The characterization of short fibres for the production of metal matrix composites

June 1988

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List of symbols

c composite
d fibre diameter (m)
\(d_{hkl}\) interplanar spacing indexed by Miller indices hkl
E Young's modulus (GPa)
f fibres or short fibres
l longitudinal, length of fibres (m)
\(l_c\) critical length (m)
m matrix
\(\text{mmc}\) metal matrix composite
t transverse
\(\tau\) shear stress
TEM transmission electron microscopy
UTS ultimate tensile strength
\(v_f\) volume fraction
\(v_c\) see text pp 8
\(v_{\text{min}}\) see text pp 8
wt% weight percent
\(\varepsilon\) strain
\(\sigma\) tensile stress (GPa)
\(\lambda\) wavelength X-rays (Å"
\(\theta\) Bragg angle (°)
1. INTRODUCTION

Recent years have shown an increasing interest in metal matrix composites (MMC) arising at least in part from the current availability of several fibres with attractive properties. The majority of effort has been centred on the reinforcement of aluminium and its alloys for use in special aerospace applications. The high performance potential of MMC systems (in terms of, for example, specific stiffness, tensile strength and wear resistance over a high range of temperature) is well documented, but economic factors and particularly difficulties in developing viable fabrication routes have limited commercial exploitation.

The purpose of the Euram contract, MAIE-0065-c(A) is to develop short fibre reinforced aluminium metal matrix composites for application in aerospace primary components using powder metallurgy techniques. The main objectives for materials used in aerospace components are a high strength and stiffness with the highest possible weight saving in combination with improved durability and damage tolerance.

Referring to the meeting at Augusta in Milano, February 18, 1988, there was confusion about the required properties of the reinforcement. Therefore, this report starts in chapter 2 with an introduction about the basic concepts of the strength and stiffness of metal matrix composites. The models described may be informative with respect to strength, stiffness and morphology of the fibres to be used.

In chapter 3 an experimental investigation is described of the three alumina silicate manufactured by Carborundum. These fibres are compared with two other fibres: 6-Al2O3 (results from the literature) and SiC (results from literature and own investigation). The report is completed (chapter 4) with conclusions and recommendations about the required properties of the fibres for the purpose of metal matrix fabrication at Alumina in Italy.
2. THE STRENGTH AND STIFFNESS OF METAL MATRIX COMPOSITES

2.1. Introduction

Fibre reinforced metal matrix composites (mmc's) offer many advantages in aerospace applications where low density, high strength and high stiffness are of prime concern. The reinforced composites can be generally classified into two types: (i) reinforcement by long continuous or unbroken fibres of a high strength material, (ii) reinforcement by discontinuous fibres, or whiskers, with a finite length/diameter ratio. Discontinuous fibre reinforced mmc's offer an additional advantage because they can be shaped by standard mechanical processes such as forging, rolling, extrusion, etc.

The strength and stiffness of metal matrix composites will be briefly reviewed by means of models from the literature. In chapter 2.2. the mechanical properties of continuous fibre reinforced mmc's will be discussed by means of the rule of mixtures. For the whisker reinforced materials some modifications of the rule of mixtures are necessary as described in chapter 2.3. Matrix and particle effects on the strength of composites will be considered in chapter 2.4. The described models have some practical value in predicting the composite strength and stiffness. Recommendations for required properties are given in chapter 2.5.

Unfortunately no models of the surprisingly low ductility values of the reinforced composites can be described in this report, because a quantitave treatment is not presented in the literature.

2.2. The rule of mixtures

The rule of mixtures is used here as a first approach for the calculation of both stiffness and strength of a unidirectional composite loaded in the fibre direction. The assumption made in this rule is that the strain is the same in the fibres and in the matrix.

2.2.1. The stiffness in the fibre direction

If both matrix and fibres deform elastically Young's modulus in the longitudinal direction, $E_{CL}$, can be calculated using eq 2.1. (ref.8):
\[ E_{CL} = E_f v_f + (1 - v_f) E_m \]  \hspace{1cm} (2.1.)

where \( E_{CL} \), \( E_f \), and \( E_m \) are the Young’s moduli of the composite, the fibres and the matrix respectively, and \( v_f \) is the volume fraction of the fibres.

If the load is further increased until the yield strength of the matrix is exceeded, see fig. 2.1, Young’s modulus of the composite can be expressed by eq 2.2:

\[ E_{CL} = E_f v_f + (1 - v_f) \frac{d\sigma_m}{d\epsilon_m} \]  \hspace{1cm} (2.2.)

where \( \frac{d\sigma_m}{d\epsilon_m} \) is the slope of the stress-strain curve of the matrix at a strain \( \epsilon \) of the composite.

Fig. 2.1. Stress-strain curves for fibre, composite and matrix, illustrating the Young's modulus of the composite at a strain \( \epsilon \) where the matrix is loaded in the plastic region.
Generally in metal matrix composites the second term in the right-hand side of eq 2.2 can be neglected and the stiffness of the composite will linearly increase with both the stiffness of the fibres and the volume fraction of the fibres.

2.2.2. Tensile strength in the fibre direction

Unidirectional fibre-reinforced composite deforms as the load increases in the following four stages, depending on the relative brittleness or ductility of the fibres and the matrix (ref. 8, 13), see fig. 2.2.

