JOURNAL OF MATERIALS SCIENCE 25 (1990) 3273–3280

Compatibility between alumina fibres and aluminium

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An investigation is carried out on the interfacial wetting behaviour and reactions between aluminium and alumina fibres (85 mass% Al_2O_3 and 15 mass% SiO_2). Aluminium is coated onto alumina fibres by a vacuum evaporation technique and the surface of the fully coated fibres and the edge of the partially coated fibres are examined by scanning electron microscope after heat treatments at various temperatures. Within a temperature regime between 943 and 1273 K, occurrence of such interfacial reactions as $4Al(l) + Al_2O_3(s) \rightarrow 3Al_2O(g)$ and $4Al(l) + 3SiO_2(s) \rightarrow 2Al_2O_3(s) + 3Si(s)$ are detected. It is found that molten aluminium can cover the alumina fibre surface but it peels off near the edge of the coating film on a partially coated fibre, showing the very weak interface cohesion. This is ascribed to the lack of a stable compound formation at the interface. Results of tensile test show that the strength of the coated fibres is degraded after heat-treating at above the melting point of aluminium. The culprits for the tensile failure of alumina fibres are evaluated by the Weibull distribution theory.

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1. Introduction

Al_2O_3–Al composites have often been fabricated by the infiltration technique [1–3] where a good wettability at the interface between Al_2O_3 and aluminium is required. In general, interface cohesion of the two phases has been known to control the interface compatibility and hence the mechanical properties of the composite.

Several authors have reported the wetting behaviour of aluminium on sapphire, ruby and recrystallized alumina by the sessile drop technique [4–10]. It has been generally recognized that alumina can not be wetted by pure aluminium below 1073 K. Above this temperature the wetting behaviour has been found to be complicated and the results showed correspondingly a large scattering. In observations made by Brennan *et al.* [7] it was found that wetting, i.e. contact angle θ being less than 90°, of a preheated sapphire plaque by a molten aluminium drop with an oxide-free surface occurred at temperatures as low as 1073 K, while for an as-received sapphire the same happened above 1143 K. In other experiments made on sapphire and ruby (chromium-doped α-alumina) plaques, Champion *et al.* [6] observed that the contact angles of molten aluminium drops became less than 90° at above 1323 K. Carnaham [4] reported that at 1473 K the spreading behaviour of molten aluminium on recrystallized alumina was significantly different from that on sapphire due to the interface reaction between molten aluminium and the substrates. On recrystallized alumina the contact angle of the aluminium drop attained a steady value, whereas on sapphire it was observed to initially spread and then contract repeatedly. Using the dip coverage method Choh and Oki [11] made similar studies on Al_2O_3–0.1 mass % SiO_2 and Al_2O_3–3 mass % SiO_2 substrates at 1273 K. With the increase in the amount of SiO_2 in Al_2O_3 the incubation period for wetting became shorter and the wetting rate was decreased.

It seems from the above results that the wetting behaviour of aluminium on alumina is influenced by the character of alumina substrates to a certain extent. It is then obvious that the wetting behaviour of molten aluminium on alumina fibre would be different from what has been described in the previous investigations since the alumina fibre to be studied is also different both in micro-crystal structure and composition. In the present work the wetting behaviour of molten aluminium on the surface of alumina fibres is investigated and occurrence of interface reactions is examined. It is a common practice to apply coatings of pure aluminium or its alloys on alumina fibres prior to fabrication of Al_2O_3–Al composites to modify the fibre surface characteristics. The results of the present work are therefore expected to provide a more direct reference to the interface characteristics in Al_2O_3–Al composites than those obtained in the sessile drop experiments.

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TABLE I Nominal properties of alumina fibre

Composition (mass %)	85 Al_2O_3 15 SiO_2
Density (Mg m^{-3})	3.2
Diameter (μm)	10.5
Tensile strength (GPa)	1.8
Elastic modulus (GPa)	210
Highest service temperature (K)	1523
Expansion coefficient (K^{-1})	8.8×10^{-6}
Specific heat (kJ kg^{-1} K^{-1})	0.71–1.00

2. Experimental procedure

Alumina fibres of 85 mass % Al_2O_3 and 15 mass % SiO_2 used in the present investigation were cordially provided by Sumitomo Chemical Co. Ltd without the usual sizy coating. Nominal properties given by the supplier of the alumina fibres are listed in Table I. Diameter measurements were made here on 54 fibres to give the values of $14.32 \pm 0.45\,\mu$m being different from Table I. The value obtained here should be more accurate and is to be used in calculation of tensile strength.

