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Synthesis and mechanical properties of bulk $Al_{76}Ni_8Ti_8Zr_4Y_4$ alloy fabricated by consolidation of mechanically alloyed amorphous powders

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ABSTRACT

Mechanically alloyed amorphous $Al_{76}Ni_8Ti_8Zr_4Y_4$ (at.%) alloy powder was consolidated by high-pressure sintering process. The influence of the consolidation temperature on the structure and mechanical properties of the consolidated bulk alloys was examined by X-ray diffraction (XRD), Optical microscopy (OM), Scanning electron microscopy (SEM), Vickers Hardness Tester and Nano Indenter. Structural investigations of the bulk materials revealed that most of the amorphous structure was retained after consolidation at 623 K, however, compaction at 723 K and 823 K caused crystallization of the amorphous phase with the appearance of white regions. The results also indicate that application of high pressure affected the crystallization products of the present alloy. Micro mechanical analysis showed that the microhardness of the bulk composites increased from $945HV_{0.1}$ to $1177HV_{0.1}$ with the consolidation temperature increasing. The evolution trends of nanohardness and Young's modulus agree well with the Vickers hardness testing results, indentation size effect (ISE) is observed in the multicomponent alloy systems. The yield strength, Meyer exponent and the indentation impressions of these bulk alloys have also been compared.

1. Introduction

Bulk amorphous–nanocrystalline alloys have breathed new life both into materials science and application areas, due to their unique combination of properties, such as extraordinary strength, high fracture toughness and good corrosion resistance [1–3]. Among them, Al-based amorphous–nanocrystalline alloys have attracted more and more attention owe to their advantageous mechanical properties in particular a high specific strength to weight ratio and a high hardness [4,5], which are not reached by conventional Al-based alloys. Unfortunately, the Al-rich glasses are difficult to manufacture on account of the low glass-forming ability (GFA) with extremely high cooling rate (10^6 K/s) [4,6], the largest critical thickness obtained so far is about 1 mm for $Al_{86}Ni_6Y_{4.5}Co_2La_{1.5}$ alloy [7] fabricated by rapid cooling method, which means that the application of Al-based metallic glasses as a structural material has been restricted.

Given the challenges for the fabrication of Al-riched bulk metallic glasses (BMGs) from liquid, an alternative way of producing bulk amorphous or nanocrystalline materials is powder metallurgy route. Since it is a solid state alloying process, a high cooling rate is not necessary and the alloy composition can be more flexible. Based on the glassy powders produced by mechanical alloying (MA), the bulk glasses can be produced via powder metallurgy route, which makes it possible to produce bulk metallic glasses in more extensive ranges of size and shape [8]. However, it faces a great challenge to consolidate the amorphous powders into dense bulk material maintaining amorphous structure or creating nanocrystalline, because the amorphous phase will crystallize and grain coarsening will occur during conventional consolidation at elevated temperature. Nevertheless, high temperature is indispensable to obtain good interparticle bonding. To achieve this, considerable efforts have been made to obtain bulk Al-based amorphous–nanocrystalline alloys by applying of a high pressure as well as limiting of the high temperature exposure time during the consolidation. Based on such consideration, Al-based bulk amorphous–nanocrystalline alloys have been successfully fabricated by high-pressure sintering (HPS) method, spark plasma sintering (SPS) process, and so on [4,9–11]. Among the different consolidation techniques, HPS consolidation, which involves the simultaneous application of pressure and temperature, shows a large potential for achieving fast and highly dense samples. Meanwhile, HPS route can also break the oxide layers coating on the powder particles, and hence gives improved bonding between the particles.

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In the present work, the synthesis of the bulk $Al_{76}Ni_8Ti_8Zr_4Y_4$ amorphous and nanocrystalline alloy by HPS process was studied, the effects of sinter temperatures on the structure evolution and micro-mechanical properties of the bulk alloys were investigated using XRD, OM, SEM, Vickers Hardness Tester and Nano Indenter. The hardness, yield strength and Meyer exponent of these alloys have been compared, the related deformation behavior and mechanism were also discussed.

2. Experimental methods

The mechanically alloyed $Al_{76}Ni_8Ti_8Zr_4Y_4$ powders milled for 120 h were used for consolidation, the structure and thermal properties have been investigated elsewhere [12]. Disk-typed $Al_{76}Ni_8Ti_8Zr_4Y_4$ bulk samples with a size of $\varnothing 10 * 2$ mm were prepared by HPS method, HPS was conducted using a cubic hinge press machine, which is widely used in the ultra-hard materials field. The consolidation processes were conducted for 5 min under high vacuum conditions (10^{-2} Pa), the applied uniaxial pressure was 4 GPa at three different temperatures. The density of the sintered samples was evaluated using the Archimedes method.

The specimens were mechanically polished to a mirror finish before structural analysis and micro-mechanical experiments. The structure of the sintered samples was studied by XRD analysis using Cu Kα radiation (X' Pert Pro MPD). For comparison, the structure of as-milled powders isothermally annealed at the same temperature for the same time was also studied by XRD.

Microhardness experiments were performed by MH-3 Vickers microhardness, the tests were carried out at five different loads of 25, 50, 100, 200 and 500 g and at a constant dwell time of 15 s. At least six points on each disk were selected for Vickers hardness testing.

Nanohardness of the bulk samples was evaluated using Nano Indenter XP system (MTS NANO Indenter) with a Berkovich diamond tip at room temperature. The experiments were conducted in the displacement-control mode with a maximum indentation depth of 1700 nm. The strain rate was controlled at 0.05 s^{-1}. The hardness and elastic modulus were averaged between the indentation depths of 200–1500 nm, six indentations were performed to verify the accuracy and scatter of the indentation data.