Stage I  Both fibres and matrix deform elastically (eq 2.1)
Stage II The fibres continue to deform elastically but plastic deformation occurs in the matrix (eq 2.2)
Stage III Both fibres and matrix deform plastically
Stage IV The fibres fracture followed by fracture of the composite

Of course, for brittle fibres, like ceramic fibres used for mmc's stage III will not occur. Generally fracture of the metal matrix composite occurs as a fibre failure. In the models considered all fibres are assumed to have equal strength and thus will fracture together at the same longitudinal position.

For the calculation of the ultimate tensile strength two different cases have to be considered, see fig. 2.3 (ref. 8). In both cases the failure strain of the fibres is assumed to be lower than the failure strain of the matrix. As a consequence, fibre failure will always occur before failure of the matrix. However, if the fibre volume content is low it is possible that the fibre loads are fully transmitted to the matrix without inducing an immediate matrix failure, see fig. 2.3A.
Fig. 2.2. Stress-strain curve of a metal matrix composite. In the curve four different deforming stages are indicated.

Fig. 2.3. Stress-strain curve of two different composites with different matrix properties (ref. 8)
In the latter case the failure stress of the composite is:

\[
\sigma_{uc} = (1 - v_f) \sigma_{um}
\]  \hfill (2.3.)

where \(\sigma_{uc}\) and \(\sigma_{um}\) is the ultimate tensile strength of the composite and the matrix.

If the fibre volume content is large fibre failure will immediately lead to complete failure, see fig. 2.3B. The composite failure stress then is:

\[
\sigma_{uc} = \sigma_{uf} v_f + (1 - v_f) \sigma_m^*
\]  \hfill (2.4.)

where \(\sigma_{uf}\) is the ultimate tensile strength of the fibres and \(\sigma_m^*\) is the matrix stress at a matrix strain equal to the maximum tensile strain in the fibres.

---

Fig. 2.4. Composite strength vs. fibre volume fraction (ref.13)

AB: eq 2.4.
CD: eq 2.3.
Eq. 2.3 and 2.4 are more easily understood when they are plotted in a rule of mixture diagram see fig. 2.4.

There the composite strength, $\sigma_{uc}$, is plotted as a function of the fibre volume content, $v_f$. This diagram shows two important volume fraction parameters. (i) $v_{\text{min}}$, the critical volume fraction at which the failure mode of the composite changes from a multiple fibre-fracture mode to instantaneous failure of the composite following fibre fracture, (ii) $v_{\text{crit}}$, the volume fraction which must be exceeded to produce reinforcement of strength.

Note that for a given Al-alloy only the slope of the line AB in figure 2.4 can be changed. A higher strength of the fibres gives (i) a lower $v_{\text{min}}$ and $v_{\text{crit}}$ and in addition it enables (ii) a higher strength of the composite.

2.3. Modifications on the rule of mixtures

In the case of discontinuous randomly oriented short-fibre arrays some modifications are required to account for the lack of reinforcing efficiency due to (i) the discontinuous nature of the fibres and (ii) the three dimensionally random orientation of the reinforcement.

2.3.1. The stiffness of short fibre reinforced composites

Mc. Danels et al (ref. 6) showed that the reinforcement content is the dominant factor in improving the modulus of elasticity of mmc's. Their results from tests on Young's modulus of a 6061 Al-matrix reinforced with SiC showed, that the stiffness of the composites is increased with increasing reinforcement content. However, this increase was not a linear function of the fibre content, as in the case of continuous fibre composites with fibres aligned in the loading direction (eq 2.2)

The longitudinal and transverse Young's moduli, $E_L$ and $E_T$ respectively, for unidirectional composites reinforced with short fibres are obtained with the Tsai-Halpin model (ref. 5,9):

$$E_L = \frac{E_m (1 + ab v_f)}{(1 - b v_f)}$$  \hspace{1cm} (2.6.)

$$E_T = \frac{E_m (1 + 2b v_f)}{(1 - b v_f)}$$  \hspace{1cm} (2.7.)
where

\[ a = 2 \left( \frac{1}{d} \right)_f, \]

\[ b = \left( \frac{E_f}{E_m} - 1 \right) / \left( \frac{E_f}{E_m} + a \right) \]

and

\[ \left( \frac{1}{d} \right)_f \] is the fibre length diameter ratio

Skibo (ref. 16) showed that Young's modulus of a randomly oriented composite \( E \) is given by

\[ E = \frac{3}{8} E_L + \frac{5}{8} E_t \] (2.8.)

Computed modulus values are in reasonable agreement with the experimental values of Al 2024 reinforced with SiC, see fig. 2.5 (ref. 10). Eq 2.8 and fig. 2.5 show that Young's modulus for a given Al-alloy will increase with (i) increasing fibre content, (ii) increasing length diameter ratio of the fibres and (iii) increasing stiffness of the fibres.

The mechanical properties of some ceramic materials used for reinforcement of metal matrices are given in table 1, pp 16. This table shows that SiC and \( \delta-Al_2O_3 \) are promising candidates for improving the stiffness of Al-alloys, because of their high Young's modulus.
Fig. 2.5. Experimental modulus values for 2024-SiC (T6 heat treatment); solid lines are calculated using eq 2.8 (ref. 10)

2.3.2. The strength of short fibre reinforced composites

The strength of short-fibre reinforced composites is always smaller than the strength of continuous fibre reinforced composites because the average tensile load in the short fibres is lower than in the case of the continuous fibres. Moreover other failure modes are possible for example fibre/matrix debonding (ref. 5).

The ultimate tensile strength of the composites can be calculated by a modification of the rule of mixtures to account for the finite length of the fibres required to transfer stress from the matrix to the fibres.

An extra term relating to the length of the fibres \( l \) to the critical length \( l_c \) must be introduced in eq 2.4 as pointed out by Friend (ref. 2)

The strength of a unidirectional short-fibre composite, for \( l > l_c \) is given by

\[
\sigma_{uc} = \sigma_{uf}v_f(1 - \frac{l}{2l}) + \sigma_m (1 - v_f)
\]  

(2.9.)
Values for $l_c$ can be calculated from the equilibrium between the tensile load in the fibres, $\sigma_{uf}$, and the shear stress, $\tau$, in the matrix material, using eq (2.10) (ref. 2)

$$l_c = \frac{\sigma_{ufd}}{2\tau}$$

(2.10.)

An approximation for the shear load can be derived from the shear yield strength of the matrix alloys, which are assumed to be half the tensile strength.

To account for the non-axial distribution of the fibres an additional factor, $C$, must be introduced in eq (2.9) to further reduce the fibre reinforcing efficiency, thus

$$\sigma_{uc} = C\sigma_{uf}v_f(1 - \frac{l_c}{2l}) + \sigma_m^* (1 - v_f)$$

(2.11.)

A number of values have been suggested for $C$. For a completely random fibre array Friend (ref. 2) suggested $C = 1/5$.

Eq 2.11 can adequately predict the experimental room temperature strength of aluminium alloys reinforced with $\delta$-Al$_2$O$_3$ fibres.

The composites were prepared by a pressure infiltration technique. These investigated composites show improvements in modulus, however little or no improvements in strength were observed. Eq 2.11 is plotted in fig. 2.6. This figure explains that no reinforcement occurs when the ultimate strength of the fibres result in a high value for the critical volume fraction ($v_{crit}$). $v_{crit}$ must be exceeded to produce an increase in strength of the Al-alloy.

For improving matrix properties a low $v_{crit}$ is necessary, this can be achieved according to eq 2.11 by (i) a high length of the fibres and (ii) a high ultimate strength of the fibres.

Table 1 shows the excellent strength of SiC-whiskers, so SiC shows to be a promising candidate for improving the strength of Al-alloys.
Fig. 2.6. Rule of mixture prediction, using eq 2.11 for $\delta$-$Al_2O_3$ reinforced Al-4wt% Zn-2wt% Mg composites: (s) experimental data (ref. 2)

Arsenault (ref. 3) analysed the strength of composites reinforced by SiC-whiskers with a broad statistical length distribution. These composites were manufactured by powder metallurgy followed by hot extrusion. During the extrusion process whisker breakage occurs and a statistical approach of the rule of mixtures is necessary. The strength of these composites can be calculated by means of eq 2.12 and a histogram of the percentage of volume fraction of fibres. By using the statistical analysis, Arsenault (ref. 3) obtains

$$\sigma_{uc} = \sum \sigma \left( \frac{l_i}{d_i} \right) v_i + \sum \sigma_{uf} \left[ \frac{1 - \sigma_{uf}}{4l_i\sigma^*} \right] v_i + \sigma^* \left[ 1 - V_f \right] \quad (2.12.)$$

where $v_i$ is the volume fraction of the fibres with a length $l_i$, and $l_c$ can be estimated by eq (2.10).
The theoretical strength of a 6061 Al matrix reinforced with 20 vol.% SiC, using eq (2.12) is 470 MPa, which is lower than the observed value of 640 MPa. The observed strength of this composite is much higher than the strength of the unreinforced 6061-Al alloy, which has an UTS of 310 MPa (T₆-heat treatment).

2.4. Other effects influencing mechanical properties

2.4.1. Interaction reinforcement on matrix properties

There is sufficient evidence from the literature (ref. 3, 4, 6, 14) that the presence of ceramic fibres in a metal matrix composite influences the matrix properties. Due to the relatively large difference between the thermal expansion coefficients of ceramic materials and metals (for SiC and Al the ratio is 1 to 10), substantial stresses result from contraction during cooling from the annealing or processing temperature. These stresses result in plastic deformation of the matrix material and the generation of dislocations near the interface.

Arsenault et al (ref. 4) observed by means of TEM in a 6061 Al-alloy reinforced with 20 vol.% SiC a high density of dislocations surrounding the SiC. These dislocations tend to cluster and form low angle cell boundaries. Liaw et al (ref. 14) also observed subgrains of approximately 0.5 - 2 μm in a 2124 Al-alloy reinforced with SiC. In these subgrains numerous dislocations were observed. The size of the subgrains was found to be dependent upon that of the SiC whiskers, that is smaller subgrains accompany smaller SiC whiskers.

These dislocations and low-angle grain boundaries can serve as heterogeneous nucleation sites for strengthening precipitates during the aging of Al-alloys composites and facilitate the attainment of peak matrix hardness at much shorter times. Christman et al (ref. 7) observed by means of matrix microhardness measurements and by means of TEM in a 2124 Al-alloy with 13 vol.% SiC an accelerated preferential growth of precipitates (Al₂CuMg) at dislocation sites.

It can be suggested that besides the SiC whiskers, the strength level of the composite is also derived from the presence of dislocations and subgrains. Arsenault (ref. 3) proposes the following expression for dislocation strengthening in terms of the ultimate tensile strength of metal matrix composites, assuming that each term contributes independently to the strength:
\[ \sigma_{uc} = \sigma_{\text{comp}} + \sigma_{\text{disl}} + \sigma_{\text{sg}} + \sigma_{\text{ah}} \]  \hspace{1cm} (2.13.)

where

\[ \sigma_{\text{comp}} \] is the strengthening due to the reinforcement as predicted by eq (2.12).

\[ \sigma_{\text{disl}} \] is the strengthening due to dislocation generation,

\[ \sigma_{\text{disl}} + \sqrt{\rho} \text{, where } \rho \text{ is the average dislocation density.} \]

\[ \sigma_{\text{sg}} \] is the subgrain component of strengthening \( \sigma_{\text{sg}} + (d)^{-1/2} \), where \( d \) is the average size of the subgrains and \( \sigma_{\text{ah}} \) is the increase in strength due to the heat treatment.

By calculating each term of eq (2.13), Arsenault (ref. 3) predicted a value of \( \sigma_{uc} \) which was in agreement with the experimental value.

\[ \text{2.4.2. Whisker and particle effects} \]

Nowadays little detailed studies are available of mechanical properties of MMC's as a function of the quality of the short fibres, for example purity, grown faults, microtwins and the morphology and the distribution of the fibres in the matrix. Also discrepancies are found between tensile properties of whisker and particle SiC-reinforced Al-alloys (ref. 5).

Mc. Daniels et al (ref. 6) investigated discontinuous Al/SiC composites and they found that the stiffness and the strength of the composites were independent of the type of reinforcement. The strength of the composites were controlled primarily by the type and temper of the matrix alloy and by the reinforcement content.

The moduli of these composites were controlled by the SiC content. Other investigators as mentioned by Nair et al (ref. 5) found somewhat higher strength and stiffness for the short fibre (SiC) composites than for the particle (SiC) case.

The influence of inclusions on mechanical properties was investigated by Ordway et al (ref. 14). They showed that the debris content of SiC products has a substantially adverse effect on the composite properties.
This debris can consist of non-whisker or particle forms of SiC in addition to other extraneous matter such as other ceramic inclusions. Following elaborate beneficial procedures to remove debris, they found an approximate doubling in both the strength and the ductility of their SiC-reinforced 2024 alloy. The debris of SiC can also act as nucleation sites for cracks as mentioned by Yau (ref. 19) who observed debris or fracture surfaces of 6061-20 vol.% SiC. From these studies one can draw the conclusion that the debris content should be as low as possible.

2.5 Discussion

According to the modified rule of mixtures, discussed in 2.3, the stiffness and strength of Al-alloys are improved by using short fibres with (i) a high strength and stiffness (ii) and by a high length/diameter ratio. Because of its high strength and stiffness, the rule of mixtures predicts that SiC is a promising candidate.

Experimental results from the literature (ref. 3, 4, 6, 10, 18) show that both stiffness and strength of Al-alloys are improved by SiC.

Reinforcing Al-alloys with Saffil-short fibres gives an improvement in stiffness, but little or no improvement in strength is observed (ref. 2, 11). No improvement in room temperature strength of some Al-alloys by \( \delta-Al_2O_3 \) fibres is also predicted by the modified rule of mixtures (Friend, ref. 2). The strength of both the Fiberfrax- and Fibermaxfibres is lower than the strength of \( \delta-Al_2O_3 \) fibres (see table 1), so little or no improvements in the room temperature strength of Al-alloys are to be expected.

Considering the stiffness of the fibres made by Carborundum little or no improvements are to be expected in the room temperature stiffness of Al-alloys using the amorphous Fiberfrax fibres.

A little increase in stiffness of Al-alloys is to be expected using the crystalline Fibermax values. Because of the poor mechanical properties of the fibres manufactured by Carborundum, it is doubtful that these fibres are suitable for the reinforcement of aluminium alloys.

As described in chapter 2.4 the debris content of the short fibres should be as low as possible. The stiffness of the composite is mainly influenced by the
fibre content, the strength of the composite can be further improved by an optimization of the consolidation, the working processes and proper heat treatments.