Aluminium was coated onto the surface of the alumina fibres using an ion beam evaporation apparatus, ULVAC DRP-40E, under a 10^{-5} torr vacuum. Aluminium ingot with a purity of 99.99% was re-shaped by arc melting in an argon atmosphere into a dimension required by the deposition apparatus. Alumina fibres were coiled around and attached to a drum frame which was rotated at a rate of 10 r.p.m. A uniform aluminium coating layer of ca. 0.2 μm thick can be achieved during 1.8 ksec deposition. The coated fibres were then encapsulated in quartz tubes under a 10^{-5} torr vacuum and heat-treated for 1.8 ksec at various temperatures between 673 and 1273 K, followed by water quenching. To avoid any chemical reaction between the aluminium coating layer and the quartz tube, the alumina fibres were sleeved by an alumina tube before the vacuum encapsulation, as shown in Fig. 1.

Wetting behaviour and interface reactions between the fibres and aluminium coating layer after the heat treatments were examined by using a JEOL JSM-15 scanning electron microscope (SEM).

Tensile test was made on single fibre by a mini-Instron type test machine made by Toyo Measuring Instrument. The gauge length was set to be 20 mm. The strength for the fibres heat-treated at each temperature was estimated based on forty measurements. To make the measured tensile strength of the coated fibres comparable with that of the uncoated, the aluminium coating layer was removed by a 30% HCl acid solution before the test.

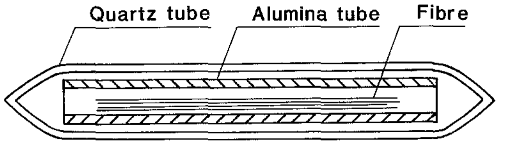

Figure 1 Sample assembly for heat treatment. Alumina fibres coated with aluminium were sleeved with an alumina tube to avoid the reaction between aluminium coating and quartz tube.

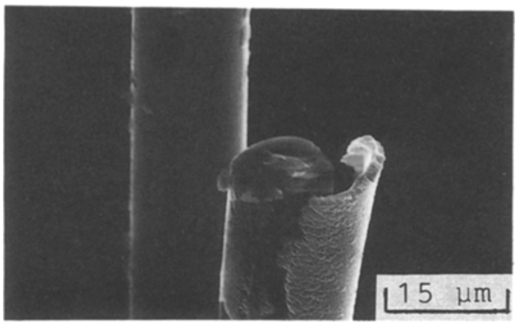

Figure 2 As-coated alumina fibres. The fracture surface shows weak interface bonding.

Multi-modal and composite Weibull distribution theories were employed to analyse the measured fibre strength.

3. Results

3.1. Interface observation

A fracture appearance of an as-coated fibre is shown in Fig. 2. It tells us that the bonding cohesion between the coating layer and the fibre should be weak.

Fig. 3 shows a coated fibre heat-treated at 973 K, which is above the melting point of aluminium, with a smooth aluminium coating layer covering the fibre surface. Internal stress due to the large difference in thermal expansion coefficient between the alumina fibre and the aluminium coating layer opens cracks to result in peeling off of the coating layer, as shown in Fig. 4, for a fibre heat-treated at 1173 K.

To examine the state of cohesion between the coating layer and the alumina fibres after the heat treatment, the surface of alumina fibres was partially coated by masking a portion of fibres along their length with an adhesive tape. Then the observation was made on the edge of the coating layer adjacent to the uncoated surface of the fibres. Although the coating layer remained on the fibre surface after the heat treatment at up to 1273 K, chemical bonding between the coating layer and the alumina fibres is apparently not likely to be built. Near the edge, the coating layer is often separated from the fibre, demonstrating the weak adhesion to the fibre surface. Fig. 5 shows such an example for a partially coated fibre heat-treated at 1173 K.

Associated with the peeling off of the coating layer, an interface reaction is found to occur above 1123 K.

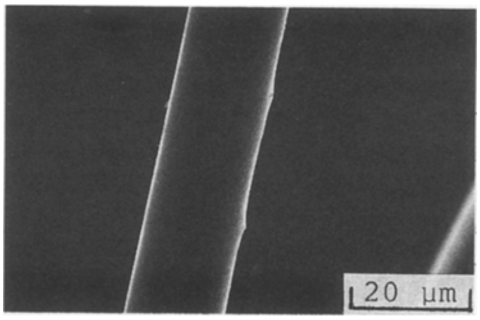

Figure 3 Surface of alumina fibre coated with aluminium and heat-treated at 973 K. Aluminium covers the fibre surface smoothly.

