In-situ Synthesis of Gamma Al_2O_3 Whiskers Reinforced Aluminum Matrix Composite

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Key Words Gamma Al_2O_3 whiskers; in-situ synthesis; aluminum matrix composites; ball milling

1 Introduction

Aluminum metal matrix composites (AMCs) emerged from the perpetual need for lighter weight, higher performance components in aerospace, aircraft and automotive industries. The common discontinuous reinforcements for AMCs include particulates, whiskers or short fibers, and platelets. Compared with other ceramic reinforcements, whiskers can slow down the degradation of ductility of the matrix, and improve the strength, which make the composites a promising substitution for aluminum alloys [1, 2]. Therefore, whiskers such as TiB [3-6], $MgAl_2O_4$ [7, 8], MgO [9], $Al_{18}B_4O_{33}$ [10, 11] and Al_2O_3 [12, 13] were utilized to reinforce Al matrix composites that are of interest in aerospace and automobile industries due to the combination of excellent properties of Al matrix and the whiskers, such as high specific strength and stiffness, good wear resistance, elevated temperature stability, and even low cost of production.

In general, there are two ways to fabricate metal matrix composites (MMCs) reinforced by whiskers, i.e., in-situ and ex-situ fabrication technologies. The ex-situ process inevitably cause several inherent problems, such as costly complicated fabricating process, high synthetic temperature, agglomeration of the reinforcements and poor interfacial bonding induced by chemical reaction at the whisker-matrix interfaces. In order to resolve these issues, ceramic particles or whiskers are in-situ fabricated to reinforced MMCs [5, 14-17].

In the past few years, gamma-Al_2O_3 was extensively studied, because of its wonderful surface activity and good mechanical properties [18, 19]. In addition, gamma-Al_2O_3 was used to coat some reinforcements to achieve well interface bonding between the Al matrix and the reinforcements [20]. It proves that gamma-Al_2O_3 has a strong binding energy with Al matrix.

In this work, in-situ synthesis was first used to prepare gamma-Al_2O_3 whiskers reinforced aluminum matrix by sintering after cold pressing (SCP) and hot-pressed sintering (HPS) method, allowing gamma-Al_2O_3 whiskers compounding in sintering process. The wonderful lattice matching between the whiskers and Al matrix was discovered. Additionally, the structure and distribution of the whiskers were investigated, and the mechanical properties of the composites were investigated, especially Al_2O_3 whiskers can effectively slow down the degradation of ductility of aluminum matrix.

2 Materials and methods

Commercial Al powders (average size 75 μm, 99% purity, Tianjin Weichen Co., Ltd.), boric acid (99% purity, Aladdin Industrial Corporation) and stearic acid (99% purity, Tianjin

Guangfu Fine Chemical Institute) were used as starting materials without further purification.

The process flow diagram is exhibited in Fig. 1 and Fig. 2. The Al powders and boric acid were firstly mixed at a mass ratio calculated according to the theoretical value of whiskers after the reaction shown in equation (1-3). And 0.2 wt% stearic acid was added as the processing control agent to prevent the metal powders from cold welding during ball milling. The mixture was milled for 2 h with the ball-to-powder mass ratio of 5:1 at a rotation speed of 400 rpm. The obtained Al-H_3BO_3 powders were cold pressed in a steel mold with 20 mm in diameter under a pressure of 600 MPa. The obtained compact was put in a muffle furnace, and sintered at 600~700 ℃ for 1h in an argon atmosphere during which the Al_2O_3 whiskers in-situ generated in the matrix and Al_2O_3 whiskers/Al composite sample was obtained. For HPS method, the mixed powder was molded only by one step. Then the composite sample was hot-extruded through a steel die with an extrusion ration of 16:1 at 550 ℃.

