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Interface study of short mullite fiber reinforced Al-4.5Cu alloy composites

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The interest in discontinuously reinforced metal matrix composites has increased extensively in recent years. It is possible nowadays to produce many new cheaper aluminum-based composites such as aluminum borate ($Al_{18}B_4O_{44}$) whisker reinforced ones and aluminosilicate ($Al_2O_3 \cdot SiO_2$) short fiber reinforced ones at even lower cost [1–8]. Among various aluminosilicate fibers, short mullite fibers ($3Al_2O_3 \cdot 2SiO_2$) show outstanding properties, which may make this composite a potential candidate for commercial applications in the near future [6–8, 14]. However, many investigators have indicated that there are severe interfacial reactions between the fibers and the matrix in aluminosilicate and/or crystallized aluminosilicate short fiber reinforced aluminum-based composites [9–11], resulting in the formation of $MgAl_2O_4$ spinel which tends to be detrimental with the attendant loss of fiber strength and leads to decreases in the properties of the composites [12, 13]. There are also researchers who indicate that $MgAl_2O_4$ spinel was not formed at the interface of mullite short fiber reinforced Al-Si alloy (containing Mg) composite [14]. The question is are there any interfacial reactions in short mullite fiber reinforced aluminum-based composites? The answer is YES based upon our recent research work. The results of the present paper are quite different from those of Cao *et al*. [14] and worth further investigation.

The aluminum alloy matrix used in the present work was an Al-4.5Cu binary alloy, having a chemical composition (in wt%): Cu: 4.45, Fe: 0.23, Mn: 0.04, Zn: 0.01 and the balance aluminum.

Mullite fibers, with a chemical composition (in wt%) $72Al_2O_3$ and $28SiO_2$, were selected as the reinforcement. Fig. 1 is the X-ray diffraction (Cu K_α, $\lambda = 0.1542$ nm) pattern of the mullite fibers, which is the same as that of [14]. The mullite fibers were chopped and then made into fiber preform as in [15]. The composites were fabricated by squeeze casting with a melt temperature of 1073 K, preform temperature of 723 K, die temperature of 573 K and infiltration pressure of 60 MPa with 2 min of holding during infiltration. The volume fraction of the reinforcing short fibers was about 18%.

The solution treatment involved heating the composites at 788 K for 10 h and then quenching them into ice water. Artificial aging was carried out at 423 K for 54 h.

Specimens for TEM observation were prepared by standard methods involving mechanical grinding, polishing and dimpling followed by ion milling of foils to perforation on a liquid nitrogen-cooled specimen stage to eliminate further aging during the thinning period. Microstructural studies were performed either in a Philips CM12 TEM operating at an accelerating voltage of 100 kV, or in a JEM-200CX TEM at 160 kV, or in a Philips TECNAI 20 at 200 kV.

Fig. 2a is a TEM micrograph of polycrystalline mullite crystals and Fig. 2b shows the $[00\bar{6}]$ electron diffraction pattern from one of the mullite polycrystalline particles. It is clearly shown that the mullite fibers in the composite are polycrystalline made up of $3Al_2O_3 \cdot 2SiO_2$ crystals, which agrees well with the XRD results. Fig. 3 is the HRTEM image of one mullite crystal particle along [100] and, from the micrograph, it was found that the facial distance between (100) planes is about 0.748 nm, which is almost the same as $a = 0.749$ nm [16]. Brandes [16] tells us that

