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High temperature interfacial studies in aluminium-stainless steel composites

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Interfacial shear strengths in single wire aluminium–stainless steel composites have been measured by the pull-out test, both at room temperature and high temperatures, as a function of annealing temperatures up to 823 K and times up to 24 h. The post-exposure interfacial shear strengths measured at room temperature have been found to be inversely proportional to the square root of the interfacial compound layer thickness. A tentative mechanism to explain this relationship has been proposed in terms of matrix–compound layer debonding. The growth of the compound layer during high-temperature exposure is accompanied by an increase in its microhardness, presumably resulting from a concurrent precipitation of intermetallics. The interfacial shear strength has been found to be independent of stainless steel wire diameter.

1. Introduction

It is well known that the mechanical properties of composites are critically dependent on the nature of the matrix–fibre interface. Studies on the interface have usually been concerned with the identification of the phases present and the interfacial growth kinetics as a function of fabrication conditions and high temperature exposure [1–3]. The dependence of room-temperature mechanical properties on the nature of interface has also been investigated [4–6]. Direct determination of the interfacial shear strengths (τ_i) in metal–matrix composites by the pull-out test has become increasingly popular during the last decade [7–9]. However, measurement of τ_i at high temperature by the pull-out test has seldom been reported. The measurement of room-temperature τ_i in cast aluminium–steel single wire composites by the pull-out test and the effect of fabrication conditions on τ_i have recently been reported [10]. In the present paper the effects of high-temperature exposure on the room-temperature and high-temperature interfacial shear strengths as well as on the interfacial compound layer thickness in an aluminium–stainless steel system are reported.

2. Experimental procedure

Single wire composites of AISI 304 stainless steel (18.25 mol % Cr, 8.02 mol % Ni and 0.07 mol % C) in a matrix of aluminium alloy (2.22 mol % Mg and 1.78 mol % Si) were prepared as follows. The wires were first degreased in trichloroethylene, pickled in 10% HCl for 10 sec and then dipped in aqueous flux solution containing 10 mol % borax for about 30 sec. The fluxed wires were then dried in hot air and immediately given a precoating of the aluminium alloy by hot-dipping at 973 K for 45 sec. The flux as well as the precoating conditions employed were selected based on the results reported by Patankar *et al.* [10]. Pull-out specimens were fabricated by placing the aluminized wire in the centre of a mild steel mould and pouring the molten aluminium alloy at 1023 K to obtain cylindrical composite specimens of 15 mm diameter and about 20 mm length. These were then machined to give test specimens with various embedded lengths of wire.

The interfacial shear strengths (τ_i) were determined by performing pull-out test at a cross-head rate of 0.1 mm sec^{-1} on a Materials Test System (MTS 810) with provisions for high-temperature testing. The critical embedded lengths (l_c) were obtained from the plots of maximum loads (that could be applied prior to pull-out or wire fracture) against embedded length and τ_i calculated using the formula [7, 9] $\tau_i = \sigma_f d/4l_c$, where σ_f and d are the tensile strength and diameter of the wire. Details of the specimen configuration for the pull-out test and estimation of l_c may be found in [10].

In order to examine the effect of high-temperature exposure on the aluminium–stainless steel interface the cast composites were isothermally annealed in air at various temperatures up to 823 K and for various times up to 96 h. The resulting variations in thickness and microhardness of the interfacial layers were examined metallographically. Pull-out tests were also carried out at the annealing (exposure) temperatures after isothermal holding times of 0.5 and 24 h.

Most of the pull-out tests were conducted on cast composites with wires of 1.01 mm diameter except in those tests meant to study the effect of decreasing wire