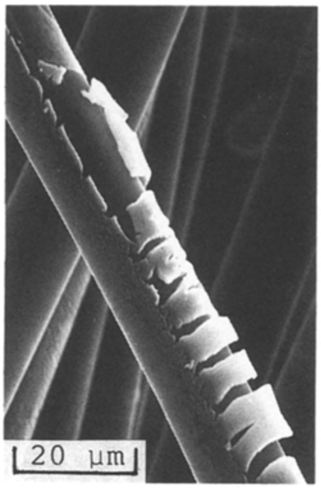

Figure 4 Cracking and peeling off of aluminium coating on the fibre surface caused by thermal stress at the interface.

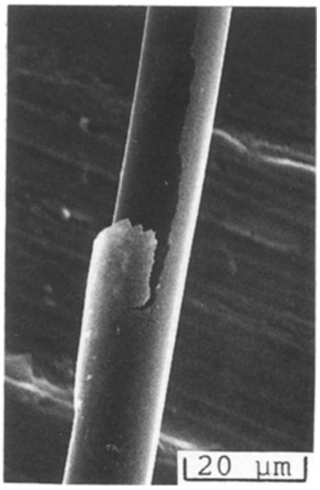

Figure 5 Surface of alumina fibre partially coated with aluminium heat-treated at 1173 K. Weak cohesion between fibre and coating layer is observed at the edge of the coating.

The swelling and bursting up of the coating layer, as shown in Fig. 6a and b, suggest that the product of the reaction should contain a gaseous phase. When the coated fibres in a quartz tube were heated above 1123 K for more than 900 sec, a thin aluminium layer could be found on the inner wall of the quartz tube. The fibre surface after the interface reaction at 1273 K is shown in Fig. 7, with the bright part showing the region with an extensive reaction.

3.2. Tensile test

To investigate the effect of interface reactions on the fibre strength, tensile tests were made on the heat-treated fibres. Note that before testing, aluminium coating layer was removed from the fibre with an HCl solution. The influence of the acid treatment on the fibre strength was proved to be negligible by comparing the measured strength between the as-received fibres before and after acid treating. The change in tensile strength with temperature for heat treatment of both coated and uncoated fibres is shown in Fig. 8. The tensile strength of the uncoated fibre increases slightly with increasing temperature for heat treatment at up to 1173 K and slowly decreases at higher temperatures. As for the coated fibres, similar results are observed for heat treatments at below 953 K, which is near the melting point of aluminium, but a sudden decrease in tensile strength is encountered above that temperature.

4. Discussion

4.1. Interface wettability and reactions

It should be first noted that the initial state of molten aluminium on the surface of alumina fibres in the present experiment is different from that in the sessile drop experiment. In the latter case wetting behaviour of molten aluminium starts from a liquid drop state, and an incubation period is always needed for the molten aluminium drop to reach an equilibrium state with the substrate. In the present experiment a uniform aluminium coating layer initially exists on the fibre surface before any heat treatment. Furthermore, aluminium was deposited on the fibre surface by using the vacuum evaporation technique, therefore aluminium coating layer would relatively be free from oxide formation. Brennan [7] has identified the difference in wetting behaviour between a molten aluminium drop having surface oxide layer and that with oxide-free surface on the same alumina substrate so that the former needs a longer incubation period than the latter.

A principle often used to discuss the wetting behaviour of a liquid metal on a solid substrate, as depicted in Fig. 9, is based on the Young's equation

$$\cos\theta = (\gamma^{s} - \gamma^{ls})/\gamma^{l}, \quad (1)$$

where γ^s is the surface tension of the solid, γ^l the surface tension of the liquid metal, γ^{ls} the interface tension between them, and θ the contact angle.

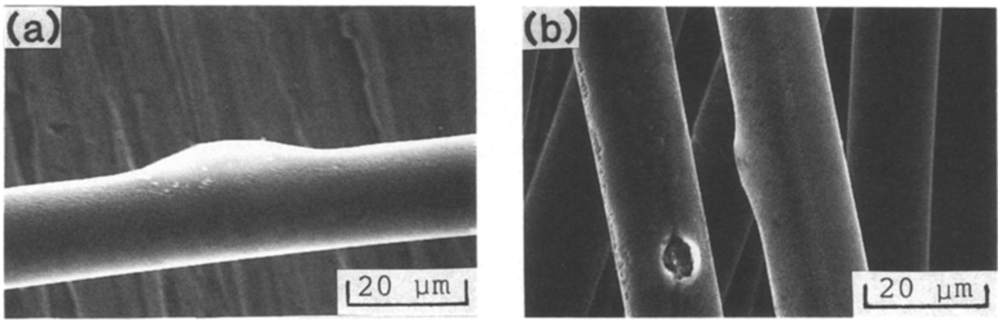

Figure 6 (a) Swelling of aluminium coating on the alumina fibre surface caused by formation of volatile Al_2O. (b) Bursting up of aluminium coating on the alumina fibre by the volatile Al_2O formed at the interface.

