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## Compaction of bulk amorphous Fe<sub>40</sub>Ni<sub>40</sub>P<sub>14</sub>B<sub>6</sub> alloys

Qiang Li<sup>a,b,\*</sup>

<sup>a</sup> Department of Physics, The Chinese University of Hong Kong, Shatin, N.T., Hong Kong, PR China <sup>b</sup> School of Physics Science and Technology, Xinjiang University, Urumqi, Xinjiang 830046, PR China

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## Abstract

The consolidations of two bulk amorphous  $Fe_{40}Ni_{40}P_{14}B_6$  alloy discs are performed via hot pressing for a short time in its supercooled liquid region under a pressure of ~1.2 GPa. When the consolidated temperature  $T_s$  is lower, the conjunction of two bulk amorphous  $Fe_{40}Ni_{40}P_{14}B_6$  alloy discs cannot be achieved. Only when  $T_s$  get to the vicinity of 675 K, two amorphous  $Fe_{40}Ni_{40}P_{14}B_6$  alloy discs have low viscosity enough to be fully fused together in a short time and the resulting compacts retain ~90% amorphous phase. To further improve the consolidated temperature  $T_s$ , a vast amount of crystallization will occur and result in the embrittlement of amorphous alloy. © 2007 Elsevier B.V. All rights reserved.

Keywords: Hot pressing; Bulk amorphous alloy; Fe40Ni40P14B6; Supercooled liquid region

## 1. Introduction

Amorphous metallic alloys have excellent properties such as high mechanical strength, good residence corrosion ability, excellent soft and hard magnetism, and unique optical and electrical properties. Conventionally, in order to produce amorphous metallic alloys in the various techniques, a higher cooling rate is required and the resulting amorphous products are generally very thin, less than  $50 \,\mu\text{m}$ , in at least one dimension [1]. However, such a small physical size has so far limited industrial/commercial applications of this class of materials. Over the past decades, there has been a large advancement in the synthesis of bulk amorphous alloys via direct casting method [2-5]. In this method, the composition of alloys is designed to gain the large glass formation ability so that 'bulk' amorphous alloys with a dimension  $\geq 1 \text{ mm}$  in all directions can be prepared at a low cooling rate. Recently, a series of bulk amorphous alloys such as Pd-, Zr-, Mg-, Ln-, Ti-, Fe-, and Ni-based bulk glasses have successively been prepared by the direct casting method [6]. However, the maximum size of bulk amorphous metallic alloys prepared by means of direct casting method is still confined to its glass formation ability. Especially, for important bulk magnetic Fe-, Ni- and Co-based amorphous alloys, the maximum size

E-mail address: qli@xju.edu.cn.

is restricted to less than 10 mm. Furthermore, in order to produce bulk amorphous alloys, the optimization of composition frequently degrades their magnetic and mechanical properties. Thus, new techniques to synthesize the bulk amorphous alloys with larger size are still in need.

There is a standard metallurgical technique to prepare large chunks of materials, which is powder consolidation. Since the amorphous metallic alloys were first synthesized in 1960 [7], attempts have always been made to produce bulk amorphous alloys from amorphous powders or ribbons via various powder consolidation techniques. In order to retain the initial amorphous structure, the consolidation of amorphous powders and ribbons should be performed well below the glass temperature  $T_{\rm g}$ . However, amorphous metallic alloys usually have very high strengths at temperatures below  $T_{\rm g}$ . As a result, it is very difficult to produce full density and well-bonded bulk amorphous alloys by means of the conventional consolidation technique. Until now the consolidation of the amorphous metallic powders can be performed by four main routines: (1) static hot pressing technique [8]. In this method, the consolidation of amorphous metallic powders is usually performed under at a very high pressure at an elevated temperature in the range  $T_p < T < T_g$ for a longer isothermal time. Here,  $T_p$  corresponds to the transition temperature from inhomogeneous deformation mode to homogeneous deformation mode and  $T_g$  is the glass transition temperature of amorphous metallic powders. A higher compaction density better than 95% of the theoretical value can be

<sup>\*</sup> Fax: +86 991 8582405.

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obtained easily with this method but the bonding of powders or ribbons is very weak. So the resulting compacts have bad mechanical and magnetic properties. (2) Dynamic consolidation techniques such as explosive or gun compaction methods [9]. In the dynamic compaction process, a shock wave is sent through the powder. The work of deformation heats the powder heterogeneously. The more deformed regions may reach the melting temperature  $T_{\rm m}$  and the less deformed regions attain much lower temperatures. Following the passage of the shock wave, the cooler regions serve as heat sinks for the melted regions. If this energy balance is properly chosen, the hotter regions cool sufficiently fast to solidify back into the amorphous phase. (3) Quasistatic consolidation techniques such as warm extrusion [8]. In this compaction process, heat is generated locally on the particle surfaces by deformation and sliding of the particles over each other. Thus, the temperature in these surfaces may exceed  $T_{g}$  of amorphous metallic powders and an efficient friction weld is produced there to form the bond between the particles. Meanwhile the inner part of the particles remains relatively cold and can serve as the cooling sinks to quench the interface melted bond zones. Compared to method (2), there is longer contact time between the particles but smaller deformation energy, i.e. a lower temperature on the surface of the particles, in method (3). By means of methods (2) and (3), both the densification and bonding of the particles in the resulting compacts are better than that produced by method (1). However, the bonding strength of the final compacted products is still unsatisfactory for industrial applications. (4) Hot pressing in the supercooled liquid region. It is based on such a fact that amorphous alloys can become undercooled liquid and do not crystallize at the temperature range from the glass transition temperature  $T_{\rm g}$  to the kinetic crystallization temperature  $T_x$  in a short time. In the supercooled liquid region amorphous alloys soften and it is helpful for the consolidation of amorphous alloys. Recently, bulk Zr-based amorphous alloys have been successfully produced via warm extrusion of starting amorphous powders in the supercooled liquid region [10]. The resulting bulk amorphous compacts are fully densified and the compaction density exceeds 99.5% of the theoretical value. Furthermore, the well-bonded bulk compacts have been achieved and the resulting mechanical properties are comparable to that of the corresponding amorphous ribbons that is inspiring.

