General paper

INTERFACE STRUCTURE AND MECHANICAL PROPERTIES OF POWDER METALLURGICAL 6061 ALUMINUM MATRIX COMPOSITES REINFORCED WITH VARIOUS KINDS OF OXIDE WHISKERS

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Abstract: The compatibility of oxide whiskers with 6061Al alloy and the age hardening behavior of composites prepared from powder metallurgy were investigated. Interface reactants containing Mg were shown to be formed during hot pressing and hot extruding for every composite. The composites did not reveal age hardening behavior due to the depletion of Mg in the matrix at all. To keep whiskers from the interfacial reaction, electroless nickel plating onto whiskers was carried out. Although the composite with Ni plated whiskers revealed the age hardening, its hot workability was too poor to apply practical fabrication because of the formation of brittle intermetallic compound Al3Ni in the matrix.

Keywords: 6061Al matrix composites, Oxide whiskers, Interface reaction, Powder metallurgy, Electroless nickel plating, Age hardening

1 INTRODUCTION

Much cheaper oxide whiskers have recently been developed and are waiting to be applied for advanced engineering materials[1]. They are aluminum borate, titanium oxide and titanium potassium oxide whiskers. Some of them have been studied to be used as fillers which are reinforcements for metal matrix composites[2-5]. The results are suggesting a wonderful possibility to be available as fillers in aluminum matrix composites. At the same time, it is pointed out that the interfacial reaction occurs during producing such composites and it might degrade their mechanical properties if the interfacial reaction is heavy[6,7]. Metal matrix composites can be produced by either squeeze casting or powder metallurgy techniques. Many studies, as mentioned above, have been almost devoted to squeeze casting technique. In such a case, the interfacial reaction between aluminum or aluminum alloy and oxide whisker occurs while molten metal infiltrates into the fibrous preforms because of their high pouring temperature of about 1073K and of their high ability to reduce oxide whiskers. We considered that aluminum alloy matrix composites reinforced with oxide whiskers might reveal superior mechanical properties by using powder metallurgical technique, because the interfacial reaction may be suppressed moderately due to much lower processing temperature than that of squeeze casting.

In the present work, the reactivity of various oxide whiskers with 6061 Al alloy and the age hardening behavior of their composites produced by powder metallurgy were investigated. In addition, the effect of electroless nickel coating onto whiskers on the interfacial reactivity and the age hardening behavior were also studied.

2 EXPERIMENTAL

Mean powder diameter of 6061 Al alloy used in this study was 20 μm. Reinforcements are titanium oxide (hereafter designated as TiOx), aluminum borate (likewise, as AlB) and titanium potassium oxide (KT) whiskers. 6061 Al alloy powder and whisker weighted out for composites to have a volume fraction of 10% were mixed, dried and consolidated by vacuum hot pressing and/or extruding. Hot pressing temperature was 873K and holding time was 3.6ks. Further details of the procedure and the physical properties of these whiskers have been shown in our previous paper[8]. Contrary to our expectation, the interfacial reaction has occurred in every composite. To prevent whiskers from contacting aluminum alloy, AlB whiskers were coated with nickel containing 0.3 mass% boron by using electroless plating technique. Electroless nickel plating solution was the Bel-Nickel solution provided by Uyemura Industries Ltd. The AlB whiskers were pre-treated with a stannate treatment at 303K before the nickel plating process. The pre-treatment consisted of immersing the whiskers in the cleaning solution, the acid solution, the stannate solution and the displacement nickel plating solution. The whiskers were rinsed with distilled water after immersion in each solution. The as-hot pressed composites were solutionized at 803K for 7.2ks and then quenched to room temperature. Thereafter, they were aged at 448K for various

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periods. Vickers hardness tests were performed to investigate age hardening behavior (49N load for 15s). Tensile test was also done at room temperature.

An analytical transmission electron microscope was used to study whisker-matrix interfacial reaction. Fracture surfaces were examined by a scanning electron microscope. Microstructures were confirmed by an X-ray diffraction equipment with an accelerating voltage of 40kV and a beam current of 200mA.