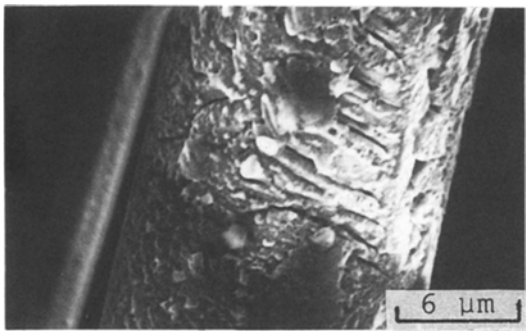

Figure 7 Surface morphology of the fibre after the interfacial reaction expressed by Equation 2. Brighter region shows the occurrence of extensive reaction.

It is generally accepted that wetting occurs only when the contact angle is acute ($\theta \leqslant 90°$). The condition for this in Equation 1 is $\gamma^{ls} \leqslant \gamma^{s}$. Studies on the interface wettability of Al_2O_3–Al system have shown that the interface tension γ^{ls} is initially high, but it decreases with increasing temperature. The values of the contact angle, the surface tension and interface tension for the Al_2O_3–Al system at different temperatures are given in Table II, which is taken from the data by Livey and Murray [12].

A way to reduce the surface tension γ^{ls} is to introduce chemical bonding between the substrate and the coating material by formation of a stable compound at the interface. In other words, chemical reaction at the interface is necessary to improve wettability.

Many researchers have investigated the reactions between molten aluminium and alumina [6, 13–16]. At temperatures below 2000 K the reaction has been reported as

$$4\,Al(l) + Al_2O_3(s) \rightarrow 3\,Al_2O(g) \qquad (2)$$

Eustathopoulos *et al.* [16] reported that for this reaction the partial pressure of the volatile Al_2O is approximately 10^{-5} torr at 1133 K. As already stated, Fig. 6a and b show that at over 1123 K the reaction involves a gaseous phase. This gaseous phase would be the reaction product being expressed by Equation 2. Also found was that during quenching deposition of aluminium occurs on the inner wall of the quartz tubes in which the aluminium coated fibres are treated. It is known that when the volatile Al_2O meets a cold substrate, aluminium is deposited by the following reaction [14, 15]

TABLE II Contact angle data for aluminium on Al_2O_3 [12]

Temp (K)	γ^{l} ($N m^{-1}$)	γ^{s} ($N m^{-1}$)	γ^{ls} ($N m^{-1}$)	θ (deg)
1213	0.819	0.991	1.797 ± 0.300	170
1243	0.808	0.988	1.673 ± 0.300	148
1528	0.712	0.959	0.897 ± 0.300	85

$$2Al_2O(g) \rightarrow 4\,Al(s) + O_2(g) \qquad (3)$$

Therefore, Equation 2 together with Equation 3 well accounts for such observations.

The reaction above does not give rise to any stable bonding between the aluminium coating layer and alumina fibres, adversely by the volatile Al_2O produced at the interface the cohesion between aluminium and alumina fibres is lost as schematically shown in Fig. 10.

Another interface reaction which may be one of the reasons accounting for the sudden decrease in tensile strength of the coated fibres, as shown in Fig. 8, is the reaction between aluminium and SiO_2 contained in the alumina fibres. The reaction is expressed as

$$4Al + 3SiO_2 \rightarrow 2Al_2O_3 + 3Si \qquad (4)$$

This reaction was verified to occur in the present experiment by heating an aluminium plate on a SiO_2 crystal in vacuum of 10^{-5} torr at 1073 K for 1.8 ksec. The surface of the transparent SiO_2 crystal contacting the aluminium plate became dark coated. A similar

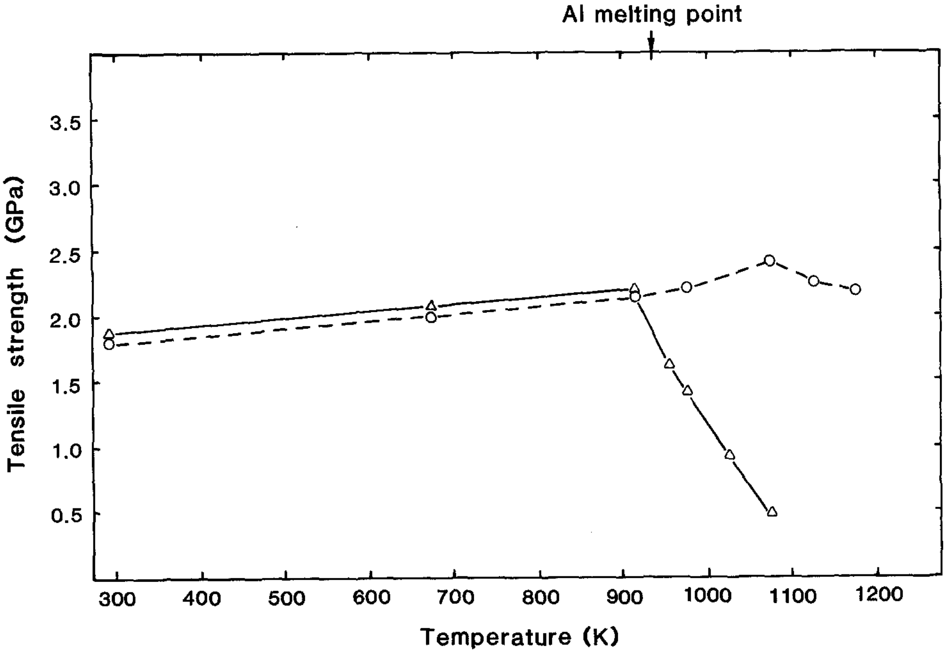

Figure 8 Effects of heat treatment on the tensile strength of the (△) coated and (○) uncoated alumina fibres. The strength is an average of 40 measurements on single fibres.

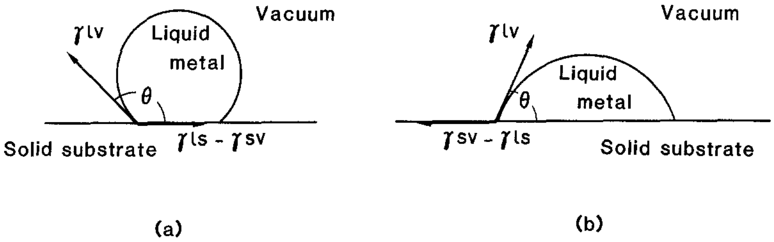

Figure 9 A schematic of a different degree of wetting of a liquid metal on a solid substrate: (a) poor wetting; (b) wetting with a contact angle less than 90°.

experiment has been made by Mori *et al.* [17] in which an aluminium plate contacting a SiO_2 substrate was heated at 1173 K for 3.6 ksec. It was found that a reaction layer was formed at the interface and an examination by electron-probe microanalysis (EPMA) revealed that it contains Al_2O_3 and silicon. This reaction is thermodynamically possible because the associated free energy change is negative between 200 and 2000 K [18]. Therefore, the effect on the fibre strength of the reaction between the alumina fibres containing SiO_2 and the molten aluminium has to be taken into account.

Weak van der Waals force between molten aluminium and alumina is considered to be responsible for the fact that the molten aluminium layer could cover the fibre smoothly as shown in Fig. 3 without the formation of droplets as in the C–Cu [19] and C–Al [20] systems. The existence of van der Waals force between molten aluminium and alumina has also been confirmed in a sessile drop experiment by Brennan [7], in which a molten aluminium drop with an oxide-free surface was found to wet an alumina substrate at the temperature as low as 873 K. Nevertheless, observation made on a partially coated fibre near the edge of the aluminium coating layer which is shown in Fig. 5 indicates that strong interfacial cohesion does not exist. Such fact can be accounted for by taking physical adsorpton into account since it is a readily reversible phenomenon. It is supposed that the molten aluminium is adsorbed on the surface of the alumina fibre due to the van der Waals bond and desorbed from the fibre surface when the coated fibre is water quenched. If chemisorption had occurred due to chemical reactions between the coating layer and the fibre, an interface would have been formed where some stable compound could be produced by the interface reaction. Such adsorption is generally irreversible.