<table>
<thead>
<tr>
<th>Table 1</th>
<th>Mechanical properties of some materials</th>
</tr>
</thead>
<tbody>
<tr>
<td>Material</td>
<td>Density ( (\text{kg/m}^3) )</td>
</tr>
<tr>
<td>SiC (whiskers)</td>
<td>3100</td>
</tr>
<tr>
<td>Saffil* ((\delta-\text{Al}_2\text{O}_3))</td>
<td>3300</td>
</tr>
<tr>
<td>Fiberfrax** ((\text{amorphous fibres}))</td>
<td>2700</td>
</tr>
<tr>
<td>Fibermax** ((\text{crystalline fibres}))</td>
<td>3000</td>
</tr>
<tr>
<td>2024 Al</td>
<td>2770</td>
</tr>
<tr>
<td>6061 Al</td>
<td>2700</td>
</tr>
<tr>
<td>7075 Al</td>
<td>2800</td>
</tr>
</tbody>
</table>

* Trade name of Imperial chemical industries

** Trade names of Carborundum resistant materials

*** at optimum heat treatment
3 EXPERIMENTAL PART

3.1 Materials

Three chopped alumina silica fibres were characterised. The chemical composition fibre content and mean diameter are listed in table 2. This table is composed from data sheet of Carborundum. All of the fibres were prepared to CEF 101 characteristics, that means the fibre length/diameter ratio should be 10-100. The Fibermax bulk fibres should be a single phase polycrystalline fibre. According to the phase diagram, see fig. 3.1, and the chemical composition this phase is mullite (= $3\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$) (ref. 16, 17). The other two fibres are amorphous and should contain no crystalline phases.

![Phase diagram for the system Al$_2$O$_3$ - SiO$_2$](image)

**Fig. 3.1.** Phase diagram for the system Al$_2$O$_3$ - SiO$_2$ (ref. 16), showing at about 75 wt% Al$_2$O$_3$ a single phase mullite region

3.2 Experimental conditions

On the alumina silicate fibres the following investigations have been performed:

1. By means of SEM the morphology of the fibres has been characterized.
ii. By means of X-ray diffraction either the crystallinity of the fibres or special crystalline phases in the material has been verified.

iii. Besides the content of $\text{Al}_2\text{O}_3$ and $\text{SiO}_2$ the amount of impurities has been determined by means of chemical analysis.

The specimens for SEM were prepared as follows: A small amount of the chopped fibres was stucked on double coated adhesive tape. By blasting with a light stream of air the excess of the fibres was removed and a thin layer of fibres was formed on the tape. The tape was mounted on the specimen holder and was coated by a layer of Au of approximately 20 nm in a sputtering apparatus of Balzers (S.C.D046) working at 6 KeV, 15 mA during three minutes. The Au-coated fibres were examined in a Jeol electron microscope, J.SM-43, working at 25 KeV.

The X-ray diffraction pattern were measured by the powder Guinier-de Wolf technique using monochromatic CuKα-radiation. These experiments were performed by the group of X-ray diffraction service in the department of Materials Science of Delft University of Technology. X-ray diffraction occurs for which the Bragg's law (1913) is satisfied:

$$2d_{hkl}\sin\theta = n\lambda \quad (3.1.)$$

where

- $d_{hkl}$ is the interplanar spacing of the material
- $\theta$ is the angle between the incident beam and the diffracting planes
- $\lambda$ is the applied wavelength of the X-rays ($\lambda\text{CuKα} = 1.5405\text{Å}$)

Using this X-ray diffraction technique the nature of the crystalline phases in materials can be determined, by comparing the diffraction pattern of the unknown material (characterized by the interplanar spacings) with a known diffraction pattern of the JCPDS index (ref. 20).

Chemical analysis of the fibres were performed by the chemical laboratory in the department of Material Science of Delft University of Technology. Qualitative analysis for the determination of the nature of impurities were performed using
the X-ray fluorescence technique. Quantitative analysis of the elements were performed by wet analysis. The fibres were melted with KOH in a crucible made of Ni and the melt was dissolved in diluted sulfuric acid. Si and Al were determined by gravimetric analysis as SiO₂ and Al₂O₃ respectively. Na and Fe were analysed using atomic absorption spectroscopy. Ti was determined by means of spectrophotometry.

3.3. Experimental results

3.3.1. The morphology of the fibres

3.3.1.1. The CEF 101FM fibres

Fig. 3.2 shows typical SEM micrographs of the single phase fibre CEF 101FM. Fig. 3.2.a shows an overview of a large area of the specimen at low magnifications, fig. 3.2.b is an enlargement of the middle of fig. 3.2.a. Other micrographs at different positions are shown in fig. 3.2.c and e. Typical surface features are demonstrated in fig. 3.2.d, which is an enlargement of fig. 3.2.c.

The following conclusions can be drawn from the micrographs of fig. 3.2.b, c and e: (i) The morphology of the chopped fibres CEF 101FM is not dependent of the position of the specimen, (ii) The morphology of the fibres is irregular (iii). The fibre content, with a length/diameter ratio substantially larger than 1, is low, probably lower than 50 wt%, the value listed in table 2. (IV) The observed diameter of the fibres are in the range of 2-3,5 μm, which is in agreement with table 2. (V) The material contains a lot of debris, non-fibre like irregularly particles, varying in size from 1-20 μm.