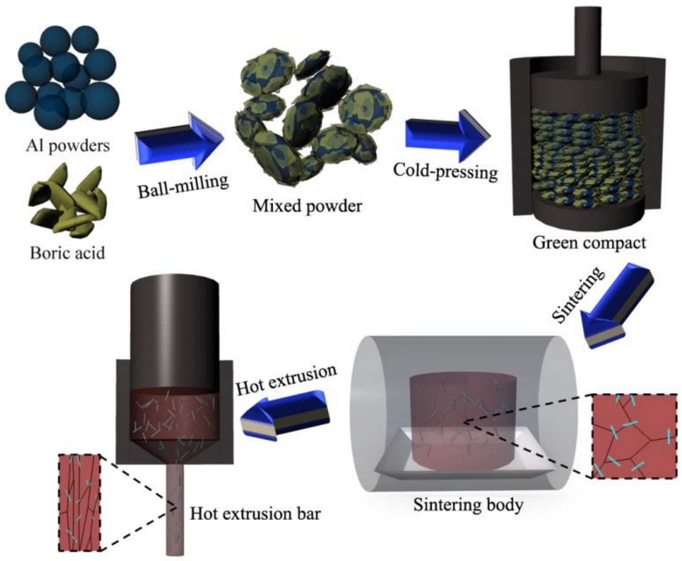

Fig.1. Schematic diagrams of the SCP process.

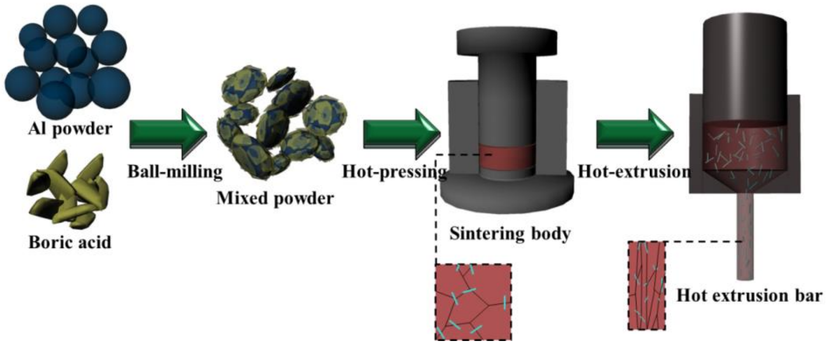

Fig.2. Schematic diagrams of the HPS process.

3 Results and discussion

3.1 In-situ growth of gamma-Al_2O_3 whiskers

Fig. 3 is from the sample made by SCP. Fig. 3 a shows the Al particles after ball milling, which presents that boric acid particles homogeneously adhere on the Al particle. The homogeneous distribution of boric acid particles on Al particle surface is beneficial for the dispersion of the whiskers in the Al matrix because the whiskers nucleate on boric acid particles. Fig. 3 b exhibits that the whiskers embedded in the Al matrix uniformly when the polished sample was etched with Keller's reagent. To reveal the morphology of the whiskers, the corrosion was prolonged for 1 min. We find that there are a number of whiskers in the corrosion pit, with the diameter of 20 nm, and the aspect ratio of 10 to 15, as shown in Fig. 3 c. XRD pattern of the extracted whiskers is shown in Fig. 3 d. All the diffraction peaks are well indexed to the standard spectra of the gamma Al_2O_3 (PDF No. 10-0425). It proves that the SCP method can in-situ synthesize pure gamma-Al_2O_3 in Al matrix.

Fig. 4 is from the sample made by HPS. Fig. 4 a-h shows the whiskers embedded in the Al matrix when the polished sample was etched with Keller's reagent. We find that the diameter of 50-100 nm, and the aspect ratio of 5 to 10, which is different to SCP. XRD pattern of the extracted whiskers is shown in Fig. 4 i. Al, Al_2O_3 and AlB_2 can be detected.

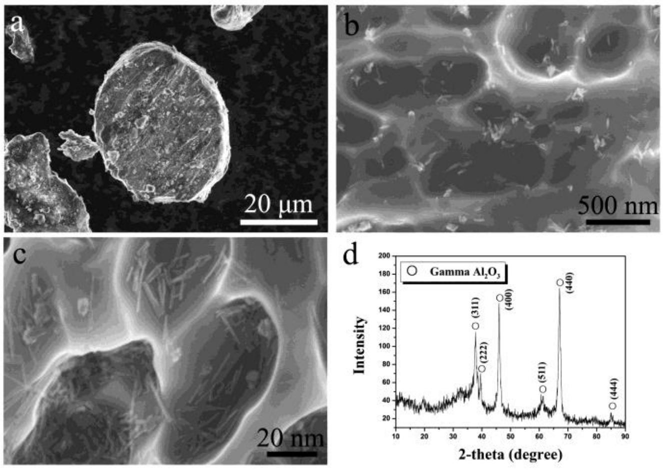

Fig. 3 SEM images of the Al particle covered by boric acid (a), Al_2O_3 whiskers (b) and (c), and XRD pattern of the whiskers (d).

