Recrystallization behavior of in-situ synthesized MgAl$_2$O$_4$ whiskers reinforced 6061 aluminum alloy composites

Geng Hao, Chunsheng Shi, Naiqin Zhao
Tianjin Key Laboratory of Composite and Functional Materials, School of Materials Science and Engineering, Tianjin University, Tianjin 300350, PR China

Aluminum matrix composites (AMCs) have been widely used in automotive, transportation, and aerospace industry due to their extraordinary properties such as high specific strength, high specific modulus, low density and high wear resistance. Generally, particles, fibers and whiskers are considered to be common discontinuous reinforcement of AMCs. Ceramic whiskers draw much attention recently because of their excellent properties, among which common used ceramic whiskers are Al$_{18}$B$_4$O$_{33}$, SiC, MgO, etc. In our previous work [1-3], MgAl$_2$O$_4$ spinel whiskers were in-situ synthesized in aluminum matrix. The MgAl$_2$O$_4$/Al composites showed excellent mechanical properties, however, there were still some problems that need further investigation, such as the recrystallization behavior of the composites. Coarse equiaxed grains appear during recrystallization annealing, resulting in mechanical property decreasing [4]. In this work, investigation of the effect of recrystallization annealing on the microstructure and mechanical properties of the MgAl$_2$O$_4$/6061 Al composites was carried out.

In our work, in-situ MgAl$_2$O$_4$ whiskers reinforced 6061 aluminum alloy composites were fabricated by a powder metallurgy method. To obtain the prefabricated powder mixture, 6061 aluminum powder with diameter ranging from 10 $\mu$m to 45$\mu$m, Mg powder with an average dimension of 10 $\mu$m and boric acid were mixed and high-energy ball milled for 4 hours at 400 rpm under argon atmosphere.
The powder mixture was compacted into a block and then sintered at 650°C for 1 hour under argon flow. MgAl₂O₄ whiskers were in-situ synthesized in 6061 Al matrix after sintering. The composites were hot-extruded to rod-like samples and then recrystallization annealed at various temperatures.

The obtained composite was examined by XRD to identify phases of the obtained composite. Fig. 1 shows XRD pattern of the composite. The peaks at 36°, 44° and 65° can be assigned to Mg₁₇Al₂O₄ spinel, proving the Mg₁₇Al₂O₄ spinel whiskers were in-situ synthesized successfully during the sintering process. Al was also recognized by XRD pattern (peaks at 38°, 44°, and 78°), and its peaks were much stronger than that of Mg₁₇Al₂O₄ spinel because of the high volume fraction of matrix in the obtained composites.

![XRD pattern of obtained composite.](image)

SEM and TEM were used to observe the microstructure of MgAl₂O₄ whiskers. Fig. 2 shows SEM image of MgAl₂O₄ whiskers. The diameter of MgAl₂O₄ whiskers ranges from 30 nm to 80 nm with the aspect ratio ranging from 10 to 20. MgAl₂O₄ whiskers uniformly distribute in the matrix, and most of them lie along extrusion direction. Fig. 3 shows TEM image of MgAl₂O₄ whiskers. A clean and smooth interface of the whisker and matrix can been seen (Fig. 3b), reflecting a well
combination of MgAl$_2$O$_4$ whiskers and 6061 aluminum alloy matrix, thus a good load transmitting can be expected. And the tensile test results confirmed the enhancement of the mechanical properties. The fracture strength of the MgAl$_2$O$_4$/6061 Al composites reached to 277 MPa, while fracture strength of 6061 aluminum alloys was 235 MPa.

![Fig. 2. SEM image of MgAl$_2$O$_4$ whiskers.](image)

![Fig. 3. TEM image of MgAl$_2$O$_4$ whiskers: (a) MgAl$_2$O$_4$ whiskers, (b) interface of whisker and matrix.](image)

Hot-extrusion is an effective method to improve the strength of composites. Generally, most grains of hot-extruded samples are elongated along the extrude direction (ED) and some grains may show equiaxed because of the dynamic recrystallization. These deformed grains are sensitive to the temperature. When the obtained samples are held at high temperature, the deformed grains tend to
recrystallized, resulting in equiaxed coarse grains. Secondary phase significantly influence the recrystallization, either promote or inhibit recrystallization, depending on the size of secondary phase [5]. In this work, the obtained MgAl2O4 whiskers reinforced 6061 Al alloys composites were annealed at 530 °C, 580 °C, 600 °C and 630 °C for 1h respectively to investigate the evolution of the microstructure of the composites.

The microstructures of as-extruded and annealed specimens are shown in Fig. 4. A very small amount of fine equiaxed grains had nucleated as a result of dynamic recrystallization (Fig. 4a). Although fine equiaxed grains existed, most grains were elongated along ED. When specimens were annealed at 530 °C, 580 °C and 600 °C, volume fraction of equiaxed grains increased and some recrystallized grains started to grow (Fig. 4b, Fig. 4c and Fig. 4d), compared with as-extruded specimen. Moreover, there was no evident difference in recrystallized grains between specimens annealed at 530 °C, 580 °C and 600 °C. The recrystallization was uncompleted in these specimens. And annealed at 630 °C, the fine recrystallized grains grew into coarse equiaxed grains sharply and no fibrous grain can be seen. This indicated that a complete recrystallization had already occurred.

![Fig. 4. Microstructure of MgAl2O4w/6061 Al composites: (a) as-extruded, (b) annealed at 530 °C, (c) annealed at 580 °C, (d) annealed at 600 °C, (e) annealed at 630 °C.](image_url)
Compared with other deformed Al-Mg-Si (6xxx series) alloys [6], of which the recrystallization annealing were usually carried out at 500 °C ~ 600 °C, the obtained MgAl$_2$O$_4$ whiskers reinforced 6061 Al alloy composites showed a significant recrystallization resistance. The recrystallization process was retarded by the MgAl$_2$O$_4$ whiskers (Fig. 5). According to Zener’s pinning theory [7], these whiskers provide a strong Zener drag to prevent the movement of dislocations and the migration of (sub-) grain boundaries, inhibiting the occurrence of recrystallization during annealing.
References:


