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A multiple grain size distributed Al-based composite consist of amorphous/nanocrystalline, submicron grain and micron grain fabricated through spark plasma sintering

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ABSTRACT

A multiple grain size distributed Al-based composite was fabricated through ball milling the amorphous powder mixed with pure Al powder followed by spark plasma sintering. The composite consisted of amorphous/nanocrystalline, submicron grain and micron grain, exhibiting outstanding mechanical properties. The superior strengthen effect of the amorphous/nanocrystalline region contributed to the exhibiting specific strength (5.66×10^5 Nm/kg) of the composite. The orderly plastic deformation occurred within FCC-Al micron grain region and submicron grain region during compression. And the progressive deformation restrained the strain localization and unstable propagation of the crack, leading to the plastic deformation (4.3%) of the composite.

1. Introduction

Nanocrystalline (NC) Al alloys have attracted great interest in the last decades owing to the excellent mechanical properties such as high specific strength, hardness and wear resistance [1–5]. However, grain refinement severely impedes the generation and motion of dislocation, subsequently decreasing the work hardening capacity and ductility of the NC materials [6–9]. One of the attractive strategies to improve the ductility of NC material is introducing micron-size grains, namely, bimodal grain size distribution structure [10–12]. The high strength originates from the NC phase and micron grains contribute to the ductility.

Powder metallurgy (PM) routes have been successfully used for fabrications of bimodal grain size distribution (BGSD) alloys [13–16]. The nanostructured powders are mixed with appropriate amount of micron grain powders firstly and sintered immediately by various consolidation methods. In general, BGSD alloys prepared by PM consist of the submicron grains (100–300 nm) and micron grains (1–2 μm), and the alloys with such grain size distribution do not exhibit outstanding strength compared with the high strength Al alloy [17–19]. Thus, to increase the strength of the alloy, we attempt to further reduce the grain size of the BGSD alloy, from submicron/micron grains to nanocrystalline/submicron grains.

According to our previous work, stable NC structure with grain size within 50 nm could be obtained through crystallization of the $Al_{65}Cu_{16.5}Ti_{18.5}$ amorphous phase after appropriate annealing [20], thereby, nanostructured powder is replaced by the Al-based amorphous alloy to obtain the homogeneous NC region. Pure Al powder after proper ball milling (BM) accompanies with appropriate sintering condition could provide the submicron grains, and the addition of Al could maintain the high specific strength of the composite.

Spark plasma sintering (SPS) was employed in the present research, which is particularly suitable for fabricating nanostructured or amorphous materials because of the low sintering temperature and short sintering time [21–24]. More remarkable, the contact position of powders usually suffers rapid superheating due to the Joule heat generated by the pulse current during SPS [25–27].

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And gradient thermal distribution caused by different resistivity and thermal conductivity of powders induce the formation of multiple grain size distributed structure. For example, when preparing Al-based amorphous alloy/high entropy alloy composite by SPS [28], an inter diffusion layer with submicron grains formed between the amorphous/nanocrystalline region and high entropy coarse grains, and the fracture strength of the composite could reach to 3200 MPa because of the transition layer. Resistivity and thermal conductivity difference between pure Al and Al-based amorphous powders may also lead to the formation of some desirable structure.

Simultaneously, to avoid the severe interface reaction or even the formation of intermetallics shell, which will deteriorate the ductility of the composite [29], the Al-based amorphous powder was embedded in the Al powder through BM. The microstructure and mechanical properties of the composite as well as the sintering behaviors were investigated.

2. Experimental

The elemental powders of Al, Cu, and Ti (purity >99.9 wt % and 50 μm in average particle size) were mixed in the desired nominal compositions (at. %) of $Al_{65}Cu_{16.5}Ti_{18.5}$ followed by BM. The milling experiments were performed on a high-energy vibrating ball mill (SPEX 8000D) with milling rate of 875 r/min. Hard alloy balls and stainless steel milling pot were employed in the present experiment, the weight ratio of ball to powder is 10:1. To avoid oxidation, all of the powders were handled in a glove box under argon atmosphere. After BM for 30 h, the as-milled amorphous $Al_{65}Cu_{16.5}Ti_{18.5}$ powder was obtained.

The Al coarse grain powder was prepared through gas-atomization under argon atmosphere. Al-based amorphous powder mixed with pure Al powder was ball milled for different time to investigate the structure evolution during BM. Given the strength and density of the composite, the volume fraction of the Al powder was determined to be 40% and 60%.

