil specimens, prepared from the cast composites, were examined by Transmission Electron Microscope (TEM). A conventional 100KV microscope and, at a later stage, a million-volt microscope were used for this investigation.

4.5 Experimental Techniques

4.5.1 Tensile Testing

Testing of tensile specimens was conducted in a Hounsfield Tensometer, using a motor drive to give a 0.0715 mm/min. crosshead speed. Complete load-extension curves were recorded for each test. Specimens were tested for ultimate tensile strengths.

4.5.2 Metallography (Optical Microscopy)

Initial metallographic polishing was done by hand in rotary grinder, using silicon carbide papers. Besides the usual precautions, care was also taken to insure that the whiskers were not damaged. Polishing through 600 grit paper provided a flat matrix surface; however, the whiskers were found to be projected above the plane of matrix. The specimens were then polished with 6 μm and 1 μm diamond paste, followed by polishing with magnesia. Minimal pressure was applied to the specimen during the final polishing, since excessive pressure resulted in erosion of the matrix adjacent to the whiskers.

The specimens were examined by a Vickers Photoplan microscope, up to the magnification of 1250, in both polished
and etched conditions. The etching solution used was: 65 ml H₂O, 25 ml HNO₃ and 10 ml HF.

4.5.3 Scanning Electron Microscopy

For composite fractured surface studies, a Cambridge 'Stereoscan' scanning electron microscope was used. The great depth of field made possible the direct examination of the irregular topography of composite fracture surfaces from various angles without replication.

In this instrument, a beam of electrons is focussed to a spot of 70 -100 Å in diameter on the sample surface and is scanned over the surface in a raster of 1000 to 2500 lines. This primary beam causes the sample to emit secondary electrons. Both the back-scattered and the secondary emission electrons are collected and their image is amplified to modulate a cathod ray tube (CRT), scanning in synchronisation with the primary beam. The ratio of the length of the scanning line on the CRT to that on the sample surface determines the magnification. Magnifications from 200 to 100,000 times can be obtained with this instrument. The variation in the number of secondary electrons, collected, is a function of the surface contour; as a consequence, the displayed image resembles the optical appearance of the surface.

The aluminium - silicon carbide composites could be studied without much difficulty and no preparation of the fracture surfaces was necessary. Apart from the fractured surfaces, polished specimens were also studied with this microscope. This was primarily to utilize the high magnification capabilities and to study the aspects of whisker orientation, relative to the plane of polish, in case of deeply etched specimens.
4.5.4. **Electron Probe Microanalysis**

Electron beam microanalysis method was used to study the distribution of different elements in the cast structure. In performing this analysis, a beam of electrons is accelerated with a high potential and focused by means of electromagnetic lenses to a diameter of about 1.5 μm onto the surface of the specimen. As in an x-ray tube, the specimen in this case, will act as a primary source of x-rays. A continuous or white spectrum is produced, as well as fluorescent x-rays, characteristic of the elements excited by the electron beam. A chemical analysis/identification of the elements, present in the excited area, can be carried out by analysing the characteristic x-ray lines by means of a single crystal x-ray spectrometer. A low power optical microscope is used for observing the specimen and positioning the area of interest, under the electron beam.

Some of the electrons are back scattered from the specimen and detected by a scintillation counter. This signal is used to form an image on one of the display tubes. The scattered electron image shows the topographical detail, similar to an optical image, formed by oblique illumination, and is useful for identifying the region, scanned by the electron probe. Since heavier atoms scatter more electrons, this image also indicates the variation in atomic number of the elements, present on the specimen surface.

The characteristic x-ray line from any single element, on the specimen surface, can be selected by the x-ray spectrometer and used to form an x-ray image on the other display tube, giving immediate semi-quantitative information about the distribution of the chosen element, over the specimen area scanned.
The electron and the x-ray images are normally displayed side-by-side for comparison. These images can also be photographed for permanent record. Linear concentration displays can be photographed, alone or superimposed on the electron image.

Quantitative measurements are made by comparing x-ray peak intensities with those from the pure elements (standards) and/or with empirical or theoretical working curves.

4.5.5 Energy Dispersive Spectrometer

Some work was carried out with an energy dispersive spectrometer, in conjunction with the SEM. Principally, unlike the use of single crystal wavelength dispersive spectrometers, as in EPM, a Li drifted Si semiconductor is used as the radiation detector.

X-rays, impinging on the detector, create free charge-carrier by photoelectric absorption and subsequent impact ionization. The number of these charge-carriers is proportional to the energy of the incident x-rays. This input is then fed to a multichannel analyser (MCA). In the MCA, the voltage pulses are separated on the basis of amplitude and stored in the memory channel, corresponding to a particular energy increment. The resulting spectrum can then be displayed on a CRT or x-y strip chart recorder, printed out by a high speed printer, placed on punch paper or transferred directly to a computer.

4.5.6 X-ray Diffraction

Virtually every crystalline solid has a unique crystal structure and gives a characteristic x-ray diffraction pattern. During this investigation, a Philips X-ray
Diffractometer was used. The samples, for examination, were ground to fine powders. A small quantity is then pressed onto a lightly greased half microscope slide, ready for insertion into the diffractometer. The diffraction pattern, obtained, takes the form of a series of peaks at different values of $2\theta'$, characteristic to different crystal structure. It is necessary to convert these $2\theta'$ values to interplaner spacings using the Bragg relationship, $\lambda = 2d \sin \theta'$, where, $\lambda$ is the wavelength of particular radiation, 'd' is the interplaner spacing and ' $\theta'$' the Bragg angle. Normally, tables are provided for this conversion. The relative intensities of each reflection is read from the diffractometer trace, usually by assigning a value of 100 to the most intense reflection.

The unknown compound can now be identified from the ASTM index, which catalogues substances on the d-values of the three most intense reflections. These three d-values, their corresponding intensities, the name and the formula of the compound are all listed. In addition, a reference number is given which refers to a card index where the complete diffraction pattern of each substance is given.

4.5.7. Transmission Electron Microscopy

Investigation, using the TEM, was aimed to study the nature of the whisker-matrix interface.

The waves associated with moving electrons are diffracted according to the Bragg condition, just as are x-rays. The wavelength, $\lambda$, of the electrons is given by de Broglie equation, $\lambda = h/p$, where, 'h' is the Plank's constant and 'p' is the momentum of the electrons. Thus, a working accelerating voltage of 100 kV yields a wavelength of only 0.037 Å. In a thin 'perfect' single crystal specimen, the absorption of the electron wave is expected to be uniform overall and no contrast will be observed on the image formed on the zinc sulphide screen of the microscope. But, wherever the periodicity of the crystal lattice is sufficiently
perturbed by the presence of defects or otherwise, the Bragg condition is not obeyed. As a result, the diffraction contrast will show up the presence of any such irregularity.

In an electron microscope, the electrons are emitted from a hot tungsten source and accelerated at the anode of the electron gun through the working potential. After being focussed onto the specimen in a parallel beam by magnetic condenser lenses, the beam is transmitted through the thin foil and diffracted in a number of directions by the crystal. Each diffracted beam is brought to a focus in the back focal plane of the objective lens. The resulting diffraction pattern may be suitably magnified and projected onto the fluorescent screen for photographic recording. Apertures of variable sizes may be inserted below the objective lens, when an image of the lower surface of the specimen is formed. When the aperture is centred on the main transmitted beam, bright field illumination is obtained, since the diffracted beams do not contribute to the image. A perfectly flat, defect-free crystal will then give a homogeneously bright (uniformly illuminated) image. Any irregularity or defect in the specimen changes the path of the diffracted beam, causes interference with the transmitted beam, and produces variations in the intensity of the bright field image. The contrast may also be reversed to give dark field illumination by tilting the electron gun so that the aperture is centred over one of the diffracted beams.

