

## Short communication

# Fabrication of aluminum matrix composites with enhanced mechanical properties reinforced by in situ generated $\text{MgAl}_2\text{O}_4$ whiskers

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## ABSTRACT

Aluminum matrix composite reinforced by in situ generated single crystalline  $\text{MgAl}_2\text{O}_4$  whiskers was fabricated by chemical synthesis method in an Al–Mg– $\text{H}_3\text{BO}_3$  system. A large number of  $\text{MgAl}_2\text{O}_4$  whiskers were generated during the sintering process and distributed homogeneously in the Al matrix. The whiskers penetrate into the matrix grains to form the framework of the materials, leading to an incredible increase in mechanical properties of the composites. The generation mechanism of the  $\text{MgAl}_2\text{O}_4$  whiskers was also discussed.

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## 1. Introduction

Aluminum matrix composite reinforced by ceramic whiskers or fibers has emerged as an important structural material with high performance for use in the aerospace and automotive industries [1–3]. As the discontinuous reinforcements, one-dimensional (1D) ceramic materials possess high Young's modulus and strength, good chemical inertness and low thermal expansion, resulting in the composites with high strength, high stiffness, excellent wear resistance and high temperature durability [4–9].

In general, metal matrix composites (MMCs) are fabricated by either casting or powder metallurgy [1,2,10–12]. The casting route offers the advantages of good formability and low cost, which is suitable for mass production of MMCs. However, excessive interfacial reactions between the reinforcement and matrix is unavoidable in the molding process, and poor wetting behavior between ceramic phase and metal matrix also limits the enhancement effect of composites. On the other hand, the powder metallurgy is a near-net-shape processing method, but the uneven distribution of the reinforcement and the poor combination between the reinforcement and matrix affect the properties of composites. Therefore, good interface between the reinforcement and matrix cannot be easily obtained by both 'ex situ' methods. In order to solve the issues, in situ fabrication technologies have been developed to fabricate ceramic particles or whiskers reinforced MMCs [13–19]. So far, the in situ generated whiskers has been applied in Ti and Mg matrix

composites [17–19], but in situ growth of whisker reinforcements in aluminum matrix composites has been less reported [20].

$\text{MgAl}_2\text{O}_4$  spinel possesses important properties such as high melting point, high hardness, relatively low density, high strength at room and at elevated temperature, high chemical inertness, and relatively low thermal expansion coefficient [21]. So it is an ideal reinforcement of Al matrix composites. In addition, Schweinfest et al. proved that  $\text{MgAl}_2\text{O}_4\{100\}/\text{Al}\{100\}$  interface has an extreme small misfit (0.25%) between the two lattices [22], indicating a coherent interface might be obtained between the reinforcement and the matrix. In the present study,  $\text{MgAl}_2\text{O}_4$  whiskers were in situ generated inside the Al matrix without obtaining other by-products. The structure and distribution of the whiskers were investigated, and the mechanical properties of the composites were studied.

## 2. Experimental details

Aluminum powder, Mg powder and boric acid were used as starting materials. The sizes of Al and Mg powder particles range between 40 and 60  $\mu\text{m}$ . The right amounts of Mg powder, boric acid, and Al powder were mixed with a mass ratio of 2:2:9. The mixture was high-energy milled for 12 h with the ball-to-powder mass ratio of 10:1 at a rotation speed of 650 rpm. The weight fraction of Mg in the mixed powder after milling pretreatment is about 15.38 wt.%. Then pure Al powder was added into the mixture to control the amount of whiskers formed during sintering. By adding 54.48 wt.% of Al powder in the mixture, the mass fraction of the Mg decreased to 7 wt.%.

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The Al–Mg–H<sub>3</sub>BO<sub>3</sub> powders were mixed evenly and then cold pressed in a steel mold with 20 mm in diameter under a pressure of 600 MPa. Subsequently, the obtained compact was put in a muffle furnace, and then sintered at 800 °C for 1 h in an argon atmosphere during which the MgAl<sub>2</sub>O<sub>4</sub> whiskers generated in the matrix and MgAl<sub>2</sub>O<sub>4</sub> whiskers/Al composite sample was obtained. Then the composite sample were hot-extruded through a steel die with an extrusion ratio of 16:1 at 500 °C, preparing a composite bar with 5 mm in diameter. A 5 wt.% Mg–Al alloy sample was prepared using the same process for comparison.

Phase identification was performed using a Rigaku D/max 2500V/pc automated X-ray diffractometer (XRD) in Cu K $\alpha$  radiation. Microstructure and the selected-area electron diffraction (SAED) of the prepared whiskers were characterized with a high-resolution transmission electron microscope (HRTEM, Philips Tecnai G2 F20). The morphology of the composites was observed in a JEOL S4800 scanning electron microscope (SEM). The hardness of the composite samples was tested using an MH-6 Vickers Micro-hardness Tester under 200 g loading. Tensile and compressive tests of consolidated composites were performed on an M350-20KN universal testing machine under 3 mm per min monotonic loading. Tensile and compressive sample dimensions were 5 mm in diameter.

### 3. Results and discussion

To identify the composition and crystal structure of the obtained whiskers, the aluminum matrix of the bulk sample was eroded by hydrochloric acid, and the gray powder of the whiskers was left. XRD pattern of the extracted whiskers is shown in Fig. 1a. The diffraction peaks in the pattern exhibit a good agreement with the standard spectra of the spinel in both the reflection profile and intensity, and no other peaks related to by-products were detected.

Fig. 1b shows a typical HRTEM image of an individual whisker. The whisker has well-crystalline structure with thin amorphous

phase in the outer layer. In the enlarged TEM image of the whisker, the distance between the neighboring parallel fringes is 0.2439 nm, corresponding to the (3 1 1) planes of MgAl<sub>2</sub>O<sub>4</sub>. Fig. 1c is the SAED pattern of the whisker, and all the diffraction spots can be indexed as (3 1 1), (4 2 2), and (7 3 1) planes of the spinel structure of MgAl<sub>2</sub>O<sub>4</sub>, in accordance with the XRD results. This indicates that the MgAl<sub>2</sub>O<sub>4</sub> whisker is well-developed in a single-crystalline structure of spinel.

SEM image of the whiskers in the fracture surface of the composite is shown in Fig. 2a. The whiskers present a hexagonal shape at the transverse section. The whiskers are 0.2–1  $\mu$ m in diameter and grow as long as dozens of micrometers. Because the whiskers were in situ synthesized in the Al matrix, the aggregation of the whiskers is avoided and the in situ synthesized MgAl<sub>2</sub>O<sub>4</sub> reinforcements distribute uniformly in the matrix.

Fig. 2b is an SEM photo of the whiskers exposed out of the fracture surface after slight corrosion. It is found that the whiskers were covered tightly by the matrix, indicating the well wettability between the matrix and the whiskers. Thus, the matrix can transfer the outer load through the interfaces to the whiskers, and the whiskers can reinforce the matrix under great pressure without being torn from the matrix. In addition, after calcining at 800 °C, the hard whisker is likely to penetrate into the melted Al–Mg alloy matrix, but the bulk composite still keeps its original shape. This may be attributed to the well wettability between the whiskers and the matrix, and both the surface tension of the molten Al–Mg alloy and the whiskers penetrating hindered the molten matrix to flow.

We proposed that the MgAl<sub>2</sub>O<sub>4</sub> whisker generation process is as follows. During the calcination process, the boric acid decomposes into B<sub>2</sub>O<sub>3</sub> and H<sub>2</sub>O at about 200 °C, and B<sub>2</sub>O<sub>3</sub> melts at about 450 °C. The chemical reaction between Mg and B<sub>2</sub>O<sub>3</sub> takes place at temperatures higher than 600 °C. The new formed MgO then dissolves into the B<sub>2</sub>O<sub>3</sub> and reacts with Al to form MgAl<sub>2</sub>O<sub>4</sub> whiskers. The reactions are supposed as [23,24]:

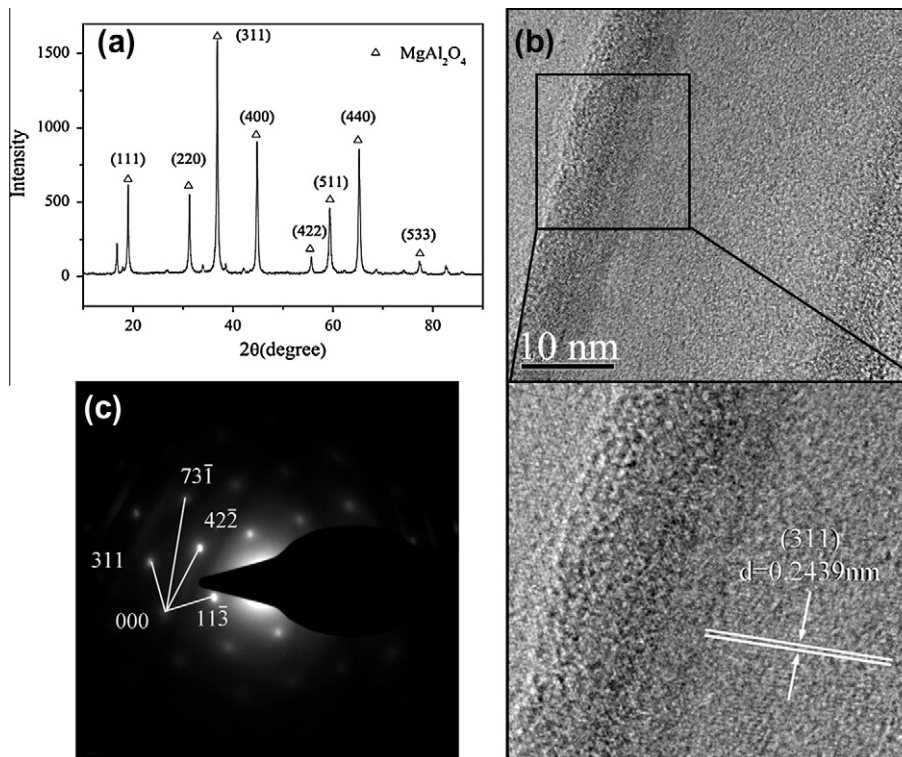


Fig. 1. (a) XRD pattern of the whiskers obtained at 800 °C, (b) HRTEM image of the whisker and the measurement of the interplanar spacing and (c) SAED pattern of the whisker.

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