Formation and Microstructure of an In Situ Aluminum Composite by Oxygen Spray Technique

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In situ particulate-reinforced Al and Al-Mg matrix composites were successfully fabricated by an oxygen spray technique. The results indicate that Al_2O_3 and $MgAl_2O_4$ particles directly nucleate and grow via gas-liquid oxidation reaction. The Al_2O_3 particles with the size of 0.1 to 0.5 μ m are formed into the Al melt, according to an appropriate system and processing parameters. The reinforcements show a good wettability with the matrix. The hot-rolling process results in an improvement of the uniformity of the particle distribution in the matrix and an increase in the hardness of the composites.

Keywords Al composite, Al-Mg alloys, in situ processing, metal matrix composite, microstructure

1. Introduction

In situ metal matrix composites are normally fabricated by solid-liquid (S-L) and gas-liquid (G-L) reaction techniques (Ref 1-4). The ceramic reinforcing phases directly nucleate and grow in the liquid metal matrix at high temperature. Therefore, the formation process offers a fine and thermodynamically stable reinforcing ceramic phase, which wets the matrix phase (Ref 5-6).

To achieve fine reinforcements, an appropriate composite system was selected, that is, the reinforcements in the liquid matrix were able to nucleate rapidly and grow slowly. In this study, a formation of in situ particles was fabricated by injecting oxygen into an aluminum and an aluminum-magnesium alloy melt. This process was based on the following oxidation reactions:

 $\begin{array}{l} \mbox{Al (liquid)} + \mbox{O}_2 \, (gas) \ensuremath{\rightarrow}\ \mbox{Al}_2 \mbox{O}_3 \, (solid) + \mbox{Al (liquid)} \\ \mbox{Al-Mg (liquid)} + \mbox{O}_2 \, (gas) \ensuremath{\rightarrow}\ \mbox{Al}_2 \mbox{O}_3 \, (solid) + \mbox{MgAl}_2 \mbox{O}_4 \\ \mbox{(solid)} + \mbox{Al (liquid)} \end{array}$

The reaction products, Al_2O_3 and $MgAl_2O_4$ as reinforcements, were dispersed in the aluminum and aluminum-magnesium alloy matrix and formed the particulate-reinforced composites.

2. Experimental Procedures

Pure aluminum and Al-1wt%Mg alloy were used as matrix materials. They were heated by an induction furnace and held in the temperature range of 750 to 820 °C for 20 to 120 min in an air atmosphere. Oxygen was injected through a four-jet sprayer (4 mm in diameter each) into the aluminum and its

alloy melt, while a vortex was made by stirring through vanes at a speed of 500 rpm. Oxygen flow was controlled from 0.08 to $0.16 \text{ m}^3/\text{h}$, depending on the viscosity of the molten metals.

First, thin Al_2O_3 films were formed, surrounding oxygen bubbles in the molten aluminum and aluminum-magnesium alloy, which were stable and prevented growth. According to the authors' observations with an atomic force microscope (AFM), as shown in Fig.1, the grain size of Al_2O_3 particles of the films near oxygen bubbles was typically 0.1 to 0.4 µm after pouring oxygen with 20 min at 700 °C. Thin Al_2O_3 polycrystalline films were crushed under the action of shearing stress resulting from stirring through the vanes and being dispersed in the molten matrix. In situ Al_2O_3 particulate-reinforced aluminum matrix composites were formed after solidification. Some samples were then hot rolled.

The microstructures of G-L in situ composite systems were identified by a combination of x-ray diffraction (XRD), scanning electron microscopy (SEM), transmission electron microscopy (TEM), and AFM. The hardness and wear resistance of composites were measured.