The foils used in transmission electron microscopy are typically about 2000 Å thick. Materials with high atomic number absorb electrons most effectively, and, therefore, must be prepared in thinner sections.

During this investigation, thin foil specimens were prepared by a two-stage electropolishing technique. For this purpose, a 'Polaron' thin foil specimen preparation unit was used.
The experimental procedure consisted of the following steps:

1) The M 400 unit (Fig. 14.a) was used to part off 0.25 mm thick discs from the 3 mm diameter specimen rods.

2) The discs were ground down to about 0.15 mm thickness. The unit - 401 A - was used for this purpose.

3) The disc blanks were then profiled or dished by electrolytic jetting in the Polaron M 401 electropolishing unit (Fig. 14.b., right hand cell).

4) The profiled discs were finally electropolished in a static bath (Fig. 14.b., left hand cell) to produce a small perforation surrounded by thinned material.

The polishing conditions and the electrolytes used were as follows:

1) **Profiling Cell**

   Electrolyte — 20 per cent. HNO₃ in water.

   Working condition — i. jet to specimen distance = 5 mm
   
   ii. voltage - 20 v.
   
   iii. current - 0.2 A.
   
   iv. time - 45 secs. each side.

2) **Perforation Cell**

   Electrolyte — 300 gms. copper nitrate
   
   900 ml. methyl alcohol
   
   30 ml. nitric acid

   Working condition — i. voltage - 5 v.
   
   ii. current - 0.7 A.
   
   iii. time - 7 mins.
(a) M 400 unit for parting off 0.25 mm thick discs.

Fig. 14. Polaron electropolishing unit, used to prepare the thin foil specimen for T.E.M.

(b) M 401 unit for profiling and perforating the discs.
CHAPTER V

EXPERIMENTAL RESULTS
CHAPTER V

Experimental Results

5.1. Tensile Strength

Composites, tested in tension, exhibited improved strengths, when compared to those for the blank control. Strengths of the experimental composites have been presented by two sets of graphs.

Figs. 16 to 20, show the variation of tensile strength with the solidification pressure, at any constant whisker volume fraction. For any particular volume (%) of whiskers, the tensile strength increases with increased solidification pressure; although, the rate of increase does not follow any linear relationship.

In Figs. 21 to 24, the composite strengths have been plotted against the whisker volume (%), the solidification pressure remaining constant. The strength of the composites increased with the increase in the whisker volume fraction.

5.2. Distribution

Microscopic examination - optical and SEM - of the polished specimens (Figs. 25 - 37) indicate that the silicon carbide whiskers were uniformly distributed in the as cast structure. The whiskers were oriented in all possible modes in the grain boundary regions. Deeply etched specimens, examined with SEM, showed whisker distribution in third dimension, with respect to the plane of polish. The nature of whisker distribution, in three dimensional random orientations, is typified by the fractograph in Fig. 39.
Fig. 16. Effect of solidification pressure on the tensile strength of the alloy, LM11 (Control).
Fig. 17. Effect of solidification pressure on the tensile strength of the composite containing 0.025 vol. per cent. of SiC whiskers.
Fig. 18. Effect of solidification pressure on the tensile strength of the composite containing 0.075 vol. per cent. of SiC whiskers.
Fig. 19. Effect of solidification pressure on the tensile strength of the composite containing 0.25 vol. per cent. of SiC whiskers.
Fig. 20. Effect of solidification pressure on the tensile strength of the composite containing 0.5 vol. per cent. of SiC whiskers.
Fig. 21. Effect of whisker volume fraction on the tensile strength of composites solidified under atmospheric pressure.
Fig. 22. Effect of whisker volume fraction on the tensile strength of composites solidified under a pressure of 68.9 MNm$^{-2}$. 
Fig. 23. Effect of whisker volume fraction on the tensile strength of the composites solidified under a pressure of 137.8 MNm$^{-2}$. 
Effect of whisker volume fraction on the tensile strength of the composites solidified under a pressure of 206.7 MNm\(^{-2}\).

Fig. 24. Effect of whisker volume fraction on the tensile strength of the composites solidified under a pressure of 206.7 MNm\(^{-2}\).
The following observations were made during the study:

1) Whiskers were, primarily, observed in the grain boundary regions of as-cast structures.

2) In most cases, they were associated with the copper-rich phase in the inter-dendritic regions.

3) On the basis of their appearance on the polished structure, the longer whiskers were, in a majority of cases, lying across the grain boundaries.

4) In some cases, (Figs. 28, 37), the whiskers continued across the grains from one boundary to the other.

5) Some of the whiskers were well within the primary aluminium grains, without any association with the copper rich phase, (Figs. 33, 34, top region).

6) On a macroscopic scale, whisker distribution did not vary measurably, from region to region in the casting.

7) Whiskers tended to form a 'stitching pattern' around the grains in the cast structure, (Figs. 32, 33).

8) Whiskers were distributed at random in three dimensions as shown in Fig. 39.

5.3. Bonding

The whisker-matrix bond was examined with EPM and EDS in conjunction with SEM. During the investigation, it became clear that under the operating conditions, the attainable resolutions with these instruments were inadequate to identify the sub-micron sized whiskers (dia. 0.1 - 1 μm) in the aluminium alloy matrix. In the characteristic x-ray distribution images, silicon concentration was never (except in the case of Fig. 41) found to be above the background level. The distribution of Al and Cu (Figs. 42, 43) were also found to be continuous, i.e., in regions where the
whiskers were visible on the optical images. This is contrary to expectations, provided that the resolution was adequate. It is evident, therefore, that the resolving power was insufficient to make any positive identification in the interface regions.

Optical microscopic studies of the polished specimens, at magnifications of up to 1200, failed to indicate any whisker matrix interaction.

Examination of the thin foil specimens, from the composites, with transmission electron microscope, further supported the optical microscopic observations. No surface reaction, of any kind, could be identified during these studies. Fig. 44 shows the absence of any discontinuity between the whisker and the matrix, either due to interaction or void formation. This observation also supports the view, that, during fabrication good wetting condition were obtained between the liquid metal and the whisker surfaces. Also, in this particular instance, the bonding appeared quite different from a simple mechanical bond. In a mechanical bond, a line of separation between the whisker and the matrix could possibly be expected at a magnification as high as 80,000, (Fig. 44.b).

Table. 8, summarizes the observations made during a brief x-ray powder diffraction study, to supplement the EMP work. From this table, it can be seen that the presence of SiC whiskers could not be identified in the composite powder. It was, however, due to the small volume fraction of the whiskers and due to their preferential segregation in the composite powder (harder materials form finer powders). As a result, it was considered worthwhile to leach away aluminium from the matrix and to extract the whiskers and other insolubles. Diffractometer traces, obtained after leaching, showed fairly strong peak for SiC. The presence of SiC (as observed during this study) and the inability to
Table 8.

X-ray Diffraction Studies

Instrument - Philips Diffractometer  
x-ray - Cu Kα radiation

Operating condition

Scanning speed rate - 10°/min  
Scale factor - i) counter - 16  
ii) rate meter - 2  
Rate meter - i) time constant (sec) - 4  
ii) multiplier - 1  
Amplifier - Scintillation counter and Discriminator

<table>
<thead>
<tr>
<th>Control (LM11)</th>
<th>Intensity Control (1)</th>
<th>Intensity SiC (1)</th>
<th>d (Å)</th>
<th>Anticipated compound (2)</th>
</tr>
</thead>
<tbody>
<tr>
<td>44°47'</td>
<td>V.S</td>
<td></td>
<td>2.0381</td>
<td>CuAl₂</td>
</tr>
<tr>
<td>38°37'</td>
<td>V.S</td>
<td></td>
<td>2.3453</td>
<td>Al</td>
</tr>
<tr>
<td></td>
<td></td>
<td>35°45' S</td>
<td>2.5321</td>
<td>SiC</td>
</tr>
<tr>
<td></td>
<td></td>
<td>35°37' S</td>
<td>2.5363</td>
<td>SiC</td>
</tr>
<tr>
<td>20°40'</td>
<td>W</td>
<td></td>
<td>4.3533</td>
<td>β FeSi</td>
</tr>
</tbody>
</table>

(1) Intensity - V.S - very strong; S - strong;  
M - medium; W - weak; V.W - very weak.