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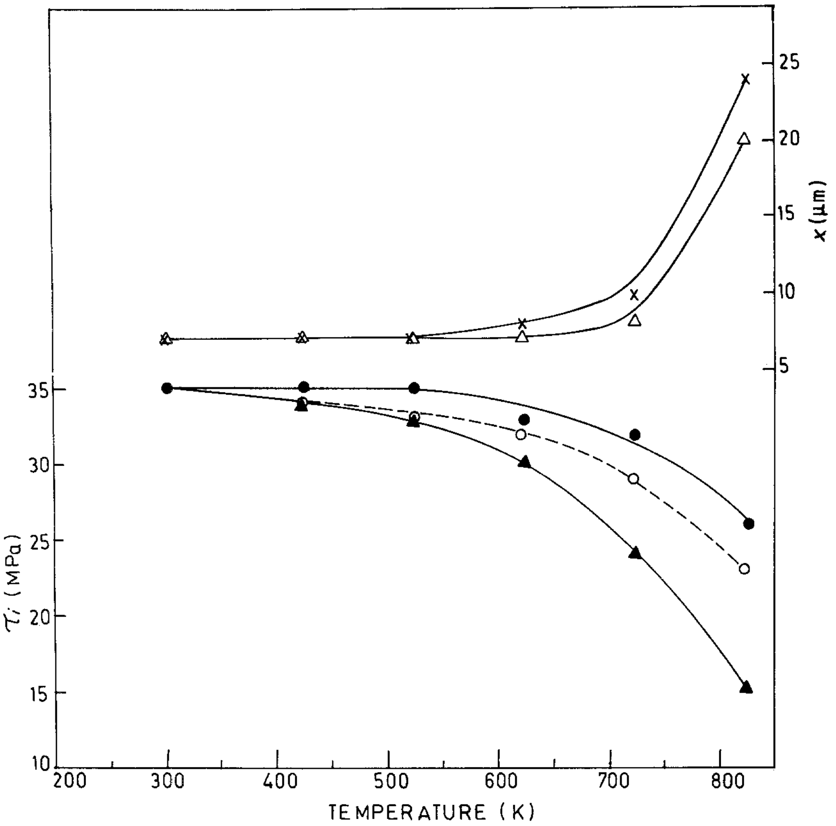

Figure 1 Influence of temperature on interfacial shear strength (τ_i) and compound layer thickness (x). Pull out tests at: (●) room temperature, holding time 24 h, and at holding temperature, holding time (○) 0.5 h, (▲) 24 h. (△, x) Measured at room temperature after cooling from holding temperature, holding times (△) 0.5 h and (x) 24 h.

diameters on τ_i, wherein diameters ranging from 1.010 to 0.126 mm were employed.

3. Results and discussion

The variation in τ_i with exposure temperature and isothermal holding time (0.5 and 24 h) is shown in the lower half of Fig. 1. The results include values of τ_i obtained from pull-out tests at the exposure temperature as well as those from tests carried out at room temperature after cooling from the exposure temperature. It can be seen that there is initially a gradual decrease in τ_i followed by a more rapid decrease at higher temperatures. The initial decrease in high-temperature τ_i is only 5% of the as-cast value provided the temperature does not exceed 500 K (for 24 h exposure) and 540 K (for 0.5 h exposure). However, if one considers the post-exposure τ_i values measured after cooling to room temperature, the same drop of 5% is observed only after an exposure of 24 h at 650 K. At temperatures above these, τ_i exhibits a relatively rapid decrease.

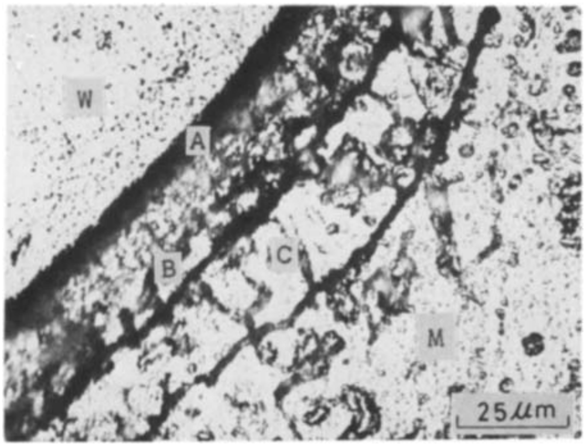

Figure 2 Optical micrograph showing details of aluminium–stainless steel interface after exposure to 423 K for 24 h; etched transverse cross-section: wire (W), dark-etching zone (A), light-etching zone (B), aluminium pre-coating (C) and aluminium matrix (M).