The indented impression was further observed under an optical microscope (OM Zeiss Imager. A1 m) and scanning electron microscope (SEM FEI Sirion 200) equipped with an X-ray energy dispersive spectroscopy (EDS) system for structural characterization and for determination of chemical composition.

3. Results and discussion

According to the DSC results of the $Al_{76}Ni_8Ti_8Zr_4Y_4$ powders milled for 120 h, 623 K, 723 K and 823 K were selected to consolidate the powders to a disk shape with a diameter of 10 mm and a thickness of 2 mm. Detailed XRD patterns of the samples consolidated under different temperatures are summarized in Fig. 1a. It can be seen that the bulk material reveals two weak crystallization peaks of fcc Al and an ambiguous amorphous background after sintering at 623 K, which suggesting that the most of amorphous state was retained. With the sintering temperature increased to 723 K, more crystalline peaks appear and the intensity of the peaks increases, meaning that the crystalline fraction in the BMG increases as the sinter temperature increased. The average grain size is about 90 nm and the crystallization products are fcc Al, Al_3Zr, Al_3Ti, AlY, Al_3Ni and other crystalline phases signed in Fig. 1a. There are some crystallized phases cannot be identified at present, because of the complexity of the multicomponent alloy. As the consolidated temperature increased to 823 K, the crystalline phase and grain size have not noticeable changes compared to the one consolidated at 723 K. The crystallinity of the samples consolidated at 4 GPa calculated from the software of X' Pert HighScore Plus was 3.3%, 24.0%, 29.5% at the sinter temperature of 623 K, 723 K, and 823 K, respectively, indicating the BMGs crystallize fraction increased with the enhanced sinter temperature.

For comparison, the as-milled powders were isothermally annealed in a tube resistance electric furnace filled with flowing high purity argon under ambient pressure, the annealing temperature and time are the same as those of the HPS bulk samples, the XRD patterns of the isothermally annealed powders are shown in Fig. 1b. It is seen that, under ambient pressure, the amorphous

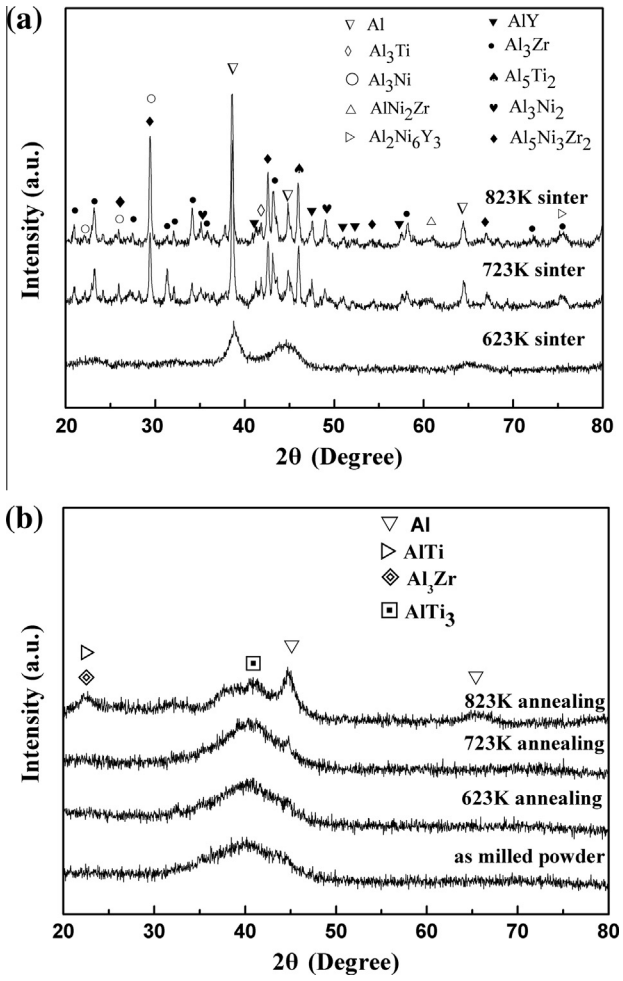

Fig. 1. XRD patterns of (a) the $Al_{76}Ni_8Ti_8Zr_4Y_4$ bulk samples consolidated at different temperatures; (b) the as-milled $Al_{76}Ni_8Ti_8Zr_4Y_4$ powders annealed at the same temperature for the same time with the bulk samples.

phase is retained when the annealing temperature is below 723 K. Further increase in annealing temperature results in the coexistence of amorphous and crystalline phases (fcc Al, Al_3Zr, AlTi and $AlTi_3$) with the extremely low intensity. It is clear that the crystallization products are not consistent with the consolidated bulk samples, which suggesting that the amorphous state of the fabricated samples are sensitive to the pressure applied.

In the present study, the major difference between the annealed and HPS specimens is the employment of high pressure. Thus, high pressure involved during consolidation can cut down on the existence of amorphous phase within the $Al_{76}Ni_8Ti_8Zr_4Y_4$ alloy. The fcc A1 precipitation temperature decreases dramatically with an increase of the applied pressure, from about 792 K to 623 K, which implies that the crystallization kinetic of Al–Ni–Ti–Zr–Y amorphous powders is altered due to the applied pressure. Pressure effect on crystallization kinetics of amorphous alloys has been investigated in several systems [13–18]. It was found that the crystallization kinetics is associated with the atomic diffusion process and the volume change effect during the initial stage of nucleation of crystals in the amorphous phase. In other words, the applied pressure effect may be twofold. On the one hand, amorphous state is thermodynamically unstable and has a larger excessive volume than the counterpart of its crystalline, crystallization is a process of increasing density, so pressure may enhance the crystallization process from a thermodynamic point of view, some researchers [14,16,18] have confirmed the conclusion. On the other hand, an