(2) From ASTM powder data file.
## Observed Data (2θ')

<table>
<thead>
<tr>
<th>Powdered Composite</th>
<th>Intensity (1)</th>
<th>Extracted Material (1)</th>
<th>'d' (Å)</th>
<th>Anticipated Compound (2)</th>
</tr>
</thead>
<tbody>
<tr>
<td>47°45'</td>
<td>W</td>
<td></td>
<td>1.916</td>
<td>CuAl₂</td>
</tr>
<tr>
<td>44°47'</td>
<td>V.S</td>
<td>44°47' V.S</td>
<td>2.0379</td>
<td>CuAl₂</td>
</tr>
<tr>
<td>44°45'</td>
<td>V.S</td>
<td>36°30' M</td>
<td>2.0381</td>
<td>CuAl₂</td>
</tr>
<tr>
<td>35°30'</td>
<td>V.W</td>
<td>35°30' W</td>
<td>2.5425</td>
<td>SiC</td>
</tr>
<tr>
<td>34°30'</td>
<td>V.W</td>
<td></td>
<td>2.6143</td>
<td>SiC</td>
</tr>
<tr>
<td>20°42'</td>
<td>M</td>
<td></td>
<td>4.3535</td>
<td>β-Fe₃Si</td>
</tr>
</tbody>
</table>

(1) Intensity - V.S - very strong; S - strong; M - medium; W - weak; V.W - very weak.

(2) from ASTM powder data file.
identify any other compound lend further support to the optical and the electron microscopic observations, already made.

5.4. Fracture Studies

Figs. 45 to 64, show some features of the tensile fractures in the composites, when examined with a scanning electron microscope. During the course of this investigation, the following observations were made:

1) Fractures, in general, were characterized by typical 'dimple fracture' which occur by formation of and the subsequent coalescence of microvoids in the naked-down regions of the test specimens. Coalescence of the microvoids appeared to have taken place by a process of internal and localized necking.

2) Non-isotropic flow of the matrix material was observed in the close vicinity of the whiskers (Fig. 60).

3) Detailed study of the fractured surfaces revealed that both shear and tensile modes of failures were operative.

4) The shear mode of fracture was associated with the elongated dimple pattern.

5) The fibrous tensile region consisted of a disordered array of mounds and troughs.

6) The association of SiC whiskers with these troughs on the fracture-surface (Figs. 55,56) indicated the possibility that failure occurred after fracture had taken place in the whiskers.

7) The fractured surfaces of the whiskers, aligned parallel (or near parallel) to the tensile axis could be clearly identified (Figs. 54,55).

8) The fractured surface of whiskers, aligned at about 45 degrees to the fracture plane were identified (Figs. 57-59).
9) In some cases, pull-out of whiskers occurred (Figs. 39, 51-53). Careful examinations, however, revealed that the exposed whisker ends were evenly coated with the matrix material (Fig. 39).

10) Whiskers, positioned at about 90 degrees to the stress axis, were found to be associated with a pronounced elliptical shaped void, formed as a result of deformation during stressing the test piece. Further examination indicated that these whiskers were oriented parallel to the major axis of the elliptic section, (Fig. 61), indicating that the anisotropic plastic flow in the matrix was probably due to the position of the whiskers. This is in keeping with the reports with the previous investigators regarding the general anisotropic flow characteristics of the matrix material, when reinforced with whiskers.

11) In some cases, (Fig. 58), whiskers were found lying loose on the fractured surfaces. These observations indicate that with the whiskers, positioned at about 90 degrees to the tensile axis, interfacial failure (whisker/matrix interface) is the prevalent mode rather than the fracture of the whiskers.

12) Isolated observations were made on the phenomenon of whisker rotation during plastic deformation. In some cases, it would appear that small whiskers, on the fractured surfaces, rotated to align themselves parallel to the fracture plane (Fig. 58, top right corner).

13) In all cases, whiskers were found coated with the matrix material indicating good interfacial bond. Also, the matrix shear markings, slightly away from the whisker-matrix interface further supplemented the view that the composites failed without any apparent decohesion of the interface.

It should be noted, at this stage, that the studies of the fracture behaviour were carried out mainly to establish the
adequacy of the bond between SiC and the matrix, and to establish that the whiskers were being loaded during testing and not with a view to carry out a critical evaluation of the fracture behaviour of the matrix material.
Fig. 25. Whisker distribution in polished and unetched specimen.

x 500, magnified 1.5 times.

Fig. 26. Whisker distribution in polished and etched specimen.

magnified 1.5 times.

a. x 200
Fig. 26. b. x 500

Fig. 27. Polished and etched specimen. x 500, magnified 1.5 times.
Fig. 28. Polished and etched specimen.

magnified 1.5 times
Fig. 29. Polished and etched specimen.

magnified 1.5 times
c. x 800

d. x 1200
Fig. 30. Polished surface, SEM photograph.
mode - secondary electron
x 680

Fig. 31. Polished surface, SEM photograph.
x 2750

a. mode - secondary electron
b. mode - reflected electron

c. mode - specimen current
a. mode - secondary electron

Fig. 32. Whisker distribution around the cast grain showing a 'stitching' pattern.  

x 1400

b. mode - reflected electron
c. **mode** - specimen current

---

**Fig. 33.** Composite containing c.25 vol. per cent. of whisker and solidified under 137.6 MNm$^{-2}$ pressure. x 700

a. **mode** - secondary electron
b. mode - reflected electron

c. mode - specimen current
Fig. 34. Upper region of the Fig. 33, showing whiskers inside the aluminium grain without any association with CuAl$_2$. x 1400

b. mode - reflected electron
Fig. 35. Composite containing 0.025 vol. per cent. of whiskers and solidified under atmospheric pressure.
x 265

a. mode - secondary electron
b. mode - reflected electron

c. mode - specimen current
a. mode - secondary electron

Fig. 36. Composite containing 0.075 vol. per cent of whiskers and solidified under atmospheric pressure. 
\( \times 2650 \)

b. mode - reflected electron
c. mode - specimen current

Fig. 37. Composite containing 0.075 vol. per cent of whiskers and solidified under atmos. pr. showing a whisker across the grain. x 1300

a. mode - secondary electron
b. mode - reflected electron

c. mode - specimen current
Fig. 38. Polished and deeply etched specimen examined with SEM.

x 1300

Fig. 39. Fractograph showing whisker pull-outs.

x 10,000
Fig. 40. Deeply etched specimen showing partly exposed whiskers.
Fig. 40.c. x 14,000

Fig. 40.d. x 26,000
(a) electron image

Fig. 41. E.P.M Photograph
x 1500

(b) Si, x-ray distribution image
Fig. 1.2. E.D.S. Photograph  
\[ x \times 6500 \]

(a) electron image

(b) Al. x-ray image

(c) Cu. x-ray image
Fig. 43. (a) E.D.S. Photograph, (b) Al. x-ray image x 5500, (c) Cu. x-ray image
Fig. 44. T.E.M. Photograph
(Examined with a million volt microscope, working at a potential of 400 kV)

(a) x 50,000
Fig. 45. x 115, beam angle - 45°.

