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Interaction of boron fibres with aluminium melt during metallization

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The aim of the paper is to investigate interphase interactions on the fibre–melt interface boundary during metallization of boron fibres by aluminium.

Using methods of selective etching and weighing it has been stated that chemical interaction of boron fibres with the aluminium melt develops in 2 stages: firstly – boron solution in liquid aluminium occurs, secondly – formation of chemical compounds AlB_2, AlB_{12} on the interface boundary.

Mechanical tests and fractographic analysis have stated that if the fibre material is soluted in the melt then fibre strength raises due to the lower influence of surface defects; the appearance of aluminium borides on the interface boundary is followed by a decrease in the initial fibre strength.

1. Introduction

The method of liquid-phase metallization of boron fibres by pulling them through melts of adhesion-active alloys, e.g. aluminium [1–3], is widely known. This method is used to obtain mono-fibres with coatings, composite plaits, profiles, etc. [1–4]. A fibre strength of 90 to 96% of the initial strength is preserved with a simultaneous decrease of the strength variation coefficient. The partial lowering of the strength is explained by chemical interaction of boron with the melt with the formation of brittle aluminium borides, and by the lowering of the strength variation coefficient by selective solution of fibres in liquid aluminium in the place of stress concentrators [3, 5, 6].

The aim of the present work was to investigate the interphase interaction on the fibre–melt interfacial boundary during metallization of boron fibres by aluminium. The composition of the reaction components and their effect on fibre properties were also studied.

2. Materials and methods

Standard high modulus boron fibres of 140 mcm diameter were coated with aluminium by pulling through the aluminium melt (purity 99.99%). The aluminium coating apparatus consists of a resistant furnace and a mechanism by which the fibres are pulled through the crucible from top to bottom [3]. At the bottom of the crucible there is a draw plate. The melt temperature and duration of the fibre contact with the melt varied in each experiment. The products of chemical interaction were detected by electron diffraction after removing the aluminium coating in 10% solutions of NaOH, KOH or HCl. Such a set of reagents was used for selective solution of boride phases [7, 8]. In addition, X-ray diagrams were made from the fibres coated with aluminium through the coating. A change of the fibre mass due to interaction of fibres with the melt and formation of interaction products, were controlled by weighting using electron-optical scales with an accuracy up to 10^{-5} g. For this purpose the fibre mass in the initial state (before interaction) was compared with the fibre mass after contact with the aluminium melt. The aluminium coating was removed by etching in a 10% aqueous solution of NaOH, which prevents the phases being preserved on the surface. Taking into account the level of relative change in the specimen mass, the following conclusions were drawn concerning the character and kinetics of the interphase processes occurring. The fibre surface after etching was examined using a JSM-U3 microscope and a light microscope "Neofot-2" to reveal phase morphology and primary places of phase formation and fibre damage. Bend tests of fibres round arbors of variable radius were conducted. The strength was calculated from the formula

$$\sigma = (2r/2R)E_f$$

where r is the fibre radius, R the arbor radius where the fibre was fractured, E_f the Young's modulus of the fibre. Tensile strength tests using "Instron" were also carried out. Tension fracture was referred to the fibre initial diameter. No less than 100 specimens were tested under each set of conditions. Fractographic analysis was used after the tests. Fractures were detected initiating from the surface defects and one which initiated from fibre inner defects (radial cracks, core defects, defects of the interface B–W boundary) [7].

3. Results and discussion

On pulling boron fibres through ADI melt a coating is formed on the fibre surface of thickness up to 2×10^{-5} m. The coating is continuous, dense if the duration of contact with the melt is above 0.1 sec, and in the shape of islands primarily located in the intergrain grooves on the fibre surface when the contact duration is less (Fig. 1).

The dependence of relative strength of the boron

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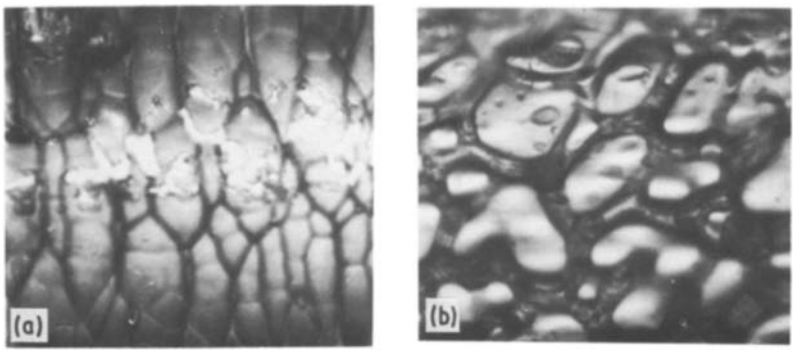

Figure 1 Surface of boron fibre after metallization when the contact time of the fibre with the melt is (a) 0.05 sec, and (b) 0.1 sec, × 1000.

fibres with the aluminium coating on the duration of the contact with the melt is shown in Fig. 2 (curve 1). The strength curve may be divided into two intervals. In the first interval the strength of fibres with an aluminium coating is higher than the initial strength. Within the time interval, a maximum value of fibre strength is achieved. Within the time interval, a maximum value of fibre strength is achieved at 1.8 of the initial strength. In the second interval the strength of fibres with aluminium coating is lower than the initial strength and falls abruptly with increasing contact time.