Fig. 3.2.d clearly indicates the small undefined impurities (size about 0,5 μm) stucked on the surface of the fibres. Because of the high debris content of the fibres no further attempts were made to measure the length/diameter ratio of the CEF 101 fibres.
Fig. 3.2. SEM micrographs of the crystalline CEF 101FM alumina silica fibres at different magnifications, as indicated by the horizontal bars.
3.3.1.2. The CEF HSA 101 fibres

Fig. 3.3 shows typical SEM micrographs of the amorphous fibre CEF HSA 101. Fig. 3.3.a shows an overview of a large area in the specimen. Fig. 3.3.b which is an enlargement of fig. 3.3.a, fig. 3.3.d and fig. 3.3.c show the morphology at two different positions. Typical surface features are visible in fig. 3.3.d.

The following conclusions can be drawn from the micrographs of fig. 3.3: (i) The morphology of the chopped fibres is not dependent of the position of the specimen (ii) The material has a high fibre content, probably higher than 85%, the value as listed in table 2 (iii) The diameter of the fibres varies a lot, probably between 0.5-5 μm. (IV) The debris content is low although very large inclusions, about 50 μm, are observed (V) On the surface of the fibres stucked impurities of about 0.5 μm are observed. Because of the large variation in both length and diameter of these fibres no further quantitative analysis were made on the length/diameter ratio.

3.3.1.3. The commercial CEF 101 fibres

Fig. 3.4 shows typical SEM micrographs of the commercial amorphous fibres CEF 101. Fig. 3.4.a shows an overview of a large area of the specimen. Fig. 3.3.b, which is an enlargement of fig. 3.4.a, and fig. 3.4.c show the morphology at two different positions in the specimen. Fig. 3.4.d, which is an enlargement of a fibre in fig. 3.4.c, demonstrates typical surface features of the fibres.

The following conclusions can be drawn from the micrographs of fig. 3.4: (i) The morphology of the chopped fibres is not dependent of the position of the specimen: (ii) The morphology of the chopped fibres is very irregular: (iii) The fibre content is very low, probably much lower than 50 wt% as listed in table 2. (IV) The observed diameters vary between 0.5-10 μm, which is a broader distribution than listed in table 2. (V) The commercial chopped fibres contain a lot of debris, non-fibre like irregular formed particles varying in size from 1-50 μm. (VI) On the surface of the fibres stucked impurities of about 0.5 μm are observed. Because of the high debris content of the fibres further attempts in measuring length/diameter ratio of CEF 101 fibres seem to be useless.
Fig. 3.3. SEM micrographs of the amorphous CEF HSA alumina silica fibres at different magnifications as indicated by the horizontal bars.
Fig. 3.4. SEM micrographs of the amorphous commercial CEF 101 alumina silica fibres at different magnifications as indicated by the horizontal bars.
3.3.1.4. SiC and $\delta$-Al$_2$O$_3$ fibres

The morphology of silicon carbide whiskers from an experimental lot were examined by means of SEM and typical results are shown in fig. 3.5. As compared with alumina silica fibres these SiC whiskers contain a high fraction of fibres with a very high length/diameter ratio. A little amount of debris is observed, an example of an undefined particle is demonstrated in fig. 3.5.a. The observed diameter varies between 0.5 - 1.5 $\mu$m. These SiC-whiskers have a corrugated surface appearance as demonstrated in fig. 3.5.b, which is a typical property for SiC made from rice hulls (ref. 5, 6, 18).

The observed morphology of these SiC-whiskers is in agreement with the result from Divecha et al (ref. 18), see fig. 3.6, who used SiC with a debris content of less than 10 wt% for the reinforcement of Al-alloys.
Fig. 3.5. SEM micrographs of silicon carbide whiskers at two different magnifications as indicated by the horizontal bars. These whiskers contain a high fraction of fibres and a low content of debris.
Fig. 3.6. SEM micrograph of silicon carbide whiskers from pyrolized rice hulls (ref. 18)

To compare the morphology of the examined alumina silica fibres with saffil fibres, fig. 3.7 shows a SEM micrograph of an uninfiltarated Saffil preform (ref. 2). For the purpose of composite fabrication by a pressure infiltration technique the $\delta$-Al$_2$O$_3$ fibres were chopped, milled and sized to give a pre-selected range of lengths. Fig. 3.7 demonstrates the fibre like nature of the short $\delta$-Al$_2$O$_3$ fibres. Typical narrow distributions of the diameter and the length of the used Saffil fibres is illustrated in fig. 3.8. The fibre length/diameter ratio varies from 100-200.

Fig. 3.7. SEM micrograph of an uninfiltarated Saffil ($\delta$-Al$_2$O$_3$) preform (ref. 2)
Fig. 3.8. Distributions of (a) diameter and (b) length of $\delta$-$\text{Al}_2\text{O}_3$ fibres employed by Clyne et al (ref. 11)

3.3.2. The crystalline phases in the fibres

Appendix I gives the JCPDS-indexes used for the identification of the crystalline phases in the alumina silica fibres.

The CEF 101FM fibres have a crystalline structure and mullite is the primary component although a small amount (about 1-5 wt%) of corundum was detected. According to this X-ray analysis these fibres have not a single phase structure, contrary to the listed structure indicated by Fryatt (ref. 12). As mentioned by Aramaki et al (ref. 17) mullite can dissolve up to 3 wt% $\text{Al}_2\text{O}_3$. The examined CEF 101FM fibres probably consist of finely grained corundum in polycrystalline mullite. Probably the low ultimate tensile strength of 0.85 GPa (table 2, pp 17) can be explained by this dual phase structure.