It is clear from the above results that the interface bonding between aluminium and alumina fibre is poor even if the chemical reaction in Equation 2 exists, because the product of this reaction is volatile Al_2O rather than a stable compound. To achieve strong interface bonding and hence good wettability some suitable interface reaction should be introduced and the reaction in Equation 2 should be controlled. Alloying addition has been proved to be effective by the present authors in dealing with the interface compatibility in C–Cu system [19] and C–Al system [20]. In the case of C–Cu system there is neither any chemical reaction nor inter-diffusion between the carbon fibre and copper, the interface wettability is therefore very poor. By alloying the copper with a small amount of some element which is relatively strong carbide former such as Fe, Mo, or Cr, the contact angle of copper on carbon fibre has been shown to be reduced to less than 90°. In the case of C–Al system, the interface wettability is also known to be poor. By adding a small amount of In, Pb or Tl to aluminium the wettability has been found to be significantly improved. Since these alloying elements have low surface tension and easily form high surface adsorption, therefore their additions could cause a large decrease in the surface tension of molten aluminium. For the present case of Al_2O_3–Al system it is reported by Chou *et al.* [21] that Al_2O_3 reacts with several divalent transition metal oxides which offer the potential for the formation of strong interatomic bonds with the matrix metal. And alloying addition of 3 at % Li to aluminium was found to be effective on the improvement of the interface wettability and interface bonding by forming the compound $LiAlO_2$. Effort is currently being made by the present authors to elucidate the effect of alloying aluminium with several elements such as Mg, Li or Cu which are expected to react with alumina fibres to form stable compounds at the interface.

4.2. Tensile strength

In order to examine the effect of interface reaction on the fibre strength, results of the tensile tests for both coated and uncoated fibres are statistically analysed by using the Weibull distribution theory.

As is well known, it is inadequate to use a single Weibull distribution model to approximate a group of data into a straight line if the fibre failure is caused by

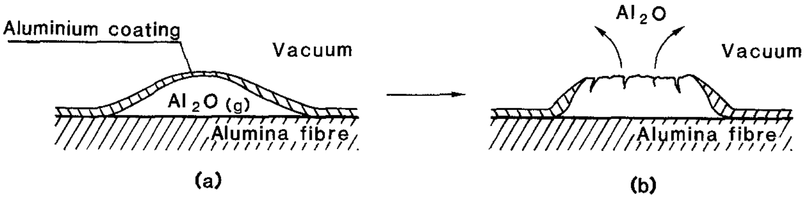

Figure 10 Schematic view of the decohesion of aluminium coating on the surface of alumina fibre by the formation of volatile Al_2O: (a) swelling up of the aluminium coating; (b) breakage of the aluminium coating.

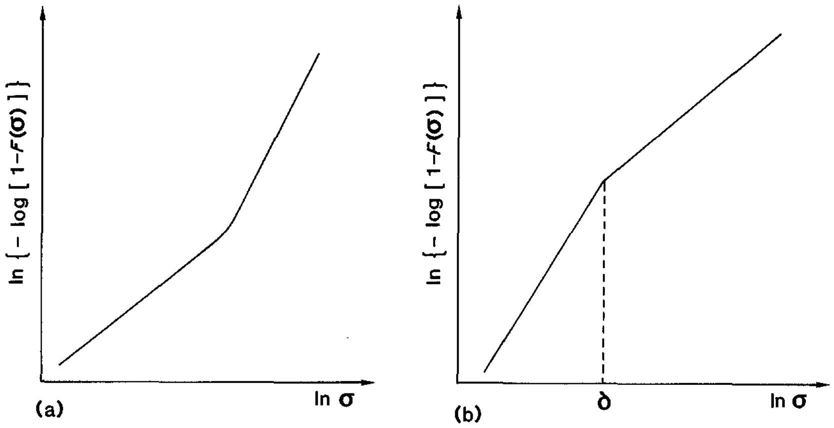

Figure 11 Weibull distribution models for (a) multi-modal Weibull distribution, (b) composite Weibull distribution, with σ being the separation parameter.

more than two kinds of defects. In the present treatment, therefore, multi-modal and composite Weibull distributions [22–26] are employed to identify the reasons for failure of the fibres.

In multi-modal Weibull distribution the distribution function is

$$F(\sigma) = 1 - [1 - F_1(\sigma)]\,[1 - F_2(\sigma)] = 1 - \exp\left[-\left(\frac{\sigma}{\sigma_{01}}\right)^{m_1} - \left(\frac{\sigma}{\sigma_{02}}\right)^{m_2}\right] \quad (5)$$

where $F_1(\sigma)$ and $F_2(\sigma)$ are the failure strength distribution functions, σ_{01} and σ_{02} the scale parameters, m_1 and m_2 the distribution shape parameters of sub-population numbers 1 and 2, respectively. This distribution function is expressed as shown in Fig. 11a.