Composite Fractured Surface

Fig. 46. x 550
(a) $x$ 16.8, beam angle - 45°

Fig. 47. Composite Fractured Surface

(b) $x$ 168, beam angle - 45°
(a) \( x \, 430 \), beam angle - 45°

Fig. 48. Composite Fractured Surface

(b) \( x \, 860 \), beam angle - 45°
Fig. 48(e). x 1720, beam angle - 45°

Fig. 49. Composite Fractured Surface

(a) x 150, beam angle - 90°
Fig. 49.b. x 1680, beam angle - 90°

Fig. 49.c. x 4200, beam angle - 90°
(a) x 900, beam angle - 90°

Fig. 50. Composite Fractured Surface

(b) x 1680, beam angle - 90°
Fig. 51. Fractographs showing whisker pull-outs
Fig. 52. Pulled out whiskers on the fractured surface.

x 11,000

Fig. 53. Exposed whisker end indicating good wetting between the whisker and the matrix.

x 10,500
(a) $\times 3,000$

Fig. 54. Fractured whiskers.

(b) $\times 9,000$
Fig. 55. Whisker fracture and matrix flow markings.
Fig. 56. Association of the fractured whiskers with the troughs on the fracture surface.

(a) $\times 2,100$

(b) $\times 10,500$
Fig. 56.e. x 5,250

Fig. 57. Fracturing of whisker, aligned at $45^\circ$ to the plane of fracture.

x 10,000
Fig. 58. x 5,500

Composite Fractured Surfaces.

Fig. 59. x 5,400
Fig. 60. Fractograph showing non-isotropic flow of the matrix in the close vicinity of the fractured whiskers.

(a) \( \times 1,100 \)

(b) \( \times 5,500 \)
(a) x 2,200

Fig. 61. Fractograph showing non-isotropic flow of the matrix around the whiskers, lying perpendicular to the direction of the applied stress.

(b) x 5,500
(a) x 2,100

Fig. 62. Whisker pull out and fractured whiskers

(b) x 5,200
Fig. 63. x 2,200

Fractograph showing matrix deformation markings near the fractured/pulled out whiskers.

Fig. 64. x 5,500
CHAPTER VI

DISCUSSION AND CONCLUSIONS
CHAPTER VI

Discussion and Conclusions

As the title of this project implies, this experimental work was undertaken as an exploratory effort to study some aspects of whisker reinforcement in metallic matrices. It is wellknown that this field of technology is fairly new and still in its early stages of development. Most of the relevant work has, so far, been done in government (defense) organisations (mostly in the U.S.A and some in this country), and, therefore, in general their findings are not available for general use. While discussing the experimental results, it is, therefore, considered worthwhile to mention and analyse all the details noted during this investigation, including those which did not appear to be directly rewarding during this programme, to keep all aspects of this exploration open for use by future workers.

6.1 Distribution

On a macroscopic scale, the whiskers were found to be randomly distributed in the cast structure. The presence of the whiskers was also markedly characterized by their close association with the copper bearing phase, \( \text{CuAl}_2 \). Photomicrographs 25 to 37, show the whisker distribution in a two dimensional random array. The whiskers appear to have all the different possible orientations, with reference to the grain boundaries. Their orientations varied from a fully aligned configuration to a completely across-position, subtending an angle of 90 degrees to the grain boundaries. In some cases, they were lying across the primary aluminium grains as well (Figs. 30-33). The true randomness of their distribution became evident during the subsequent SEM studies.
of the polished and deeply etched specimens (Figs. 38, 40) and the fractured surfaces (Fig. 39).

Although, almost all possible orientations of whiskers were observed in individual specimens, on the basis of the large number of specimens examined, the following generalization is possible. With the increased rate of solidification and/or the increased length of the whiskers, whiskers showed an increased tendency to lie across the grain boundaries.

In this context, the effect of whisker length appears to be related to what can be best described as the 'accommodation' capability of the grain boundaries. The shorter the whiskers, the greater would be the chance for their accommodation within the available length of the grain boundaries. As the whisker length increases and/or the grain size decreases, some restraint would, obviously, be imposed on these whiskers. This effect would become, even more pronounced, as the whisker population goes up. When all these conditions would be operative, whiskers would have configurations which would show departure from the usual alignment, and in extreme cases, they might occupy areas well within the grains. Figs. 33 and 34 illustrate one such case. It will be observed, that in this micro-region of interest, the whisker population is quite high. From the two-dimensional view, it appears, that, under restraint some whiskers were accommodated inside the grains, without any apparent association with the copper bearing phase. However, it is quite probable that the whiskers in question, were, in fact, across a grain boundary, but at some orientation to the polished surface of the specimen. Figs. 40.a. to 40.d., indeed indicate the latter possibility. Here, the matrix aluminium layer was preferentially etched away with dil. HCl (3%) solution to expose the whiskers, embedded deep into the matrix. If we, now, visualize a plane at some orientation to the grain boundary but sectioning
the whisker, almost parallel to its axis, it would show up on the subsequently polished surface, similar to those, as seen in Fig. 33.

The preferential location of the whiskers, at the grain boundaries, can be best discussed from the solidification characteristics of the melt in presence of foreign particles (SiC) and their mutual interactions. It is accepted that in commercial alloys, solidification starts by heterogeneous nucleation. Fundamentally, it is because of the energy requirements for homogeneous nucleation, which is higher than that required for the heterogeneous nucleation to occur. It is also known, that to initiate nucleation, the critical size of the nucleus depends entirely upon the radius of the embryo and not upon the number of atoms per embryo. Thus if the atoms, in the embryo, can be aggregated into the shape of a spherical cap on a solid substrate, as shown in Fig. 65a, this would be entirely equivalent, from the nucleation point of view, to a spherical nucleus of radius, \( r^* \). In heterogeneous nucleation, therefore, we require fewer atoms in their solid configuration to obtain a viable crystal nucleus. The conditions for heterogeneous nucleation, as depicted in Fig. 65, hold good if the periphery of the spherical cap is stable.

A solid, which acts as a nucleation catalyst, does in fact stabilize the crystalline spherical cap by providing low energy sites to initiate nucleation. The potentiality of any such substrate would, therefore, depend upon the surface energy relationships between the liquid, solid and the substrate. A measure of this stabilising power of a solid substrate on the crystal nucleus is indicated by the magnitude of the contact angle, \( \theta \), (Fig. 65b). Small contact angles are, in general, a characteristic of good heterogeneous nucleation catalysts, and, if equilibrium conditions are assumed to prevail between the interfacial
Fig. 65. Schematic model for heterogeneous nucleation. (a) spherical cap of the solid on a catalyst substrate (b) section through spherical cap (c) energy balance at the solid-liquid-substrate junction.
energies at the edge of the spherical cap, the balance of forces can be represented by the vector diagram as shown in Fig. 65.c. This yields the popular relationship,

\[ \gamma_{LC} = \gamma_{SC} + \gamma_{SL} \cos \theta \]

Although, the wetting angle, \( \theta \), is a very important parameter in the analysis of heterogeneous nucleation, very little is known about its magnitude in most real systems. This is simply because the contact angles are very difficult to measure experimentally. From the available information, it appears that \( \gamma_{LC} \) (SiC) at 1200 °C is \( \approx 840 \times 10^{-3} \) Jm\(^{-2}\) (9) and \( \gamma_{SL} \) (Al) at 800 °C is \( \approx 865 \times 10^{-3} \) Nm\(^{-1}\) (40). From these values, it is expected that, given sufficient time, molten aluminium will wet SiC substrate and, therefore, the whiskers might act as potential nucleating agents. However, it should be noted at this stage, that in general and particularly while considering a liquid to solid transformation (in real systems) the interfacial energies can reasonably be assumed to be isotropic.