The curve of the change in the strength variation coefficient (Fig. 2, curve 2) may also be divided into two intervals. If the contact duration is short, an essential strength stabilization takes place, and becomes two times less. The strength begins to become unstable when the contact time is slightly larger than required for the beginning of the strength reduction.

When the duration of the contact with the melt increases, the role of surface defects in the fracture of fibres with an aluminium coating will intensify. Table I illustrates the dependence of the fibre fracture character on the contact duration. Up to 15% of the fibres fracture in the initial state, because of surface defects. After metallization when the contact time is short, the number of fractures initiated by surface defects does not exceed 10%; when the contact duration with the melt is long, up to 94% of all fibres tested fractured due to surface defects.

Fig. 2 (curve 3) shows the change of fibre mass as the result of contact with the melt. When the contact time is up to 0.5 sec one can observe continuous lowering of the mass. Longer contact time leads to a sharp increase of the fibres mass. According to Nikitin [11], loss of specimen mass as a result of the interaction with the melt is connected with solution of the specimen material in the melt and a mass increase is connected with chemical reaction between the specimen and the melt, with the formation of reaction products on the specimen surface.

The mechanism of chemical interaction of a fibre with liquid aluminium may be illustrated as follows. While in solution the boron atoms transfer into the melt through the interfacial boundary. The material of the intergrain grooves is dissolved initially. As a result, the geometry of the intergrain grooves changes; to be exact, their sharpness decreases. That is why the role of the surface in the fracture initiation is reduced. The formation of a saturated solution near the fibre and simultaneous diffusion of aluminium into the fibre,

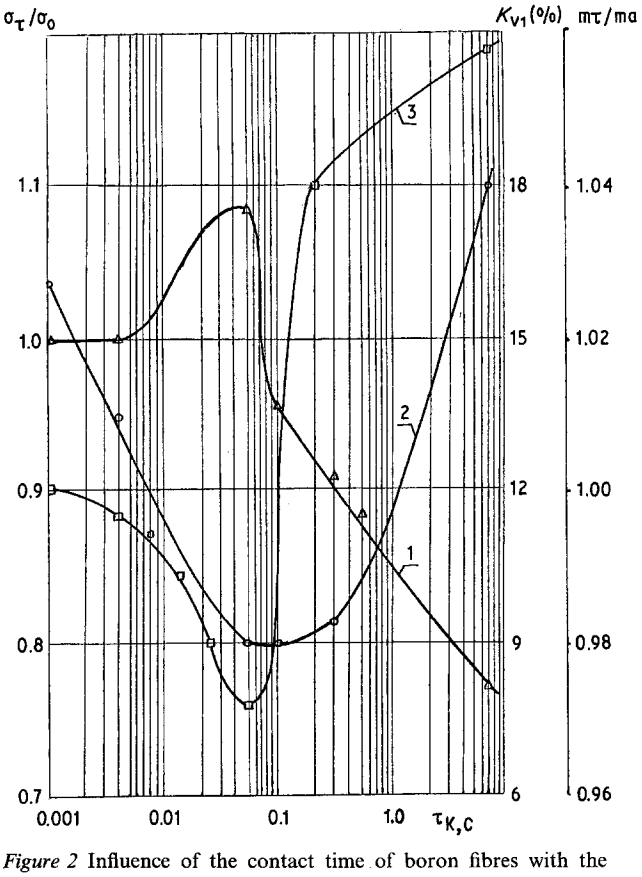

Figure 2 Influence of the contact time of boron fibres with the aluminium melt on the relative strength of fibres with aluminium coating (1); the coefficient of strength variation (2); and change of fibre mass (3).

TABLE I Dependence of fibre fracture type on the duration of the contact with the melt

	Contact time, τ_k (sec)					
	Initial	0.004	0.05	0.1	0.5	5
Number of fractures due to surface defects, P_d (%)	15	10	8	16	38	94
Number of fractures due to inner defects, V_d (%)	85	90	92	84	62	6

TABLE II Phases revealed on the surface of boron fibre by electron diffraction

Fibre	Coating	Reagent–solvent	Phases
B	–	–	B_2O_3
B	Al	KOH	AlB_{12}
B	Al	HCl	AlB_2, AlB_{12}
B	Al	NaOH	AlB_2, AlB_{12}

first by grain-boundary diffusion, provide conditions for initiation and further increase of borides. According to thermodynamic calculations in the Al–B system the formation of AlB_2 and AlB_{12} phases or transformation of AlB_{12} into AlB_2 is possible. Development of boride formation reaction creates an additional stress concentration on the fibre surface because of the negative volume effect that results in a low fibre strength. Boride formation leads to a retardation of the solution reaction.