The CEF 101HSA fibres primary have an amorphous structure (at low Bragg-angles diffuse scattering was observed), but also some crystalline $\alpha$-$\text{Al}_2\text{O}_3$ and $\alpha$-$\text{SiO}_2$ was detected.

The CEF 101 commercial fibres also have an amorphous structure, but some crystalline $\alpha$-$\text{Al}_2\text{O}_3$ was determined.
3.3.3. Chemical analysis of the fibres

The following data are the results of the chemical analyses. The quantity of the elements, indicated by their oxide, is given in weight percent (wt%).

investigated fibre

<table>
<thead>
<tr>
<th>oxide</th>
<th>CEF 101FM</th>
<th>CEF 101HSA</th>
<th>CEF 101</th>
</tr>
</thead>
<tbody>
<tr>
<td>Al₂O₃</td>
<td>67.5 ± 1.0</td>
<td>50.4 ± 1.0</td>
<td>50.8 ± 1.0</td>
</tr>
<tr>
<td>SiO₂</td>
<td>30.4 ± 1.0</td>
<td>47.2 ± 1.0</td>
<td>48.5 ± 1.0</td>
</tr>
<tr>
<td>Na₂O</td>
<td>~ 0.2</td>
<td>~ 0.2</td>
<td>~ 0.2</td>
</tr>
<tr>
<td>Fe₂O₃</td>
<td>~ 0.1</td>
<td>0.60 ± 0.05</td>
<td>~ 0.1</td>
</tr>
<tr>
<td>TiO₂</td>
<td>0.5 ± 0.1</td>
<td>2.0 ± 0.1</td>
<td></td>
</tr>
<tr>
<td>CaO</td>
<td>0.5 ± 0.1</td>
<td>0.5 ± 0.1</td>
<td>0.5 ± 0.1</td>
</tr>
<tr>
<td>K₂O</td>
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<td>~ 0.02</td>
<td>~ 0.02</td>
</tr>
<tr>
<td>SrO</td>
<td>&lt; 0.01</td>
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<tr>
<td>ZrO</td>
<td>&lt; 0.01</td>
<td>&lt; 0.01</td>
<td>&lt; 0.01</td>
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</table>

Comparing this result with the listed data of table 2 (pp 32) the following observations are made: (i) The Al₂O₃- and SiO₂-quantity is in reasonable agreement with the listed amount. (ii) The TiO₂ quantity (0.5%) of the CEF 101FM fibre is much higher than the listed value (0.01%). (iii) In all of the fibres 0.2% Na₂O, 0.5% CaO and some ZrO was detected contrary to the data of table 2.

3.4. Discussion of the experimental results

The three examined alumina silica fibres possess a very irregular morphology in comparisation with the appearance of SiC-short fibres or Saffil-fibres. Firstly a high debris content was observed especially in the CEF 101FM and the CEF 101 fibres, although the CEF 101HSA fibres contain large particles of about 50 μm.
Secondly a large variety in lengths and diameters was observed especially in the CEF HSA101 fibres. For the purpose of composite fabrication it is recommended that the debris of the fibres should be removed and the fibres are preselected to give narrow distributions of lengths and diameters comparable with the results of Clyne et al (ref. 11), who employed the Saffil-fibres.

The X-ray diffraction measurements indicate that the crystalline fibres CEF 101FM are not single phase fibres, but at least consist of two crystalline phases of mullite and $\alpha-Al_2O_3$.

In both the amorphous fibres some crystalline phases were observed. The CEF 101HSA fibres contain some $\alpha-SiO_2$ and the commercial CEF 101 fibres contain some $\alpha-SiO_2$ and $\alpha-Al_2O_3$.

The strength of especially the crystalline fibres probably can be improved by developing a manufacturing process which gives a one crystalline fibre with a single phase mullite structure (ref. 17).

The chemical composition is in reasonable agreement with the composition listed by Carborundum (table 2), although some other impurities like Na and Ca were detected. These impurities can dissolve in the Al-matrix and probably deteriorate the properties of the metal matrix composites.

<table>
<thead>
<tr>
<th>code</th>
<th>chem. composition (wt%)</th>
<th>fibre content (wt%)</th>
<th>d (um)</th>
</tr>
</thead>
<tbody>
<tr>
<td>CEF 101FM</td>
<td>72% $Al_2O_3$</td>
<td>50</td>
<td>2-3.5</td>
</tr>
<tr>
<td>Fibermax bulk fibres</td>
<td>28% $SiO_2$, 0.002% $Fe_2O_3$</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>0.01% $TiO_2$</td>
<td></td>
<td></td>
</tr>
<tr>
<td>CEF 101HSA</td>
<td>43.4% $Al_2O_3$, 53.9% $SiO_2$</td>
<td>$\geq$ 85</td>
<td>1.2</td>
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<tr>
<td>Fiberfax HSA fibres</td>
<td>0.8% $Fe_2O_3$, 1.6% $TiO_2$</td>
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<td></td>
</tr>
<tr>
<td></td>
<td>0.1% $K_2O$, 0.1% $Na_2O$</td>
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</tr>
<tr>
<td>CEF 101</td>
<td>47% $Al_2O_3$, 52.8% $SiO_2$</td>
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<td>2-2.5</td>
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<tr>
<td>Fiberfax R bulk fibre</td>
<td>0.15% $Na_2O$, 0.04% $Fe_2O_3$</td>
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</tbody>
</table>

Table 2 Data of investigated fibres (based on datasheet of Carborundum)
4. CONCLUSIONS, RECOMMENDATIONS

To improve the room temperature stiffness and strength of aluminium alloys with ceramic short fibres the following properties of the reinforcement are required: (i) good mechanical properties i.e. a Young's modulus and an ultimate tensile strength, which are much higher than those of the Al-alloys, (ii) a high length/diameter ratio probably higher than 50 (ref. 11), (iii) an as low as possible debris content, (lower than 10%, ref. 18).