An alternative approach (41) to analysing heterogeneous nucleation behaviour and specially relevant for systems having low values of \( \theta \) is to consider the possible variations in the lattice misfit across the interface between the nucleating catalyst and the crystal nucleus. Lattice misfit or registry, \( \delta \), can be expressed as,

\[ \delta = \frac{a_c - a_n}{a_n} \]

where, '\( a_c \)' and '\( a_n \)' are lattice parameters for the unstrained catalyst and the nucleus respectively. Small registry, \( \delta \ll 0.05 \) are found to be accommodated by elastic strain in the nucleus. Such a boundary would, obviously, have perfect atomic matching across the interface and will be coherent. For registries, higher than that which can be accommodated by lattice strain but less
than that at which matching is impossible, i.e., \(0.05 \leq \delta \leq 0.25\), the boundary comprises regions of good fit interspersed by a network of dislocations. Also, it has been shown (41) that the free energy barrier to heterogeneous nucleation is related to the disregistry between the lattices and a relationship has been derived between the critical undercooling and disregistry of the form, \(\Delta T \sim \delta^2\), for small \(\delta\).

The second approach to heterogeneous nucleation appears to be more realistic than the spherical cap approach, and there is also certain experimental evidence (although not fully conclusive) in support of it. Following this, in the experimental system, there is a possibility of interface formation, of semicoherent nature. This would mean that the SiC whiskers, in the molten metal bath, are in fact potential nucleation catalysts.

However, it should be borne in mind that no comprehensive theory on heterogeneous nucleation has been advanced which can predict all the nucleation phenomena. Because of the lack of comprehensive theory on this subject, the development of effective nucleation catalysts for use with industrial alloys require long and expensive programmes of work to determine the efficiency of each possible inoculant, on a trial and error basis.

From the foregoing discussion it appears, therefore, that silicon carbide whiskers, in the molten aluminium alloy bath, could initiate solidification by heterogeneous nucleation. However, once nucleation has started in the melt, one has to take into account the interaction of these whiskers with the liquid-solid interface which would, eventually, determine their locations in the as-cast structure. This is because, the nucleation condition applies for equilibrium and, therefore, depends upon the orientation of the nucleating particle (the nucleated crystal would, presumably, have specific orientation relationship with the particles having
a low surface energy). Once nucleation has started, in real castings, solidification proceeds in a non-equilibrium and in a dynamic manner. In effect, as a stationary particle (whisker) is approached by the interface, contact would be made in random orientations of particle to solid. This, in general, may give rise to an interface having high interfacial energy. Such particles may then be pushed by the interface between the cell boundaries and finally trapped within these regions. If, however, contact is made at some particular orientation of particle to solid so that the minimum surface energy condition can be fulfilled, the solid may continue growing around these particles. In such cases, the whiskers would be found trapped within the primary grains.

Uhlman et al., (42) have studied the interactions of foreign particles with solid-liquid interface during the crystallization of certain organic liquids. By analogy to their findings, it is apparent that for a constant particle size, there is a critical velocity of the interface movement below which the particles are rejected by the interface and above which they would be trapped in the solid. As a result, the more rapid the solidification, the more will be the chance for the whiskers to be distributed uniformly in the cast structure. On the other hand, slower the rate of solidification and smaller the size of the whiskers, it is likely that most of the whiskers will be pushed into the remaining liquid and would be found segregated in the regions, where the metal solidifies last.

6.2. Bonding

Various theories have been reported, in the literature, in regard to the principles of fibre reinforcement and the prediction of composite properties. Almost all these analyses assume equal strain in the fibre and the matrix, under the
applied stress. The load is transmitted to the fibres by shear along the fibre-matrix interface. Therefore, a strong bond, preferably of chemical nature, is necessary from the standpoint of maintaining the integrity and stability of the interface under the work environmental conditions. If only a mechanical bond is in existence at the interface, premature failure of the composite is possible, particularly during use at elevated temperatures. The result of interdiffusion at the boundary is the formation of a chemical bond through alloying. On the other hand, excessive diffusion may have some adverse effects on the composites, due to the homogenization and dilution phenomenon which may possibly eliminate the benefits of reinforcement in a heterogeneous structure.

Metallographic studies were undertaken to evaluate the microstructural features of the composites, related to the integrity and stability of the whisker-matrix bond. Figs. 25 to 37 are the photomicrographs of the specimens, taken from the typical SiC - Al alloy composite. The whisker-matrix bond appears to be sound. There is no indication of gross diffusion which could result in the formation of any interface layer.

The nature of the interface was further evaluated by electron microscopy. Metallographic (TEM) examinations of the composite (thin foil specimens) showed that a voidless metallurgical bond could be achieved. The smooth interface (Fig. 44) indicated the absence of any surface reaction, at least to the extent to give any reaction product, that could be identified during the study.

X-ray powder diffraction studies further supported this view that during the composite fabrication, no chemical interaction of any kind did occur to give any reaction product that could be identified under the experimental conditions.
The extent of diffusion bonding (if any) were then studied by EPM. In the application to diffusion studies, the electron probe is moved across the diffusion layer (interface) and the spectrometers are set to detect the characteristic radiations of the elements of interest. The results obtained by point by point analysis are then recorded and analysed.

Initially, qualitative examinations of the structures were carried out to find out the distribution of different elements over the entire area of interest. Fig. 41 shows the electron image and the corresponding Si Kα - x-ray image obtained from one such region. However, investigations with the EPM did not come out to be directly rewarding. It was not possible to identify any silicon carbide whisker, although they were visible in the optical image. Point analysis and line scanning were tried but without any success. The level of silicon concentration (counts) was never found to be more than that of the background. In light of these unsuccessful efforts, it was concluded, that the photograph in Fig. 41.b. for silicon distribution was not a representative one. The concentration of silicon, observed in that x-ray image, was presumably due to the segregation of whiskers in the cast structure. Incidentally, this sample was from one of the castings, made during the early developing stages of the casting process. However, this apparent inability to identify the SiC whiskers in the cast structure, during the EPM studies, indirectly established the success of the fabrication process to achieve proper individualization of the whiskers, prior to their incorporation in the matrix.

It is to be noted that the physical properties of the whiskers and the matrix can often constitute a problem in producing a uniformly distributed and bonded composite structure. Factors such as, surface tension, wettability, density difference, matrix viscosity, impede the formation of uniform dispersions. Electrostatic surface attractions of small
whiskers often cause a clustering of whiskers into 'balls' which could result in 'island-like' clusters in the final structure as evidenced in Figs. 41. a & b.

It was reported (33) that Rosenberg and Ryan also experienced identical difficulties during their EPM work, undertaken to carry out the diffusion studies in SiC-Co-base alloy composites. During this work, whiskers of diameters 0.5 - 3 µm, were examined with an electron probe of diameter 1 µm. During the present programme of study, the available probe had a diameter of ~1.5 µm. The whiskers were of diameters ranging from 0.1 µm to 1 µm. It appears, therefore, that unlike the common belief, probe diameter is not, in anyway, a direct measure of the resolution that can be achieved.

Let us briefly consider the analytical resolution, attained in practice, which appears considerably greater than the beam diameter 'd_p' on the specimen surface. The interaction of an electron beam with a solid can be schematically presented as shown in Fig. 66. The distribution in depth of the generated x-ray radiation is dependent upon the manner in which the impinging electrons are scattered and absorbed throughout this depth. The impinging electrons undergo elastic (change of direction with negligible energy loss) and inelastic (change of direction with energy loss) interactions with the atomic nucleus and the bound atomic electrons.

For the purpose of microanalysis work, and in the energy range of concern, the elastic scattering by the atomic nucleus and the inelastic interactions with the bound electrons are the two major possibilities to consider. According to Beaman and Isasi (43), the scattering cross section at constant energy varies with 'Z^2', where 'Z' is the atomic number. Also, the probability of scattering varies as Z^2/E^2, where 'E' is the excitation energy.
Fig. 66. Schematic representation of the interaction of an electron beam with a solid of moderate to low atomic number.