Table II shows the results of electron diffraction analysis of phases on the fibre surface after solution of the aluminium coating in various reagents. As seen from the table, aqueous solutions of NaOH and HCl leave all the boride phases on the fibre surface. AlB_2 is dissolved in KOH. The solid solution of boron in aluminium was not observed by the electron diffraction method. This phase is obviously dissolved on removal of the coating, but we managed to find it by X-ray diffraction of the fibre with a coating. No oxygen-containing phases on the surface of a fibre with an aluminium coating were detected. Oxygen is removed during fibre pulling through the melt. Both oxides and oxygen absorbed by the surface are removed. Contact with liquid aluminium caused the fibre to heat up to the melt temperature which is essentially higher than the melting temperature of the boride oxide B_2O_3 (450° C [12]). As a result, the aluminium oxide formed is released to the surface at every fibre movement and free boron diffuses into the melt. The fibre heating period up to the melt temperature does not exceed 10^{-3} sec, i.e. the process of oxygen removal from the surface is completed essentially in a shorter period than the contact time with the melt [13]. That is why the fibre surface free of oxides takes part in interaction with the melt.

Boron grain boundaries are primary places of boron phase initiation. On increasing the fibre contact time with the melt and raising the melt temperature, one observes phase spreading along the boron grain boundary. A reduction of the fibre diameter is observed at some points after solution of the aluminium coating in KOH solution. It is connected with intensive germination of phases into the fibre depth and their subsequent solution (Fig. 3d).

To evaluate the contribution of boride phases to the strength reduction of the fibres we used the method of selective etching of fibres which had an aluminium coating. As already mentioned, KOH reagent dissolves the AlB_{12} phase, NaOH reagent preserves both boride phases. The boron fibres which were in contact with the melt for 5 sec were treated in these solutions and then bent and tensile tested. The strength changes in comparison with the initial state is given in Table III. It is seen that removal of AlB_2 boride from the interfacial surface leads to a reduction of the fibre properties. Fractographic analysis of the fibres after mechanical tests shows that the part played by fibre fracture due to surface defects is reduced after dissolution of AlB_2.

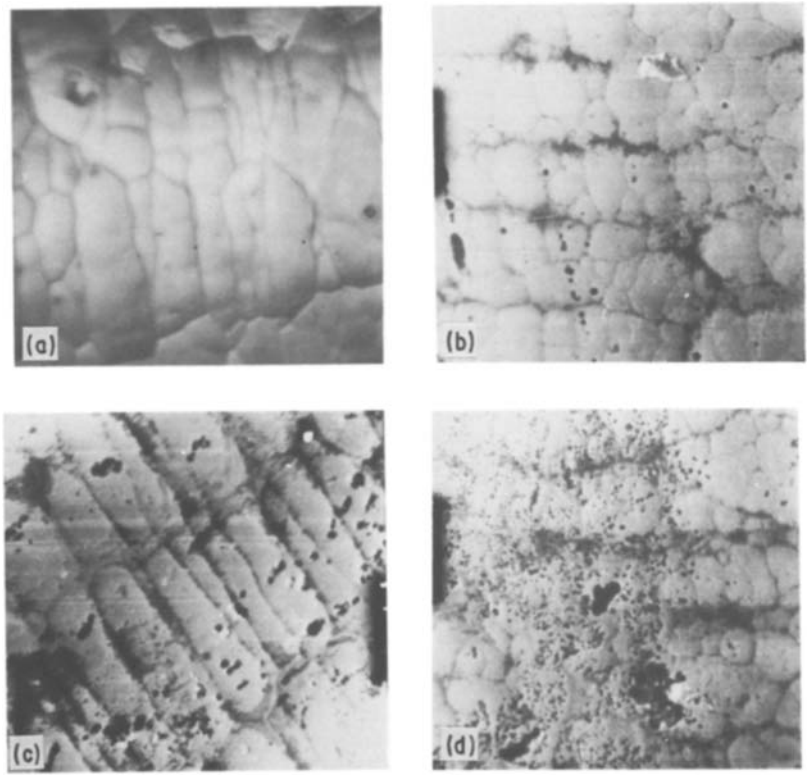

Figure 3 The surface of boron fibres, × 1000: (a) the initial fibre; (b, c, d) after aluminium coating and coating dissolution in KOH.

TABLE III Results of mechanical tests of fibres coated with aluminium

	Fibre condition			
	Initial	After metallization	After etching	
			KOH	NaOH
Relative strength:				
bend tests	1.00	0.83	0.98	0.87
tensile tests	1.00	0.74	0.97	0.79

4. Conclusion

Using the methods of selective etching and weighing it has been found that chemical interaction of boron fibres with the aluminium melt develops into two stages: boron solution in liquid aluminium, and formation of chemical compounds at the interfacial boundary.

Interaction products of boron fibres with the aluminium melt are revealed: there are AlB_2, AlB_{12} phases on the interfacial boundary and boron solid solution in aluminium.

The primary places of chemical interaction development are inter-grain grooves formed by adjoining boron grains emerging to the fibre surface.

Mechanical tests and fractographic analysis have shown that if the fibre material is dissolved in the melt, the fibre strength is raised due to a lesser influence of surface defects; the appearance of aluminium borides on the interfacial boundary is followed by a decrease in the fibre's initial strength.

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