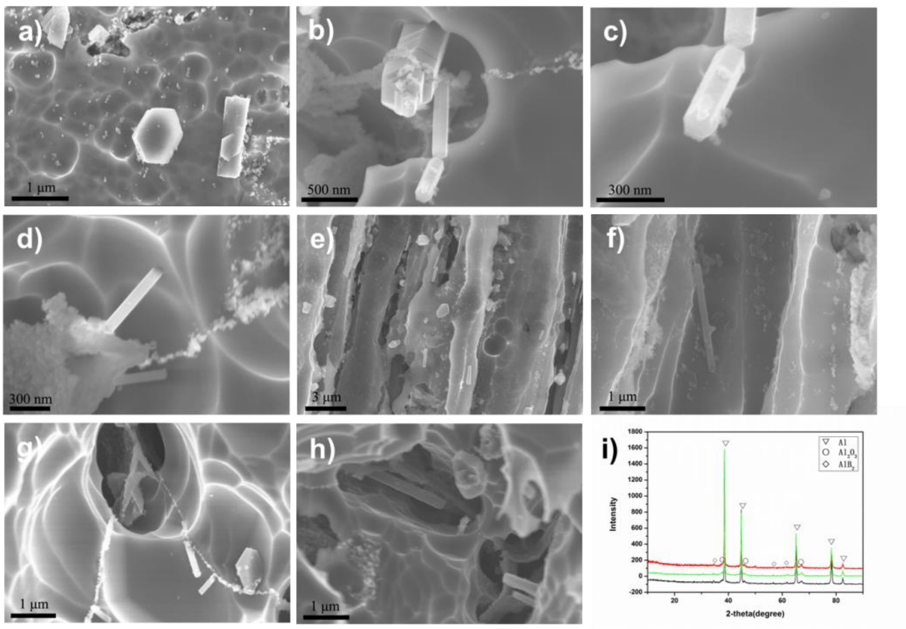

Fig. 4 SEM images of the Al2O3 whiskers (a-h), XRD pattern of the whiskers (i)

3.2 Interface between gamma-Al_2O_3 whisker and Al matrix

The interfacial bonding between the reinforcement and matrix plays a significant role on the comprehensive performance of the composites. The high-resolution TEM observation reveals that the gamma-Al_2O_3 whiskers/Al interface is clean, smooth and free from interfacial product, as shown in Fig. 5 a. Fig. 5 b reveals that the interface could not be recognized clearly which declares that the interface between gamma-Al_2O_3 whisker and Al matrix combines perfectly. But with the help of fast fourier transformation (FFT) at the zone (1) (2) (3), we can recognize the Al matrix, interface and gamma-Al_2O_3 whisker. Fig. 5 c is obtained by a software named Digital Micrograph (DM) from Fig. 5 b. By measuring, we determine that (200) planes of Al and (400) planes of gamma Al_2O_3 with the interplanar spacing of 0.2024 nm and 0.1977 nm, respectively, are shown in Fig. 5 c. The two interplanaers intersect, and the measured angle between the $(200)_{Al}$ and $(400)_{Al2O3}$ planes is about 93°.

Fig. 5 d schematically depicts the crystal configuration of the gamma-Al_2O_3 and Al matrix. Select the intersection point of $(200)_{Al}$ planes and $(400)_{Al2O3}$ planes as the initial vertex. After that, five parallels are draw down from the two crystal faces above, which are represented by blue and pink lines in Fig. 5 d. Subsequently, other five points are got successively. Connect the points to a line which is the black line shown in Fig.5 d. As the measuring result showed, the angle between the $(200)_{Al}$ planes and interface is about 47°, between the $(400)_{Al2O3}$ planes and interface is about 46°. On the basis of sine rule, we computed X and Y, shown in Fig. 5 d, as 0.27675 and 0.27484 respectively. Then the mismatch is 0.69% obtained. It can be inferred that during the in-situ generation process of gamma-Al_2O_3 whiskers, the Al and O atoms rearranged and formed Al_2O_3, meanwhile, the (200) plane of the whisker intersects with the (400) plane of the matrix at an approximate angle of 93°. And the Al atoms on the interface are shared between whisker and matrix leading to a little movement of both matrix and whisker in the transition zone. It makes a wonderful lattice matching between the sides of whiskers and Al matrix. So that the strong bonds of whisker-Al matrix is the key to protect whiskers from sliding in matrix. That is why the in-situ gamma-Al_2O_3 whiskers/Al can reach an excellent mechanical property.