Based on the crystallization behavior of $Al_{65}Cu_{16.5}Ti_{18.5}$ amorphous powder [20], crystallization temperature (T_x) of the amorphous powder were 619.3 K. NC with grain size of about 50 nm formed when annealing at 673 K–773 K. Based on the previous research [30], strong metallurgic bonding among powders could be obtained when sintering above the supercooled liquid region, which led to high strength. Unfortunately, high temperature led the amorphous powder to crystallize severely, even coarse grains of the crystallized amorphous alloy. Besides, when sintering at 823 K, localized high temperature during SPS may exceed the melting point of Al phase, leading to the melting of Al. The above factors all deteriorated mechanical property of the alloy. Therefore, the temperature was optimized to be 773 K. Consolidation was conducted by SPS with pressure of 400 MPa and the vacuum of less than 10 Pa. The density of the sintered composites was tested by Archimedes-method. The microstructure was characterized by X-ray diffraction (XRD) using a Philips PW1050 diffractometer (Co Ka radiation), transmission electron microscopy (TEM) using TECNAI F30 and scanning electron microscopy (SEM) using a Hitachi TM-1000 tabletop microscope. The mechanical property of the specimens was tested with an INSTRON 8562 testing facility with an extensometer under quasi-static loading (strain rate of $1 \times 10^{-3}s^{-1}$) at room temperature, at least five samples with size of $6 \times 3 \times 3$ mm were measured to ensure the reliability.

3. Results

3.1. Structure

3.1.1. Powder

Fig. 1 shows the XRD patterns of the as-milled Al-based amorphous powder and the composite powder (60% volume fraction of Al powder, PAl60) after BM for 5 h and 10 h. The XRD pattern of the amorphous powder exhibits almost amorphous structure except for a weak TiH_2 peak, which was attributed to the reaction between Ti and toluene [31]. The pattern of the composite powder after BM for 5 h displays sharp Al peaks belonging to pure Al powder, together with the broad peak due to the amorphous phase. With an increase of the BM time to 10 h, Al_2Cu and AlCu appear on the XRD pattern, which is caused by the interface diffusion reaction between the Al powder and the amorphous powder or the crystallization of $Al_{65}Cu_{16.5}Ti_{18.5}$ amorphous phase during BM. Al-Ti intermetallic was not found on the XRD pattern (Fig. 1), on one side, the diffusion reaction between Al and Ti may induce the formation of Al(Ti) solid solution, on the other hand, when the amount of Al-Ti intermetallic was little, XRD cannot detect the existence of Al-Ti intermetallic. Both of the intermetallics and grain refinement caused by long time BM will reduce the ductility of the following sintered sample. Thus, the BM time was determined to be 5 h.

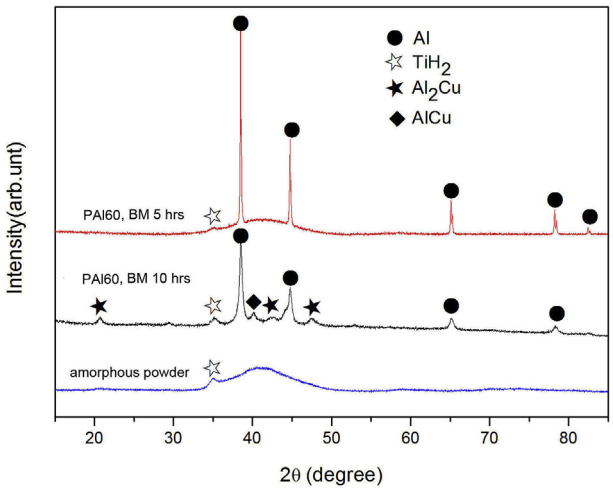

Fig. 1. XRD patterns of the amorphous powder and the composite powder PAl60 after BM for 5 h and 10 h.

SEM images of the amorphous powder, Al powder, and the cross section of the composite powder PAl40 (BM for 5 h) are shown in Fig. 2. The initial amorphous powder (Fig. 2a) broke into smaller particles with size of 2–4 μm under the effect of BM (Fig. 2c), and they embedded into the Al powder. Nearly no pore is found in the mixed powder in the magnified image (Fig. 2d), and no obvious interface reaction layer is found between the amorphous powder and Al powder.

3.1.2. Sintered sample

The density of the composites SAl40 and SAl60 were 3.27 g/m^3 (relative density 99.2%) and 3.02 g/cm^3 (relative density 99.6%), respectively. The XRD patterns of the sintered composites with 40 and 60 vol % Al powder (SAl40 and SAl60) are shown in Fig. 3. Several peaks identified as Al, Al_3Ti, Al_2Cu, and AlCu superimposed on a broad scatting hump on the XRD patterns, demonstrating the mixed structure of Al phase, intermetallics and amorphous phase. The average grain size of the intermetallics was estimated to be 20–30 nm according to the Scherrer formulation. TiH_2 is thermal metastable [31], so the peak of TiH_2 disappeared after sintering. Since the sintering temperature is higher than T_x of the amorphous powder [22], crystal phases precipitated in the composite. Increasing Al content only strengthened the Al intensity but affected little on the intermetallics intensity.

Microstructure investigation including SEM images and corresponding energy dispersive X-ray (EDX) analysis of composite