In the composite Weibull distribution, the density function is expressed as

$$f(\sigma) = \begin{cases} f_1(\sigma) = \dfrac{m_1}{\sigma_{01}}\sigma^{m_1-1}\exp\left[-\left(\dfrac{\sigma}{\sigma_{01}}\right)^{m_1}\right], \\ \delta > \sigma \geqslant 0 \\ f_2(\sigma) = \dfrac{m_2}{\sigma_{02}}\sigma^{m_2-1}\exp\left[-\left(\dfrac{\sigma}{\sigma_{02}}\right)^{m_2}\right], \\ \sigma \geqslant \delta \end{cases} \quad (6)$$

where $f_1(\sigma)$ amd $f_2(\sigma)$ are the sub-distribution density functions of numbers 1 and 2, respectively, and δ is a separation parameter. δ should satisfy the function

$$F_1(\delta) = \int_0^{\delta} f_1(\sigma)\,d\sigma = \int_{\infty}^{\delta} f_2(\sigma)\,d\sigma = F_2(\delta) \quad (7)$$

where, $F_1\sigma)$ and $F_2(\sigma)$ are the corresponding distribution functions. The composite Weibull distribution function is expressed as shown in Fig. 11b.

For convenience, in the following discussion we will use $F_i(\sigma)$, $F_s(\sigma)$ and $F_r(\sigma)$ to describe the distribution functions of the failure strength limited by the inherent inner defects, inherent surface defects, and the defects caused by interface reactions, namely, reaction defects, respectively.

The results of the tensile tests are plotted in Fig. 12 for the uncoated fibres and in Fig. 13 for the coated fibres. Fig. 12a shows that two kinds of defects control the failures. In a similar work on the tensile strength of alumina fibre by Goda and Fukunaga [22], two defects limiting the strength were also recognized. They are concluded to be inherent inner defects and surface defects, the former being the controller of failure at higher strength level and the latter at lower strength level. The distribution obtained in the present

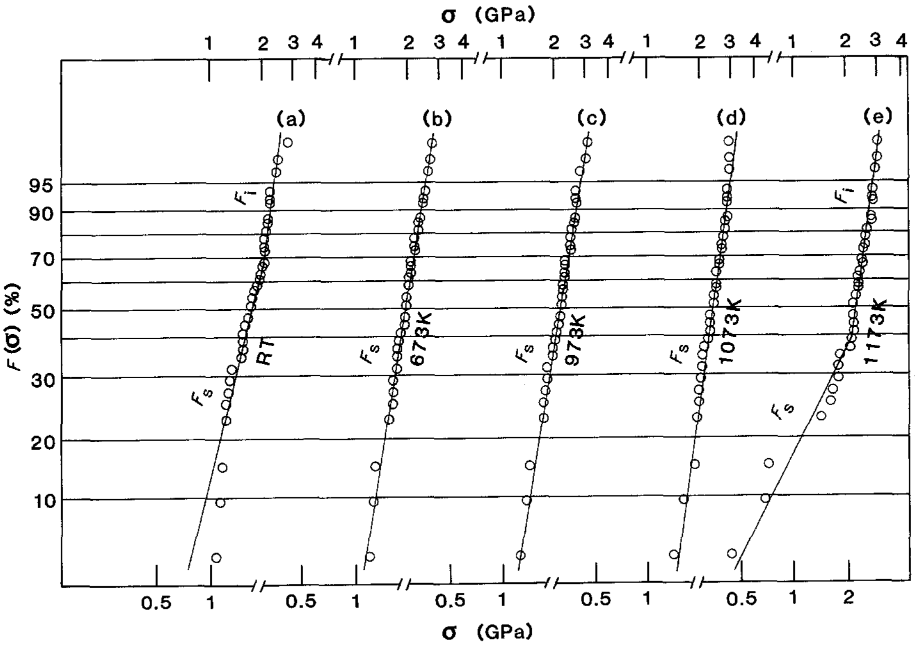

Figure 12 Weibull distribution plots for the uncoated fibres (a) without heat-treatment, and those heat-treated at (b) 673 K, (c) 973 K, (d) 1073 K, and (e) 1173 K. Note that the melting point of aluminium is ca. 940 K. F_i and F_s denote the distribution functions of the failure strength limited by inherent inner and surface defects respectively.