Fig. 2 is an optical micrograph showing the interface after a moderate high-temperature exposure (423 K for 24 h). The interface can be seen to consist of two distinct zones, a dark etching zone A close to the stainless steel wire and a light etching zone B towards the aluminium matrix. Similar features were observed in all the moderately exposed samples as well as in the as-cast sample. Energy dispersive X-ray analysis (EDAX) showed that the average atomic concentrations of the major elements in zones A and B, after 0.5 h exposure at 423 K, were 77.3 Al, 14.8 Fe, 4.9 Cr, 1.0 Ni and 96.2 Al, 1.0 Fe, 0.4 Cr, 0.4 Ni, respectively. Zone B, therefore, appears to have a composition very close to that of the matrix itself. In samples exposed to severe conditions (temperatures > 423 K and exposure times > 24 h) the distinction between zones A and B is lost and the interface consists of only the dark etching zone A. An example of such an interface is shown in Fig. 3. Mannan *et al.* [3] have also reported similar loss of distinction between the two zones. Based on electron probe micro-analysis observations, Patankar *et al.* [10] have shown that the dark etching zone essentially consists of intermetallic compounds. This zone is therefore referred to as the compound layer. The variation in the thickness (x) of this compound layer with exposure temperature is shown in the top half of Fig. 1. It is apparent that there is a close parallel between the thickening of the compound layer and the drop in τ_i.

It has been suggested [10] that pull-out at room temperature occurs most likely through the following

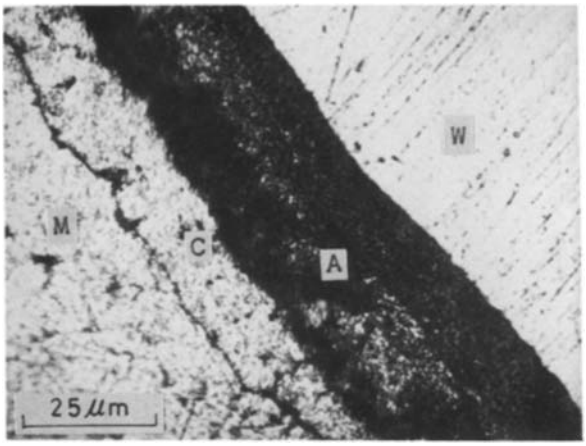

Figure 3 Optical micrograph showing aluminium–stainless steel interface after exposure to 823 K for 48 h. Wire (W), dark-etching zone (A), aluminium pre-coating (C) and aluminium matrix (M).

two steps: (i) formation and growth of cracks within the brittle compound layer, and (ii) propagation of the crack along the interface between the compound layer and the aluminium matrix. These cracks could be nucleated by the internal stresses developed in the compound layer during cooling from the exposure temperature due to thermal expansion mismatch. The thermal expansion coefficient of the compound layer is likely to be higher than that of stainless steel and closer to that of aluminium as it consists of nearly 77 at % Al (EDAX analysis). In samples cooled from soaking temperatures higher than 700 K the likelihood of such crack initiation would be higher because of the appreciable thickening of the compound layer (top half of Fig. 1). Fig. 4 shows a scanning electron micrograph of a polished longitudinal cross-section of the pulled-out wire. Cracks of various lengths and orientations can be seen in the compound layer that remains adherent to the pulled out wire. The initial stage of the pull-out could have occurred through the growth of any of those cracks which are found to terminate at the free surface of the compound layer (i.e. at the top edge of Fig. 4). If the debonding of the compound layer–matrix interface requires a critical stress and if this stress is caused by the stress concentration at the tip of the cracks in the compound layer, then τ_i should be proportional to the square root of the length of the largest crack normal to the interface. This assumption seems reasonable in view of the brittleness of the compound layer. As the length of the largest crack normal to the interface will be equal to the thickness of the compound layer, τ_i should be inversely proportional to square root of the compound layer thickness (x). Fig. 5 shows the variation of room temperature τ_i with $x^{-1/2}$ obtained by least square regression analysis. It can be seen that there is an excellent linear correlation with correlation coefficient as high as 0.99, thereby confirming the validity of the proposed model.

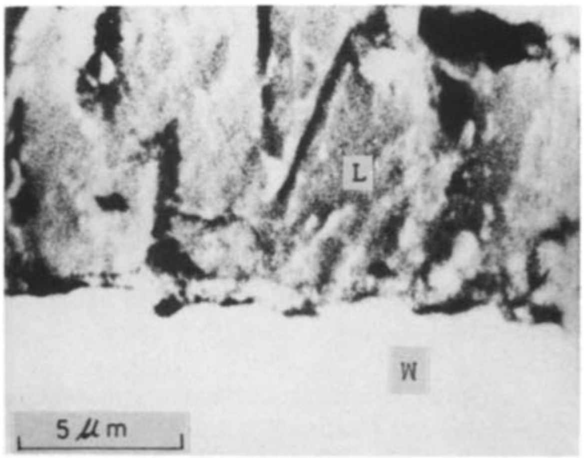

Figure 4 SEM of longitudinal cross-section of pulled-out stainless steel wire showing cracks in the compound layer. Wire (W) and compound layer (L).