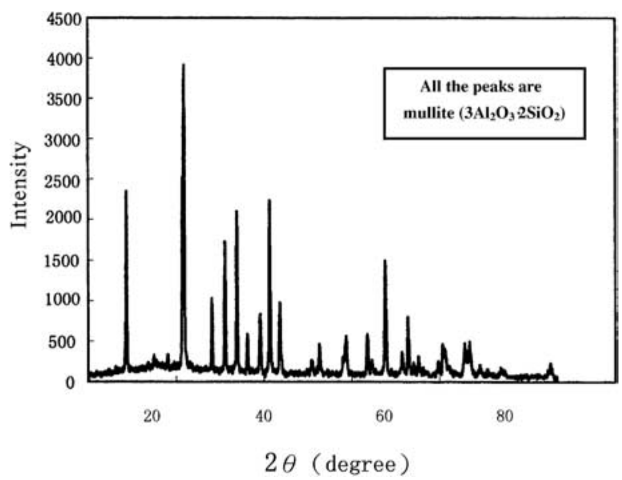

Figure 1 X-ray diffraction (XRD) pattern of the mullite fibers.

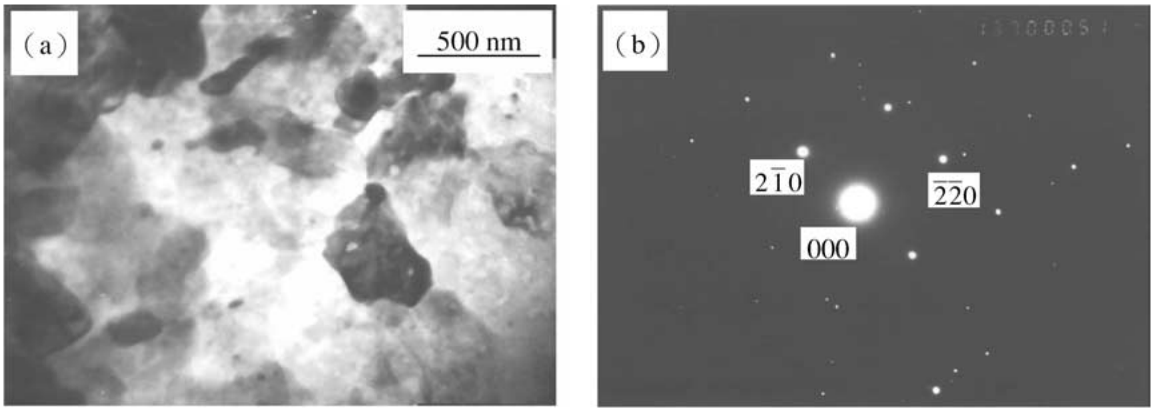

Figure 2 TEM images of polycrystalline mullite (a) and electron diffraction pattern of one crystal mullite particle (b).

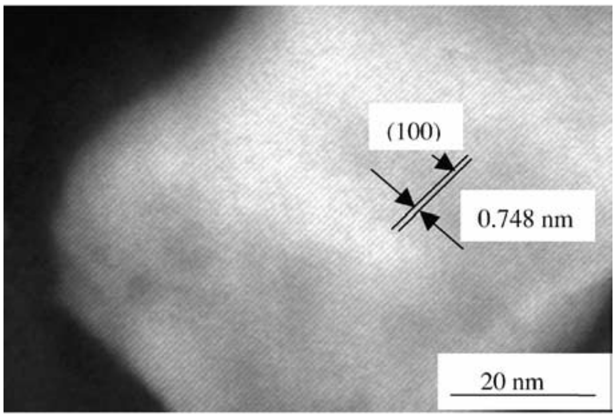

Figure 3 HRTEM image (DF) of one crystal mullite particle along [100].

mullite is orthorhombic in structure with lattice constants: $a = 0.749$ nm, $b = 0.927$ nm and $c = 0.581$ nm. The result of the present study is different from that of [14] in which mullite is taken as tetragonal in structure with lattice constants: $a = 0.755$ nm, $b = 0.769$ nm and $c = 0.288$ nm. Fig. 4a is a TEM micrograph of a typical interface in $(3Al_2O_3{\cdot}2SiO_2)$/Al-4.5Cu composites. It can be seen from Fig. 4a that there exist many aged θ' (Al_2Cu) precipitates in the Al-4.5Cu matrix and also a few interfacial reaction products at the mullite fiber/matrix interface. Fig. 4b is the electron diffraction pattern of one of the reaction products, which has a cubic type structure with a lattice constant $a = 0.808$ nm. This product can be identified as $CuAl_2O_4$ [16].