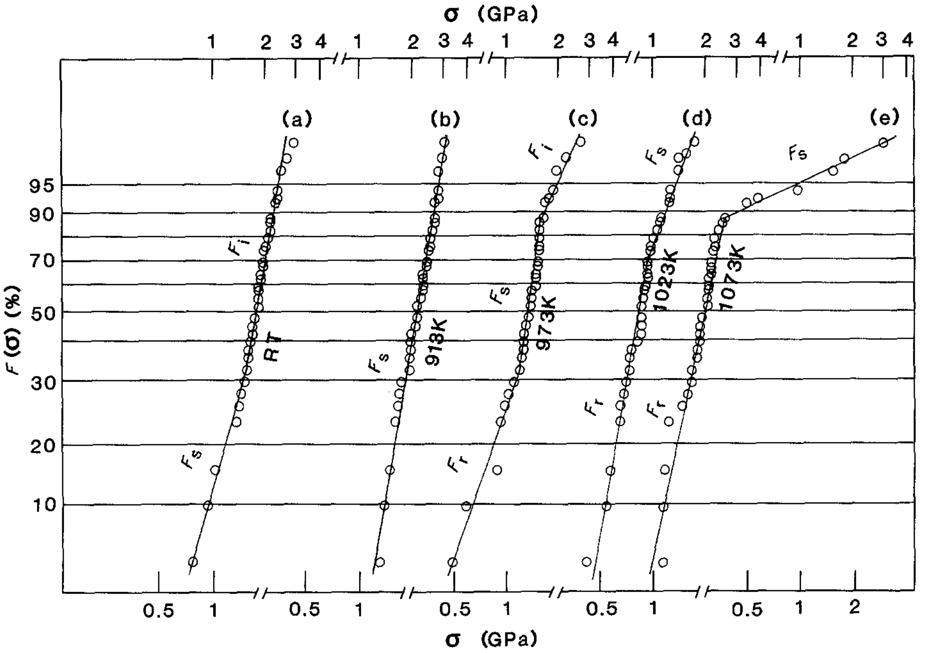

Figure 13 Weibull distribution plots for the coated fibres (a) without heat-treatment, and those heat-treated at (b) 913 K, (c) 973 K, (d) 1023 K, and (e) 1073 K. F_r is the distribution function of the failure strength limited by reaction defects.

experiment for the uncoated fibres without heat treatment is very similar to that by Goda and Fukunaga [22].

From Fig. 8 we know that below 1100 K the tensile strength of the uncoated fibres increases slightly with increasing temperature of the heat treatment and decrease again over that temperature. Corresponding to this tendency, the plotted points, as shown in Fig. 12b to e, appear to satisfy straight line and then to fit broken line again. This change is supposed to be related with the interaction between the SiO_2 and Al/$_2$O$_3$ in the fibres which influences the distribution of the potency of the inherent inner defects so that the amount of active defects could be changed during the heat treatment. A suggestive explanation for this phenomenon was given by Becher and Tiegs [27] as that the reaction couple of SiO_2–Al_2O_3 can result in the formation of aluminosilicate glass, and then after sufficiently long time and at enough time temperature, in the formation of mullite. The formation of such glass phase is expected to influence the mechanical behaviour of the fibres.

For the coated fibres heat-treated at below 913 K their strength distribution plots, as shown in Fig. 13a and b, exhibit features similar to those of the uncoated fibres heat-treated at the same temperatures. But for those heat-treated at 973 K, the Weibull plot of Fig. 13c is different from the corresponding one of the uncoated fibres (Fig. 12c). It exhibits the geometric characters of both multi-modal and composite Weibull distributions. Three kinds of defects are therefore considered to control the strength in Fig. 13c. Besides the two kinds of inherent defects, the fibre strength is also controlled by the defects caused by the interface reactions occurring above 973 K. Because the latter ones must be more serious for the tensile failure than the former inherent defects, they are assumed to appear on the low strength side. As the interface reaction becomes more extensive with increasing temperature, the defect formation rate due to the interface reactions increases, and therefore the failures occur mainly due to the two types of defects limiting the strength at relatively low level, i.e. inherent surface and reaction defects, as shown in Fig. 13d and e. The Weibull plot in Fig. 13e shows a typical composite Weibull distribution with distinct existence of a separation parameter δ, above which the failures are caused by the inherent surface defects and below which by the reaction defects.

5. Conclusion

With the results obtained from this investigation the following conclusions are drawn.

1. Aluminium film formed by vacuum evaporation can cover the alumina fibre upon melting due to the weak van der Waals force between them.
2. Interface reaction between aluminium and alumina occurs above 1123 K to produce volatile Al_2O. At lower temperatures, the reaction between molten aluminium and solid SiO_2 which is contained in the alumina fibre was verified.
3. Up to 1273 K the reactions between molten aluminium and the alumina fibre could not introduce a stable interface involving a compound formation.
4. The sudden decrease in tensile strength of the coated fibres heat-treated at the temperature a little higher than the melting point of aluminium is attributed to the interface reaction between the molten aluminium and the fibres.
5. Failures of the coated fibres heat-treated below the melting point of aluminium were controlled by the inherent inner and surface defects. Above the melting point of aluminium they were mainly controlled by the inherent surface defects and the reaction defects.

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Received 5 April
and accepted 14 September 1989