Fig. 1 Atomic force microscope image showing submicrosized particles of the Al_2O_3 film

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3. Results and Discussion

3.1 The Relationship between Fabricating, Processing, and Microstructures

The in situ reaction products were identified by both XRD and electron diffraction analysis. This showed that the particles produced consisted of α -Al₂O₃ and κ -Al₂O₃ phases when the aluminum system was used as the matrix, whereas the reaction phases produced consisted of α -Al₂O₃, κ -Al₂O₃, and MgAl₂O₄ when the aluminum-magnesium alloy system was used as the matrix. More ceramic reinforcements, up to 30 vol%, were observed in the aluminum-magnesium system than in the aluminum system under the same experimental conditions, as shown in Fig. 2.

The size, shape, and distribution of the reinforcing phases were strongly affected by the processing parameters. Transmission electron microscopy at a higher magnification (Fig. 3) showed that the fine Al_2O_3 particles (0.1 to 0.5 µm), typically hemispherical, were formed after oxygen spray into the aluminum melt at 750 °C for 20 min. Imaging was performed in a Hitachi, LTD (Tokyo, Japan) Hitachi H-700H TEM operating at 200 kV. It was also found that the size of in situ formed Al_2O_3 particles in the composites increased with increasing spraying time. Figure 4 shows that the particles grew up to 3 µm in diameter after 750 °C for 60 min. Most of the fine Al_2O_3 particles were dispersed along the grain boundaries (Fig. 4). Figure 5 shows the blocky MgAl₂O₄ phase with particles 5 to 8 µm.

A hot-rolling process was used to improve the hardness and distribution of particles. The hardness values (HRB) increased after the hot rolling, as shown in Fig. 6. Most of the Al_2O_3 particles dispersed along the grain boundaries before hot rolling, whereas they were refined and dispersed homogeneously after hot rolling.

3.2 Wettability between Particles and Matrix

A good wetting interface between the particles and the matrix was obtained. No bubble or second phase visibly surrounded particle borders or appeared in aluminum grains, as shown in Fig. 3 to 5, respectively. The reinforcing phases directly nucleated and grew via G-L in situ oxidation reaction processing, which can involve the formation of a thermodynamically stable refractory compound in the metal matrix.



Fig. 2 Influence of matrix and spray time on volume fracture (vol%) of particles



Fig. 3 Transmission electron microscopy image of fine Al_2O_3 particles in the grains. $21,000 \times$



Fig. 4 Transmission electron microscopy image showing Al₂O₃ particles along boundaries. 27,000×



Fig. 5 Transmission electron microscopy image showing MgAl₂O₄ phase in the aluminum-magnesium matrix. 15,000×



Fig. 6 Hardness before and after hot rolling

3.3 Measurement of the Wear Resistance

The wear tests of composites were performed with a M-200 wear testing machine (Xuanhua Material Testing Machine Factory, Hebei, China) under three loadings for 20 min each. The wear widths of the samples as wear values were examined. From Fig. 7, it is clear that both aluminum and aluminum-magnesium matrix composites show the smaller wear values, as compared to pure aluminum materials. The differences of wear values between composites and pure aluminum materials increased with increasing the loads. This means that the composites may be suited for use in higher loading conditions, which have potential engineering applications. Differences of wear values between aluminum and aluminum-magnesium matrix composites may result from the differences of the shapes and hardness of the Al₂O₃ and MgAl₂O₄ reinforcing phases.

4. Conclusions

In situ particulate-reinforced aluminum and aluminummagnesium matrix composites were successfully fabricated. The submicrosized and microsized Al₂O₃ and MgAl₂O₄ phases were directly formed via the oxygen spray technique, with the resulting microstructure dependent on the system and on processing parameters. This novel process provides for conventionally fabricated and cost effective composites. The in situ Al₂O₃ and MgAl₂O₄ reinforcements show a good wettability with the aluminum and aluminum-magnesium alloy matrix. The hotrolling process results in an improvement of the uniformity of



Fig. 7 Comparison of the wear resistance

the reinforcement distribution in the metal matrix and an increase in the hardness of the composites.

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