In [14], based on the phase diagram of the Al-Si binary system and the TEM micrograph of the mullite fiber/Al-Si matrix interface, the authors deduced that the Mg_2Si particle at the interface is an eutectic phase, not a reaction product between the mullite fiber and the matrix alloy. They explained further that the main difference between the aluminosilicate fibers and the mullite fibers is the lower silica (SiO_2) content (28 wt%) in mullite fibers and that the interaction time (20 s for solidification) between the silica in mullite fibers and magnesium in matrix alloy is limited. So, there could not be sufficient silica and time for spinel ($MgAl_2O_4$) formation.

Unlike the case of [14], there is a reaction product $CuAl_2O_4$ formed at the interface of mullite fiber reinforced Al-4.5Cu alloy composites. The phase diagram of the Al-Cu binary alloy system shows that Al_2Cu is a eutectic phase. In as-cast conditions, it is easy to observe the non-equilibrium eutectic Al_2Cu phase. After solution treatment of heating at 788 K for 10 h, followed by ice water quenching, the Al_2Cu phase might re-melt into the matrix, resulting in much fewer precipitates of Al_2Cu at the fiber/matrix interface. It is possible that some eutectic Al_2Cu phase may still exist at the fiber/matrix interface. Even in this situation, it is not difficult to identify the Al_2Cu phase from the $CuAl_2O_4$ spinel.

From the above observations and discussion, we can conclude that chopped mullite fibers are polycrystalline in structure composed of many fine mullite

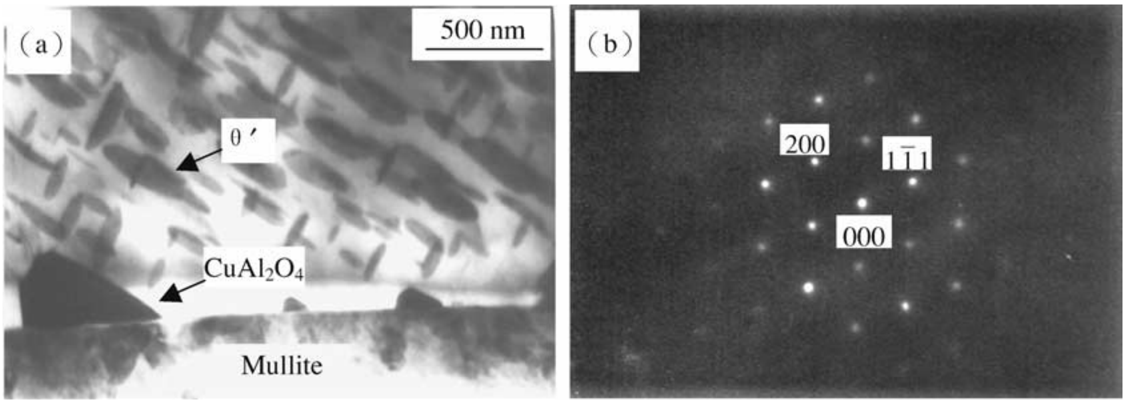

Figure 4 TEM image of a typical $3Al_2O_3{\cdot}2SiO_2$/Al-4.5Cu interface (a) and electron diffraction pattern of one of the interface reaction products (b).

crystal particles. Mullite is orthorhombic with lattice constants of $a = 0.749$ nm, $b = 0.927$ nm and $c = 0.581$ nm based on electron diffraction analysis and HRTEM observation of crystal mullite. After solution and aging treatment, there appeared a reaction product $CuAl_2O_4$ formed at the fiber/matrix interface in $(3Al_2O_3{\cdot}2SiO_2)$/Al-4.5Cu composites.

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