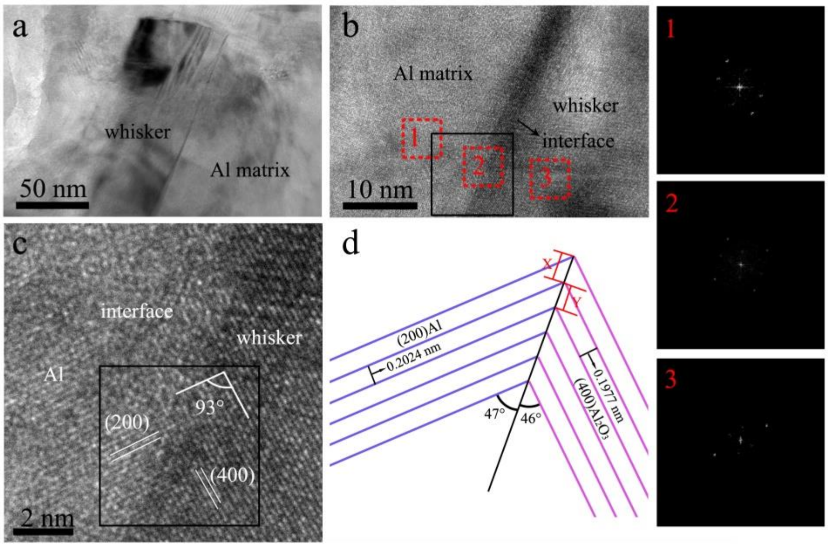

Fig. 5 TEM images of gamma Al2O3 whisker (a), gamma-Al2O3 whiskers/Al interface (b),

the interface between whisker and matrix (c), schematic diagram of the interfacial structure (d) (Black, blue and pink lines represent the interface, crystal faces of Al matrix and gamma-Al2O3 whisker, respectively), and FFT of the zone (1) (2) (3).

3.3 Mechanical property of the in-situ synthesized composites

The tensile stress-strain curves of the Al_2O_3/Al composites with 5 wt. %, 10 wt. %, and 15 wt. % whiskers made by SCP are shown in Fig. 8. The tensile stress-strain curve of cool-pressed sintered pure Al is also displayed for comparison. With the content of gamma-Al_2O_3 whiskers increasing from 0 to 10 wt. %, tensile strength increased, and the tensile increased by about 60.7% compared to pristine Al. However, when the whisker content reaches 15 wt. % the tensile strength falls down, which can be attributed to agglomeration of the gamma-Al_2O_3 whiskers. In addition, elongation at break decreases with increasing the content of the whiskers, indicating the increase of the brittleness of the Al matrix composites by inducing Al_2O_3 whiskers. The ultimate tensile strength of the 10 wt. % Al_2O_3 whiskers/Al composite is increased by 60.7%, displaying a plasticity of 11%, which is higher than the critical ductility (5%) that is required for many structural applications. It testifies that whiskers can slow down the degradation of ductility of the matrix, and improve the strength.

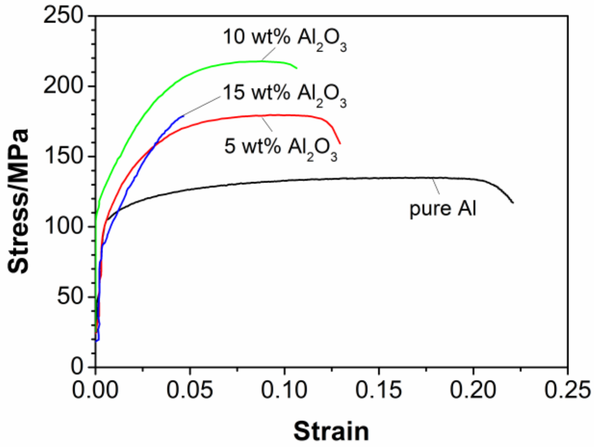

Fig. 6 Tensile stress-strain curves of pure Al and Al2O3/Al composite with different whiskers content made by SCP.

4 Conclusion

The in-situ generated gamma-Al_2O_3 whiskers/Al composite was fabricated through cold-press sintering followed by hot extrusion. The perfect lattice matching between gamma-Al_2O_3 and Al with the mismatch of 0.69% induced a well interfacial bonding between the reinforcement and matrix. The in-situ generated whiskers, which improve the ultimate tensile strength of pure Al by 60.7%. And the plasticity could be protected well, when the tensile strength was improved by the gamma-Al_2O_3 whiskers.

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