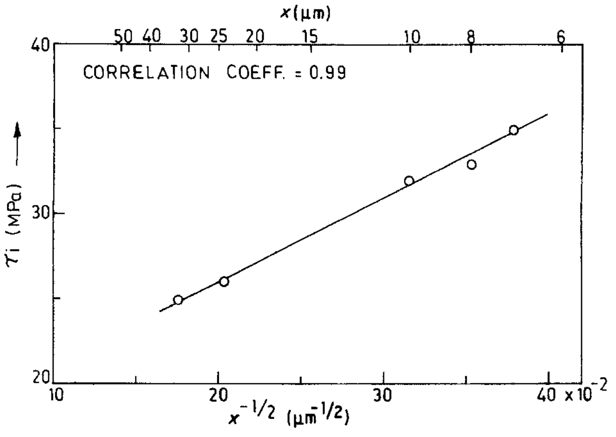

Figure 5 Correlation between interfacial shear strength (τ_i) and compound layer thickness (x).

The discussion so far pertained only to τ_i values measured at room temperature. The observation (Fig. 1) that τ_i values measured at the exposure (24 h) temperatures themselves were always lower than those measured at room temperature, may be due to the drop in yield strength of materials with increasing

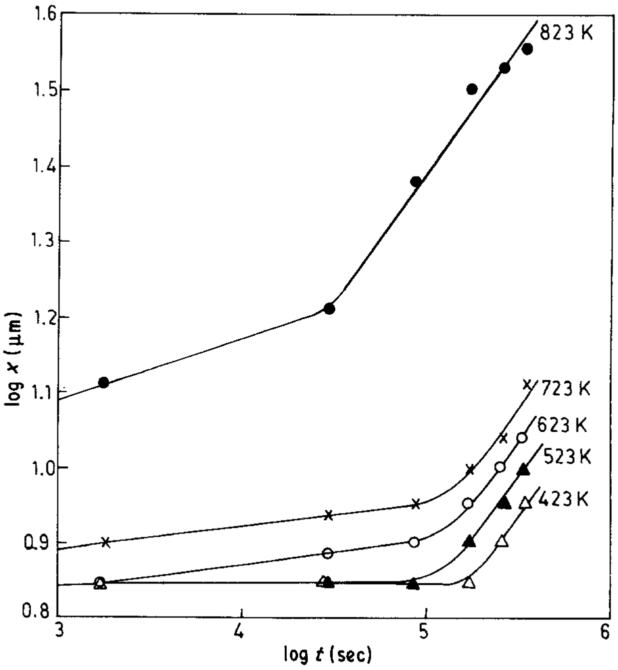

Figure 6 Variation of compound layer thickness (x) with annealing time at different temperatures.

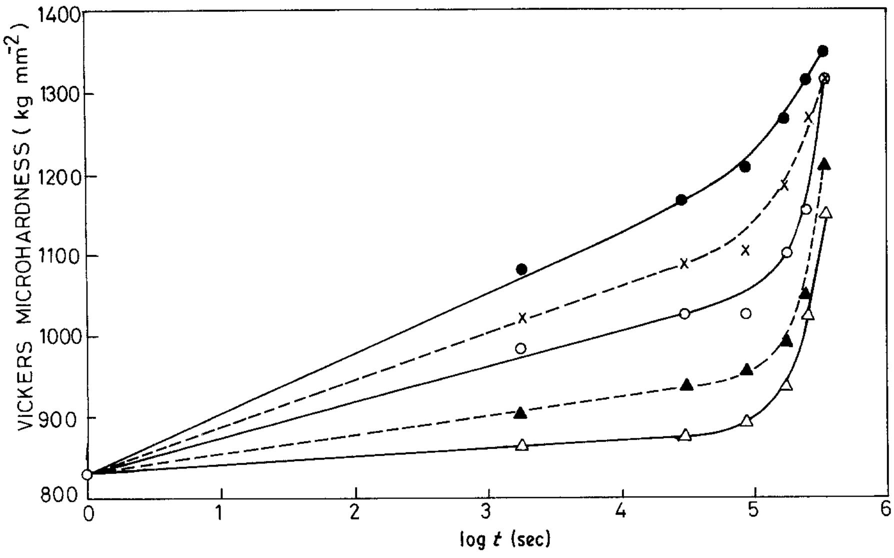

Figure 7 Compound layer microhardness as a function of exposure time at different temperatures. (△) 423 K, (▲) 523 K, (○) 623 K, (x) 723 K, (●) 823 K.

temperature. As most of the exposure temperatures in the present experiments were above half the melting point of aluminium (i.e. above 466 K) this drop could be quite rapid. Exponential drops of this nature for hardness and tensile strength values in face centred cubic metals have been reported in the literature [11]. The proposed mechanism of pull-out at room temperature is likely to be less applicable at higher temperatures because of reduced incidence of crack initiation as well as the stress-relief effect associated with the increased plasticity.