Comparing the above various compaction methods, it can be found that the method (4) is the only way available to produce the compacts for industrial applications. However, this method was only achieved for these systems with the large supercooled liquid region such as Zr-based amorphous alloys. It is not reported until now that this method has been successfully applied to systems with the small supercooled liquid region such as the important magnetic amorphous Fe-, Ni- and Co-based alloys.

Recently, it was found [11] that ferromagnetic bulk amorphous  $Fe_{40}Ni_{40}P_{14}B_6$  alloy rods with the maximum diameter of ~2.5 mm can be prepared by means of a rapid quenching technique. In this experiment, we will attempt to perform the compaction of bulk amorphous  $Fe_{40}Ni_{40}P_{14}B_6$  alloy in its supercooled liquid region. Further, as the preliminary and basic studies, the compaction between only two bulk amorphous  $Fe_{40}Ni_{40}P_{14}B_6$  alloy discs was considered in this experiment.

## 2. Experimental procedures

 $Fe_{40}Ni_{40}P_{14}B_6$  ingots were prepared from Fe chips (99.98%) pure), Ni spheres (99.95% pure), B pieces (99% pure), and Ni<sub>2</sub>P ingots (the Ni<sub>2</sub>P ingots used were themselves prepared from powders (98% pure)). After the right proportion was weighed, they were put in a clean fused silica tube and alloying was brought about in a rf induction furnace under Ar atmosphere. All the as-prepared specimens had a mass of  $\sim 2$  g and would be purified via the fluxing technique. The as-prepared  $Fe_{40}Ni_{40}P_{14}B_6$  ingots and the fluxing agent, anhydrous  $B_2O_3$ , were put in a clean fused silica tube. The whole system was evacuated to  $\sim 10^{-3}$  Torr and heated up to a temperature about  $\sim$ 200 K above the liquidus  $T_1$  of Fe<sub>40</sub>Ni<sub>40</sub>P<sub>14</sub>B<sub>6</sub> (=1184 K). Prolonged high temperature treatment was applied for about 4 h, the impurities and oxides inside the molten Fe<sub>40</sub>Ni<sub>40</sub>P<sub>14</sub>B<sub>6</sub> alloy can be removed into the fluxing agent. Then the system was cooled down to room temperature and the fluxed specimen, which was crystalline, was removed and cleaned for subsequent water quenching experiment. In the water quenching experiment, the fluxed ingots were again melted in Ar atmosphere and then introduced into a clean, thin-walled (0.1-0.2 mm) fused silica tube with the inner diameter of 1.6-1.8 mm. Then the whole system was plunged into water for rapid quenching. So, the amorphous Fe<sub>40</sub>Ni<sub>40</sub>P<sub>14</sub>B<sub>6</sub> alloy rods with the diameter of 1.6-1.8 mm and the length of  $\sim 4 \text{ cm}$  can be produced, and their amorphous nature had been verified by means of XRD, DSC and TEM. The details can be found elsewhere [11].

The as-prepared amorphous Fe<sub>40</sub>Ni<sub>40</sub>P<sub>14</sub>B<sub>6</sub> alloy rods with diameters of 1.6-1.8 mm were cut into discs with a thickness of 1.5 mm with a oil-cooled BUEHLER ISOMET low speed saw. These amorphous discs were cleaned in 100% alcohol and were subjected to subsequent hot pressing experiments. The consolidation of the amorphous Fe<sub>40</sub>Ni<sub>40</sub>P<sub>14</sub>B<sub>6</sub> alloy discs was performed in a standard high temperature hot pressing furnace as shown in Fig. 1(a). The pressure was provided by a manual hydraulic jack with the maximum load of 10 tonnes. A graphite die with an inner diameter of 23 mm was used here. In order to avoid contamination of the graphite die during the hot pressing, two steel discs with a thickness of 1 mm and a diameter of 22.5 mm were used to separate the graphite die from the specimens. The two as-prepared amorphous  $Fe_{40}Ni_{40}P_{14}B_6$  alloy discs were placed one on top of the other to perform the hot pressing experiment as shown in Fig. 1(b). In order to prevent the separation of two amorphous discs in the process of hot pressing, the specimens were put into a copper cylinder with a height of 2 mm and an inner/outer diameter of 2 mm/2.5 mm. A K type thermocouple was fastened closely on the top end of the upper mount of the graphite die to detect the temperature of the specimens. Since the graphite die has a very good thermal conductivity, and the space occupied by the specimens was very small, and meanwhile the distance between the thermocouple and the middle part of the specimens was less than 2 mm, it can be concluded that the temperature of the specimens can be measured accurately and timely by the thermocouple in the whole hot pressing process.

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