3 RESULTS AND DISCUSSIONS

3.1 Macrostructure

Figure 1(a) and (b) respectively show examples of macrostructure of hot pressed and extruded composite reinforced with AIB whisker. An aggregation of whiskers at parent aluminum grain boundaries was seen in every hot pressed composites as in Fig.1(a). The extent of the aggregation was dependent on the whiskers size; the smaller the whisker is, the more it has a tendency to aggregate at parent aluminum alloy powder boundaries. Figure 1(b) indicates that whiskers are nearly aligned in extrusion direction in extruded composites. However, angular deviation of whiskers from the extrusion direction seems to get larger than that of pure aluminum matrix composites. In addition, whisker length has been shortened[9]. The mean AIB whisker length was about 3.2 μm which is one third of pure aluminum matrix composite. These results suggest that the extrusion tends to damage whiskers in 6061 Al alloys and/or whisker matrix interfacial reaction may occur.

3.2 Microstructure

The whiskers were extracted from the hot pressed composites by immersing them in a solution containing 10% NaOH in order to study the reactivity of various whiskers with 6061 Al matrix. Figure 2 represents the surface topographs of AIB whisker(a), TiO2 whisker(b) and KT whisker(c). The surfaces of TiO2 whiskers look smooth, but those of AIB whiskers reveal a scaled pattern indicating the occurrence of heavy interfacial reaction. In the case of
KT whisker, the reactivity with 6061 Al is not so large as in the case of AIB whisker. Then, an analytical transmission electron microscopic experiment was carried out to investigate closely the manner of the interfacial reactivity in each composite. Figure 3 shows TEM microstructure(a) and an energy dispersive X ray spectroscopy microanalysis (b) of hot pressed AIB/6061 composite. Interfacial reactants can be seen at whisker-matrix interface and the thickness of reactant reaches about 150nm. It is clear that this layer contains Mg, Al and O elements and is constituted by a lot of ultrafine grains. Figure 4 indicates the X ray diffraction pattern showing weak peaks of the spinel MgAl2O4 phase. These results indicates that Mg dissolved in matrix can diffuse easily toward interface and reacts there with AIB whiskers during hot pressing. It follows that the MgAl2O4 phase was created at the interface and Mg in the matrix was consequently depleted. The reason for the formation of MgAl2O4 phase at the interface could be attributed to the fact that both Al and Mg easily combine with oxygen, owing to much lower free energy change ΔG of their oxides than that of boron oxide[10]. Figure 5 represents microstructure (a) and EDS microanalysis (b) of TiO2/6061 composite as hot pressed. Likewise, whisker-matrix interaction can be seen. The thickness of reactant was about 60nm and it was thinner than that in AIB/6061 composite. Also, Al, Ti, Mg and O elements were detected in the reactant layer by means of EDS analyzer. In addition, we can see from Fig.5 (a) that there are many ultrafine grains in the interface layer. Figure 6 represents the X ray diffraction pattern for hot pressed TiO2/6061 composite. It is clear that the interface layer was constructed by Al2O3, MgTiO3 and Al3Ti crystals. From these results, it is likely that Mg and Al can readily diffuse toward whiskers and Ti can diffuse inversely toward the matrix. Just as before, Mg in the matrix must be consumed too.

Finally, Fig.7 indicates TEM microstructure(a) and EDS microanalyses (b) of KT/6061 composite. Whisker matrix interaction can be also seen and the size of crystals of the reactant is similarly very fine. Figure 7(b) is EDS microanalysis from the reactant. Al, Mg and O elements were contained and the reactant can be identified as MgAl2O4. Though the structure of KT looks like TiO2, Ti was not detected in the interface layer. On the other hand, Al2Ti, Al3O5 and MgTiO3 phases were detected by X ray measurement as shown in Fig.8. There is an obvious

Fig.3. Transmission electron micrograph(a) and energy dispersive X ray spectroscopy microanalysis(b) at the interface reactant of hot pressed Al borate whisker reinforced 6061 Al matrix composite.

Fig.4. X ray diffraction pattern for hot pressed Al borate whisker reinforced 6061 Al matrix composite.

Fig.5. Transmission electron micrograph(a) and EDS microanalysis(b) at the interface reactant of hot pressed TiO2 whisker reinforced 6061 Al matrix composite.

Fig.6. X ray diffraction pattern for hot pressed TiO2/6061 comosite.

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discrepancy between these results. This may be due to the uncertainty in identification of substances by X-ray measurement because X-ray diffraction pattern is very complicated. At the present time, we cannot explain exactly the interfacial reaction for KT/6061 composite. Further investigation is required to give a full account of the phenomenon. However, it is clear that Mg is depleted in the matrix of KT/6061 composite.