As the compound layer thickness has been found to have a profound effect on τ_i, high-temperature exposures for durations up to 96 h were carried out to follow the growth of the compound layer. Fig. 6 shows the dependence of x on exposure time, t, at various temperatures. At each temperature, there appears to be two distinct stages of growth, an early stage of slow growth lasting up to about 10^5 sec followed by a faster but constant growth rate independent of temperature. This constant growth rate is found to correspond to a slope of ~ 1/3. Isothermal growth of compound (reaction) layer as a function of time is generally described by an empirical relation of the type $x = Kt^n$, where K is a temperature dependent constant. The exponent n is generally found to be 0.5 indicating a parabolic growth as predicted for truly diffusion-controlled growth kinetics [3, 12, 13]. However, it has been suggested that n can differ from 0.5 if formation of a new (intermetallic) phase occurs in the compound layer [14]. As already reported [10], precipitation of intermetallic compounds does occur in the aluminium–stainless steel system. The value of 0.33 observed for n in the present system may be due to such precipitation effects. A value of 0.33 has also been recently reported by Ochiai *et al.* [6], for the Al–B fibre system. Indirect evidence for the precipitation of intermetallics concurrent with compound layer growth was obtained from the variation in the average Vickers microhardness (40 g, average of five readings with a scatter of ± 2%) of the compound layer region with exposure time, as shown in Fig. 7. There is indeed a marked increase in microhardness for exposure times exceeding 10^5 sec, exactly corresponding to the faster compound layer growth rate seen in Fig. 6. No attempt was made to determine values of activation energies because simple Arrhenius-type temperature dependence is unlikely in a complex multiphase system such as ours [15].

All the results discussed so far were obtained using a wire diameter of 1.01 mm for experimental convenience in conducting the pull-out test. In order to examine whether these results will be equally applicable to composites containing finer wires, the experimental values of τ_i were plotted against the wire diameters (Fig. 8). It is apparent that, within the limits

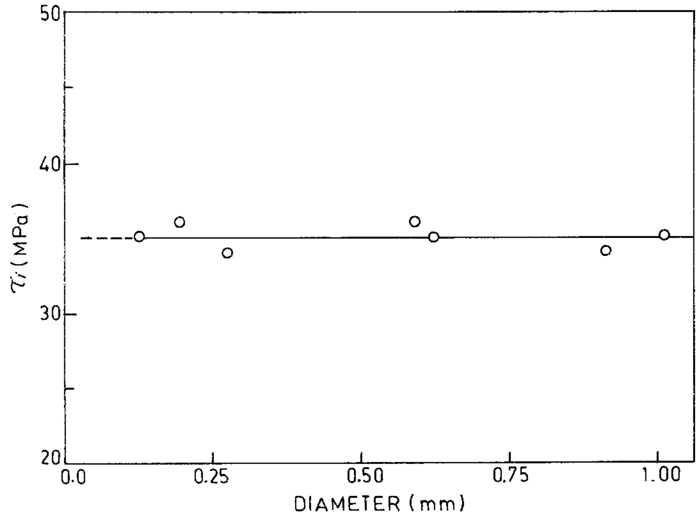

Figure 8 Effect of wire diameter on interfacial shear strength (τ_i).

of experimental accuracy, τ_i is independent of wire diameter.

4. Conclusions

1. High-temperature interfacial shear strength values in the aluminium–stainless steel system decrease by only 5% from the as-cast value of 35 MPa even after 24 h exposure at 500 K.

2. Post-exposure interfacial shear strength values measured after cooling to room temperature are found to be inversely proportional to the square root of the interfacial compound layer thickness.

3. Precipitation of intermetallics seems to occur concurrently with growth of a compound layer during high-temperature exposure.

4. Interfacial shear strength values have been found to be independent of stainless steel wire diameter.

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