It is doubtful if the three examined alumina silica fibres are suitable for the reinforcement of Al-alloys because of (i) the low Young's modulus and UTS, when they are compared with Al-alloys, (ii) the very irregular morphology and high debris content.

The mechanical properties of the alumina silica fibres perhaps can be improved by optimizing the structure of the fibres. The morphology can be improved by pre-selecting the fibres and removing the debris.

It is recommended that SiC and 6-Al2O3 fibres are further characterized because of their promising mechanical properties and morphology.

Acknowledgements

The author thanks professor J. Schijve for reading the manuscript, N.M. van der Pers and J.F. van Lent for performing the X-ray experiments, A.H.L.M. Kleinhout and G.J. Mareschijn for doing the chemical analysis, F.G.C. van Oostrum for developing the micrographs and S. Alibaks for typing this report.
5. REFERENCES

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5. SiC-reinforced aluminium metal matrix composites, S.V. Nair, I.K. Tien, R.C. Bates

   NASA-TP-2302 (28 pages)


8. Introduction to metal matrix composites, K.G. Kreider.
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   Westport, Conn., Technomic publishing co., 1969, pp 77.


16. Ternary equilibrium diagrams, D.R.E. West.

17. Revised Phase diagram for the system Al2O3-SiO2, S. Aramaki, R. Roy.


20. JCPDS indexes of the joint committee on powder diffraction standards (1986).
Appendix I JCPDS-indexes

The following tables give the ICPDS-indexes of Mullite (\( = 3 \text{Al}_2\text{O}_3 \cdot 2 \text{SiO}_2 \)), number 15-776, corundum (\( = \alpha-\text{Al}_2\text{O}_3 \)), number 10-173 and alpha-Quartz (\( = \alpha-\text{SiO}_2 \)), number 5-0490.

The left corner of each index shows the most important diffracting planes, characterized by \( d_{hkl} (\AA) \) giving the strongest intensities, indicated by \( I/I_0 \).

### 15-776 MINOR CORRECTION

<table>
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<tr>
<th>( d )</th>
<th>3.39</th>
<th>3.43</th>
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<tr>
<td>1/1</td>
<td>100</td>
<td>95</td>
<td>60</td>
<td>50</td>
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</table>

| \( d \) | 5.39 | 3.774 | 3.428 | 3.530 | 2.886 | 2.694 | 2.542 | 2.428 | 2.393 | 2.308 | 2.292 | 2.206 | 2.121 | 2.106 | 1.969 | 1.923 | 1.887 | 1.863 | 1.841 | 1.7954 |
|--------|------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|
| 1/1    | 50   | 200   | 120   | 210   | 20    | 220   | 111   | 130   | 310   | 202   | 201   | 20    | 120   | 320   | 221   | 204   | 320   | 221   | 311   | 230   |
| \( hkl \) | 1,7125 | 1,7001 | 1,6940 | 1,5999 | 1,5786 | 1,5644 | 1,5467 | 1,5242 | 1,5067 | 1,4811 | 1,4731 | 1,4605 | 1,4421 | 1,4260 | 1,4046 | 1,3932 | 1,3494 | 1,3467 | 1,3564 | 1,330 |

\( d_{hkl} \) (\( \AA \)) for Mullite

Sample was prepared at NBS by C. Robbins. Spec. anal.: 0.01 to 0.1% Fe, and 0.001 to 0.01% each of Ca, Cr, Mg, Mn, Ni, Ti, and Ir.

Pattern was made at 25°C.

Chem. anal. showed 61.6 Al\(_2\)O\(_3\), 38 (mole. %) SiO\(_2\).

### 10-173 MINOR CORRECTION

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<tr>
<td>( hkl )</td>
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<td>100</td>
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\( d_{hkl} \) (\( \AA \)) for Corundum

Sample annealed at 1400°C for four hours in an Al\(_2\)O\(_3\) crucible. Spec. anal. showed <0.1% K, Na, Si; <0.01% Ca, Cu, Fe, Mg, Pb; <0.001% B, Cr, Li, Mn, Ni.

Corundum structure.

Pattern made at 25°C.
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**Silicon IV Oxide**

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**Alpha Quartz**

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**Mineral from Lake Tegaway, N.C.**

- CO₃:Si Al₂:CO₂:Ca:Cu:Fe:Mg:
- X-ray pattern at 35°C.

<table>
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**Cut off:** CuKα₁ 1.5406

**Reflections:** 1.5445, 1.553

**Color:**