Emission: Secondary electrons, Backscattered electrons(1), Characteristic x-rays(2), Bremstralung radiation(3), Secondary fluorescence(4), Back reflected x-rays(5)
It is thus found that the diameter of the excited volume is primarily a function of energy and its shape is a function of atomic number. Qualitatively, for low atomic number materials, there is very little scattering near the surface. As the electron penetrates and loses energy, the scattering increases until the time it penetrates to such an extent that it can no longer escape, i.e., complete diffusion is established. Thus, there is little back scattering; the negligible amount occurs as a result of early single scattering events. As the incident energy increases, the shape remains unchanged, but the penetration and, hence, the diameter increases.

Castaing (I44) has shown that this spread 'R' can be estimated as,

\[ R = 0.033 \left( E_0^{1.7} - E_c^{1.7} \right) \frac{A}{\rho Z} \text{ micron} \quad \ldots \ldots \ldots \ldots (19) \]

where, \( E_0 \) is the probe voltage, \( E_c \) is the excitation potential of the line examined, \( A \) is the atomic weight, \( Z \) is the atomic number and \( \rho \) is the density. The effective x-ray resolution is then nearly equal to the probe diameter plus the spread 'R'.

From the above, it becomes evident that with a probe diameter of \( \sim 1.5 \mu \text{m} \) and working potential of 25 k.e.v for the Si Kα-line, the maximum possible resolution is of the order of 8.25 \( \mu \text{m} \), (details in Appendix. IV). This explains why no success could be achieved either by Rosenberg or during the present investigation. For the same reason, EDS work in conjunction with the SEM did not prove to be any better. Although, the electron beam diameter in SEM is 80 - 100 \( \AA \), with the normal working potential of 30 k.e.v, the sensitivity is inadequate to resolve the whiskers of diameter 0.1 - 1 \( \mu \text{m} \).

Efforts were made to improve the sensitivity of EPM by reducing the excitation voltage, to the extent of 5 k.e.v.
and thereby attaining a probe diameter of \( \sim 0.33 \mu m \). But under these working conditions, it was found to be extremely difficult to maintain the submicron probe diameter because of the sensitivity of the beam to astigmatism and electronic noise (hum) from stray electrical fields.

At this stage, this work was abandoned as it was realized (45) that, not only considerable skill in electronic optics would be required to modify an instrument for this purpose but the chances of success were also very remote.

It is believed, however, that the diffusion studies could be done with an EMMA (electron microscope and microprobe analyser), using thin foil specimens prepared by technique such as 'ion-thinning'. Foils, during this investigation, were prepared by a two-stage electropolishing method.

Although, the nature of the interfacial bond could not be established, unambiguously, there is little doubt about the quality of the same. Studies of the composite fractured surfaces showed excellent bond, existing between the whisker and the matrix. Both whisker pull-outs and fracturing of the whiskers were noted. Careful examination of the pulled out whiskers showed that they were, in most cases, evenly coated with the matrix material indicating good wetting and bonding between the two phases.

Figs. 57 & 59, show further examples of matrix coatings on the pulled out whiskers. The higher magnifications show that the matrix layer often exhibits markings parallel to the direction of pull-outs. It would appear from these observations that the mode of failure was due to matrix shear, slightly away from the whisker-matrix interface, rather than the decohesion of the actual interface.
6.3. Tensile Strength

The experimental results, obtained on composites of silicon carbide whiskers embedded in the aluminium alloy matrix, clearly indicated their superior tensile strengths over the blank control. The strength of the composites has been found to increase with the volume fraction of the whiskers added. Further improvements in strength could be observed when the composites were solidified under pressure. The improvement in strength did not have any linear relationship with the increase in whisker volume fraction and/or the solidification pressure.

The tensile behaviour of the composites can be typified by the load-extension curve as shown in Fig. 67. As anticipated, with the whiskers of mixed sizes, the load-extension curve has been found to have a decreasing slope with increase in extension. This nature of these plots can be explained as follows.

Composites, of the type considered during this programme of study, contained a spectrum of whiskers of different lengths, and in three dimensional random orientations. In cases like this, i.e., of misaligned fibres, the contribution of the fibres towards the strengthening as predicted by equation (1) is to be reduced accordingly (29). For fibres in a random two dimensional array, the overall contribution is to be reduced to a third and if they are randomly dispersed in three dimensions, this factor is one-sixth. Thus the equation (1) can be rewritten as,

\[ \sigma_c = K \cdot \sigma_f \cdot V_f + E_m \cdot \epsilon' (1 - V_f) \] ........................(20)

where, \( K \) is the orientation factor having a value 1 to 0.167 and \( \epsilon' \) is the ultimate strain of the composite.
Fig. 67. Hounsfield Tensometer plot for Tensile Strength vs. Extension.

Specimen No. 2; Composite containing 0.5 vol. per cent. of whiskers and solidified under 137.8 MNm$^{-2}$ pressure.
For a fibre of length greater than the critical length, we find from equation (12),

\[
\bar{\sigma} = \sigma_f \left( 1 - \frac{l_c}{2l} \right) = \frac{E_f \varepsilon' (1 - \frac{\sigma_f' r_0}{2 \gamma})}{2} = \frac{E_f \varepsilon' (1 - \frac{E_f \varepsilon' r_0}{2 \gamma})}{2} \quad \text{.................(21)}
\]

In cases, where, the fibre length is less than the critical, i.e., \( l < l_c \), from equation (12) we get,

\[
\bar{\sigma} = \frac{\sigma_f}{2} = \frac{2 \gamma l}{2 r_0} \frac{l}{2} = \frac{1 \gamma}{2 r_0} \quad \text{.................(22)}
\]

The equation (22) gives the average fibre stress in case of the subcritical sized fibres.

Equations (21) and (22) imply that the absolute fibre length is related to the average stress carried by the fibres, and at any value of the composite strain \( \varepsilon' \), there is a critical fibre length \( l_e \). Fibres, shorter than this, will carry an average stress as given by the equation (22) which is always less than \( (1/2) E_f \varepsilon' \). Fibres, longer than this, would carry an average stress as indicated in equation (21) which would always be greater than \( (1/2) E_f \varepsilon' \). \( l_e \) will then be given by,

\[
l_e = \frac{E_f \varepsilon' r_0}{\gamma} \quad \text{.................(23)}
\]

In case of composites, reinforced with mixed sized fibres/whiskers, there will be broadly two groups of fibres — subcritical and supercritical. At low strains, all fibres...
will make a contribution to the reinforcement as given by the equation (21), since \( l_0 \) will be small. As the strain is increased, a progressively smaller proportion of the fibres will reinforce according to equation (21) and an increasing portion would follow equation (22). We may, therefore, expect the slope of the load-extension graph, for such a composite material, to decrease as the extension (strain) is increased.

The overall situation can be summarized by combining the concepts of the equations, (20), (21), (22) and (23), and can be shown as,

\[
\sigma_c = F \cdot X + F \cdot Y + M \tag{24}
\]

where, 'F.X' is the contribution of the subcritical whiskers, 'F.Y' is that of the supercritical whiskers and 'M' is the contribution of the matrix. They can be expanded as,

\[
X = \sum_{1 \leq l \leq l_1} \frac{\gamma \cdot l_1 \cdot V_1}{2 \cdot r_0} \tag{25}
\]

\[
Y = \sum_{l_2 \geq l_1} E_f \cdot e' \left( 1 - \frac{E_f \cdot e' \cdot r}{2 \cdot 12 \cdot \gamma} \right) V_2 \tag{26}
\]

\[
M = E_m \cdot e' \left( 1 - V_f \right) \tag{27}
\]

where, \( l_1 \) and \( l_2 \) are the subcritical and supercritical whiskers when \( V_1 \) and \( V_2 \) are their respective volume fractions.