3.3 Age Hardening Behavior and Tensile Property

Whisker–matrix interaction occurred in every composite and it is found that the diffusion of magnesium always played an important role in this phenomena. The most serious problem is that magnesium is depleted in the matrix. 6061 Al alloy is well known as an age hardenable alloy. Age hardening of 6061 Al alloy is due to fine precipitation of Mg2Si phase. From microstructural experiments, these matrices may be presumed to have weak ability of age hardening. Figure 9 shows age hardening curves for monolithic 6061 Al alloy and 10vol% oxide whiskers reinforced 6061 Al alloy matrix composites. The data of SiC whisker reinforced composite is presented in the same figure for reference. All composites excepting SiC/6061 composite did not harden at all. Figure 10 represents an example of tensile properties of extruded Al borate whisker reinforced 6061 Al matrix composites. It is clear that tensile strength and ductility of composite are inferior to that of monolithic 6061 alloy and is the same as that of pure aluminum matrix composite. Figure 11 shows an optical micrograph near fracture surface of 10vol% AlB/6061 composite. Whiskers were broken, reflecting the enhanced cohesive force due to the existence of interface layer. These results as mentioned above are beyond our expectation, because we expected that powder metallurgical composites reinforced with oxide whiskers should exhibit better mechanical properties than the composites fabricated by squeeze casting due to the suppression of interfacial reaction for powder metallurgical composites which are produced at lower temperature. These facts suggest that the interfacial reaction can progress remarkably by long period of heating for hot pressing even at medium processing.
3.4 Effect of Electroless Nickel Plating onto Al Borate Whiskers on Microstructure and Age Hardening Characteristic of the Composite

Electroless nickel plating layer is about 0.2 μm in thickness. Figure 12(a) shows the microstructure of as hot pressed composite and Fig.12(b) indicates TEM microstructure. Whisker-matrix interaction and segregation of Mg into interface were not observed within the accuracy of the present work. Thus, Ni plating onto whiskers suppresses the interfacial reaction. However, it can be seen that the matrix is mottled as shown in Fig.12(a). Figure 13 indicates X ray diffraction pattern indicating the formation of Al3Ni phase. This occurrence proves that the diffusion rate of nickel toward matrix is relatively high at hot pressing temperature. Intermetallic compound is intrinsically brittle in general, and it is expected that Al3Ni has a demoralizing influence upon hot workability. Figure 14 shows age hardening behavior of hot pressed composite reinforced with Ni coated AlB whiskers. Age hardening can be seen slightly. The hardness is higher than that of SiC/6061 composite because of the existence of Al3Ni phase in the matrix. Figure 15 indicates microstructures of extruded composite with nickel coated AlB whiskers. Whiskers are damaged remarkably and the degree of their ordering to the extrusion direction becomes very low. Furthermore, large voids are formed and they reach to 20 μm in length. In addition, the MgAl2O4 phase is formed after extruding. These results are attributed to the existence of brittle Al3Ni phase and the occurrence of whisker matrix interfacial reaction due to the exposed fresh surfaces of whiskers caused by snaps of whiskers during hot extrusion. The tensile strength is less than 130MPa and intergranular brittle fracture occurred, reflecting the complex structure, as mentioned above.
Fig. 15. Microstructures of hot extruded 6061 Al matrix composite with electroless nickel plated Al borate whiskers. (a) reveals the morphology of whiskers and (b) shows a lot of microvoids.

4 CONCLUSION

In this paper, the interfacial reactions between various oxide whiskers and 6061 Al alloy during fabrication of powder metallurgical composites and age hardening behavior have been investigated. The results obtained are summarized as follows.

For all combinations of the present whiskers and 6061 Al, oxide reactants containing magnesium were formed at whisker-matrix interface during hot pressing. Age hardening ability disappeared due to the depletion of magnesium atoms in the matrix and the strength of the extruded composite reinforced with aluminum borate whisker was inferior to that of monolithic 6061 alloy.

The diffusion of magnesium toward oxide whisker could be controlled to some extent by electroless nickel plating onto whiskers. In such a case, age hardening appeared. However, the composite was embrittled due to the formation of intermetallic compound Al3Ni in the matrix.

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