Therefore,

\[
\sigma_c = F \left[ \sum_{1 \leq l \leq l_1} \frac{\gamma \cdot l_1 \cdot V_1}{2 \cdot r_0} + \sum_{l_2 \geq l_1} E_f \cdot e' \left( 1 - \frac{E_f \cdot e' \cdot r}{2 \cdot 12 \cdot \gamma} \right) V_2 \right] + E_m \cdot e' \left( 1 - V_f \right) \tag{26}
\]
In practical systems (46), the terms \( E_f \), \( E_m \) and \( r_o \) can be readily obtained and relationship between \( \sigma_c \) and \( e' \) may be obtained from the tensile test. \( F \) and \( \gamma \) are, however, generally not known but the fibre length distribution can be found out. Now, assuming the orientation factor, \( F \), independent of strain and is of the same value for all the fibres, this relationship allows both \( F \) and \( \gamma \) to be determined from the tensile test data.

6.4. Concluding Remarks

From the foregoing discussion, it is evident that the addition of varying amounts of silicon carbide whiskers improved the tensile strength of the experimental aluminium alloy matrix, which was directly related to the amount of whiskers added; although, not directly proportional to the volume fraction.

Although, this investigation was not directed, in any way, to verify the validity of the well accepted "rule of mixtures" (ROM), it is quite conspicuous that the ROM can not be directly applied to predict the tensile strength of the composites, when reinforced with mixed sized whiskers and in three dimensional random orientations. However, for the purpose of this discussion, the ROM was applied, with some reservations (details in the Appendix. V.) to predict the strength of the experimental composites. These results, compared with the experimental results, are given in Fig. 68.

It has, already, been shown that a volume fraction of 0.019 of whiskers, having a critical aspect ratio of 21.7, should be exceeded to obtain any improvement in strength of the experimental matrix. Therefore, accepting the validity of the equation (5), strengthening should not have been
Fig. 68. Experimental strength of the composites when compared to the strength predicted from ROM.
observed even if all the whiskers were of aspect ratios equal to or greater than the critical, 21.7. The aspect ratios of the whiskers, used during this investigation, varied from 5 to 500.

By analogy to the different probable strengthening mechanisms, suggested in the literature, the observed strengths of the composites can be explained as follows:

1) Whiskers will strengthen the matrix by a process as observed in the dispersion hardened metals.

2) Some of the whiskers would contribute to strengthening according to the classical 'rule of mixtures'.

The combined effect of these would give rise to a positive deviation from the strength that could be predicted from the rule of mixtures.

There is considerable evidence in the literature that the addition of varying amounts of fibres/whiskers to metal matrices produce improvements in tensile strength. The relevant details have been discussed before. Good interfacial bond is a necessary criterion for efficient load transfer from the matrix to the fibres. In this context, it should be noted that although the nature of the whisker-matrix bond could not be established conclusively, some mechanical bond is always expected. Silicon carbide whiskers often grow with irregular surfaces, resembling "bamboo" like outline. Fig. 69, shows this particular type of whisker surface. It is, therefore, quite probable that even if there exists no chemical bonding, due to differential solidification contraction, matrix material would grip the whiskers, very tightly. As a result, some load transfer would be possible due to the frictional forces, during the flow of the matrix material. Load transfer, by this process, would obviously be possible when the composites are tested at room temperature. At elevated temperatures, due to
Fig. 69. Whiskers showing 'bamboo' like outer surface

(a) x 5,000

(b) x 5,000
differential thermal expansion of the interface would prevent load transfer. However, this programme did not include testing at elevated temperatures.

Positive deviation of composite tensile properties, from that predicted by the ROM, have been investigated by several workers (32, 46-50) and have been attributed to either of the two sources. Some workers claim that positive deviations arise from dislocations blocking effects, while others believe that they arise from biaxial constraints caused by different Poisson contractions of the two phases.

In the first case, the dislocations in the matrix are thought to pile up against the fibres. The resulting back stresses act against dislocation sources, resulting in higher matrix flow stress. The proposed mechanism is analogous to the Petch effect strengthening of polycrystalline metals. On the other hand, the difference in the Poisson coefficients of two bonded phases results in the generation of stresses normal to the interface, when the composite is deformed parallel to the interface. This normal stress reduces the resolved shear stress in the two phases. Consequently, dislocation motion and matrix flow occur at higher applied stresses than expected.

Although, some ambiguity is there as to which of these two effects is dominant in the metal matrix composites, there is little doubt that fibres/whiskers interact with the dislocations on a slip plane in the matrix. The fibres, penetrating the slip planes are thought to affect the dislocation movement in the same manner as the dispersed particles do. Both dislocation bowing between fibres and pile ups against fibres were reported (48) to occur during the deformation of iron fibre reinforced copper composites. Electron microscopic studies indicated the preferential
association of the dislocations with the fibres. Hansen (51), reporting on the strengthening of aluminium by a three dimensional network of aluminium oxide powders, indicated that the observed strengthening was almost as much as a uniform dispersion of particles would have.

The application of pressure, during the solidification of the matrix alloy, further intensified the improvements in the composite tensile strengths. The effects of pressure, applied on liquid alloys, have been investigated (36) and reported earlier (37,38).

The alloy used during this investigation was LM 11, a commercial aluminium alloy having copper (4-5%) as the alloying element. Unlike the alloys used during the earlier investigation, LM 11 is a solid-solution type. Also, it has a wide solidification temperature range. It is known that alloys of this type suffer most from gross porosity of distributed nature. Pressure, applied during solidification, reduces or eliminates the porosity by effective feeding and/or by inhibiting pore formation, in case they are caused by soluble gases. The overall effect would obviously be reflected in the improved soundness of the castings.

Also, pressure applied on the liquid alloy would increase the melting temperature of the alloy. This coupled with the increased rate of solidification (resulted from greater degree of undercooling) would result in increased solubility of copper in aluminium at room temperature. The latter would mean an increased strength of the primary aluminium. Both these factors, i.e., improved soundness and solid solution hardening would have contributed towards the observed improvements in the strength of the control sample.
The further improvements in the strength of the composites, when solidified under pressure, would obviously due to the combined effects of superior soundness and strength of the metallic matrix, in addition to the strengthening caused by the whiskers themselves.

6.5 Conclusions

For the experimental condition selected, the conclusions that can be drawn from this investigation are as follows:

1) Whiskers could be randomly oriented and uniformly distributed in the as cast structure.

2) Addition of whiskers, in the experimental alloy matrix, yielded composites with improved tensile strengths. Although, this improvement was related to the volume fraction of the whiskers, introduced, it was not directly proportional to the whisker volume fraction.

3) From the observed strengthening, it appeared that the 'rule of mixtures' could not be directly applied to predict the strength of the composites.

4) Composites, reinforced in three dimensional random orientation, exhibited isotropic tensile strengths.

5) Although, the nature of the whisker-matrix bond could not be established, unambiguously, the bond was strong enough, so that the whiskers could be loaded upto their fracture stresses.

6) Pressure, applied during the solidification, further improved the tensile strength of the composites.

7) Under the normal working conditions, common electron optical instruments, eg., EPM, SEM, do not have the requisite resolution, and, therefore, they can not be used for interfacial studies of composites, reinforced with submicron sized whiskers.
The present investigation was of an exploratory nature. However, reinforcement of metal matrices, in three dimensional random orientation, with mixed sized whiskers show enough potential and, therefore, detailed investigation on the different aspects of fabrication and strengthening to have a clear understanding of the various fundamental processes, involved.

With a view to commercial utilization of these metal-whisker composites, further fruitful investigations can be carried out as:

1) Investigation into the aspects of the fabrication process to establish minimum whisker damage.

2) The exact nature of whisker-matrix interfaces and the stability of the bond should be established, under conditions of thermal cycling and thermal fluctuations.

3) Metal-whisker composites, interaction and bonding, could also be evaluated after heat-treatment (for heat treatable metal matrices) and the stability and strength assessed.
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### References

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APPENDICES
DIE DESIGN AND DETAILS OF SUGGESTED HEAT TREATMENT PROCEDURE

Notes:

1. Scale- ¼ Full Size.

2. Material for parts No. 2, 3, 5 & 6 is H13 die steel of composition 0.35 % C, 5 % Cr, 1.5 % Mo and 1 % V.

3. Material for part No. 4 is En8 steel of composition 0.35-0.4 % C, 0.05-0.35 % Si, 0.6-1 % Mn, 0.06 % max. S and 0.06 % max. P.

Heat Treatment procedure to be followed:

i. H13 die steel

a. Anneal packed in a neutral material to temperature 875 to 900 °C. Cooling rate after annealing should not exceed 10 °C per hour to at least 540 °C. Annealing hardness - 192 to 225 BHN. (Stress-relief anneal at 710 to 730 °C may be employed before final finish machining).

b. Hardening temperature - 1000 to 1040 °C
Quenching medium - Air
As quenched hardness - R_c 50 to R_c 55
(Preheating at 650-675 °C and then 875 °C is recommended)

c. Tempering at 550 to 650 °C to give final hardness R_c 40 to R_c 45.

ii. En8 steel

a. Annealing at 830 to 860 °C

b. Hardening at 830 to 860 °C followed by oil or water quench.

c. Tempering at 550 to 660 °C

Table A.1.1
0.0714" hole for thermocouple

0.01" dia vents

Six 0.25" holes
Part 3.
Part 4.

Part 6.
APPENDIX II
<table>
<thead>
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<th>Specimen No.</th>
<th>U.T.S (HNm⁻²)</th>
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Table A.2.1
Composite (0.025 vol. per cent. of whisker)

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<td></td>
<td>4(3T)</td>
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<tr>
<td>0.1</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>1</td>
<td>165.98</td>
</tr>
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Table A.2.2
Composite (0.075 vol. per cent. of whisker)

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<td>7(6T)</td>
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Table A.2.2
Composite (0.25 vol. per cent. whisker)

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*Table A.2.4*
Composite (0.5 vol. per cent. whisker)

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<th>U.T.S (MNm⁻²)</th>
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<tr>
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<td>3</td>
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<td>4</td>
<td>196.86</td>
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Table A.2.5
APPENDIX III
APPENDIX IV
Appendix IV

X-ray Resolution in Electron Probe Microanalyser

In order to predict the x-ray resolution in an EPM the lateral electron diffusion must be known. Normally, this is worked out experimentally, from the measurements of electron transmission through, and energy losses in, thin film specimens. These values are then related to the penetration.

Several empirical relationships have been put forward by various workers among which the one suggested by Castaing (44)

\[ R = 0.033 \left( E_o^{1.7} - E_c^{1.7} \right) \frac{A}{\rho Z} \mu m \] ................. (i)

was considered for this calculation. Following this the x-ray resolution

\[ R_x = R + d, \] where,

\[ R \] - electron spread,
\[ E_o \] - probe voltage,
\[ E_c \] - excitation potential of the x-ray line under consideration,
\[ A \] - atomic weight of the element studied,
\[ Z \] - the atomic number,
\[ \rho \] - the density (gms/cm³), and,
\[ d \] - the probe diameter (\( \mu m \)).

While examining for silicon in silicon carbide, the following values are to be considered for estimating the resolution of Si Kα x-ray line,

\[ E_o = 25 \text{ k.e.v.}, \]
\[ E_c = 1.83 \text{ k.e.v.}, \] and for Si,
\[ A = 28, \]
\[ Z = 14, \] and, \[ \rho = 2.3 \text{ gms/cm}^3. \]
Therefore,

\[ R = 0.033 \left( 25^{1.7} - 1.83^{1.7} \right) \frac{28}{14 \times 2.3} \mu\text{m} \]

\[ = 6.7465 \mu\text{m}. \]

i.e.,

\[ R_x = (6.7465 + 1.5) \mu\text{m} \]

\[ = 8.2465 \mu\text{m}. \]

Additional Remarks:

From equation (i), it is evident that the resolution gain can only be realized when 'd' represents a significant portion of 'R', i.e., 'R' is small. This can only occur when the overvoltage \( U = \frac{E_o}{E_c} \) and, therefore, the acceleration potential are low.

From the available information, it appears that operation at normal overvoltages of 2 to 3 will provide x-ray resolution gain ranging from 0-2000 Å and permit analysis of particles down to about 0.3 μm. A nomogram, referred by Reed (43) has been represented in Fig. A.4.1., that can be used for qualitative assessment of the available x-ray resolution.
Fig. A.4.1. A nomogram for calculating the qualitative x-ray resolution. The analysis of Si ($E_c = 1.83$ kV) at 25 kV in a specimen with a density $2.3 \times 10^3$ kg/m$^3$ is illustrated. A qualitative x-ray resolution of 4.5 $\mu$m is predicted (43).
APPENDIX V
Appendix. V

Prediction of composite tensile strength from the 'Rule of Mixtures', (ROM).

The tensile strengths of the experimental composites were predicted from 'ROM' on the following basis:

1) All the whiskers were of aspect ratios, equal to or greater than that predicted from the equation (6), which was equal to 21.7 whereas the actual aspect ratio varied from 5 to 500.

2) It was also assumed that for the same volume fraction, reinforcement due to the whiskers in a random three dimensional array is equivalent to one sixth \((\frac{1}{6})\) of that, achieved with whiskers aligned unidirectionally.

Therefore, we find:

<table>
<thead>
<tr>
<th>Whisker volume per. cent.</th>
<th>Equivalent according to the assumption (2)</th>
<th>Effective vol. fraction</th>
</tr>
</thead>
<tbody>
<tr>
<td>Actual -used during the experiment</td>
<td>Equivalent</td>
<td>Effective vol. fraction</td>
</tr>
<tr>
<td>I. 0.025</td>
<td>(\approx 0.00416)</td>
<td>0.0000416</td>
</tr>
<tr>
<td>II. 0.075</td>
<td>(\approx 0.0125)</td>
<td>0.000125</td>
</tr>
<tr>
<td>III. 0.25</td>
<td>(\approx 0.0416)</td>
<td>0.000416</td>
</tr>
<tr>
<td>IV. 0.5</td>
<td>(\approx 0.0833)</td>
<td>0.000833</td>
</tr>
</tbody>
</table>

The strength of the composites were then calculated from the equation (3),
For this calculation, the values of 'S_f' and 'σ^*_m' were considered to be equal to,

\[ S_f = 2756 \text{ MNm}^{-2} \text{ (a)} \]
\[ σ^*_m = 63.25 \text{ MNm}^{-2} \text{ (b)} \]

Therefore,

\[ S_{c/1} = (2756 \times 0.0000416) + (63.25 \times 0.9999584) = 63.362 \text{ MNm}^{-2}. \]
\[ S_{c/II} = (2756 \times 0.000125) + (63.25 \times 0.999875) = 63.565 \text{ MNm}^{-2}. \]
\[ S_{c/III} = (2756 \times 0.000416) + (63.25 \times 0.999584) = 64.3702 \text{ MNm}^{-2}. \]
\[ S_{c/IV} = (2756 \times 0.000833) + (63.25 \times 0.999167) = 65.493 \text{ MNm}^{-2}. \]


(b). \[ σ^*_m \approx γ \approx 0.55 S_m, \]

where, \( S_m = 115 \text{ MNm}^{-2} \), obtained during this experimental programme.