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Effect of cold rolling on compressive and tensile mechanical properties of $Zr_{52.5}Ti_5Cu_{18}Ni_{14.5}Al_{10}$ bulk metallic glass

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ABSTRACT

The effect of cold-rolling on structure, thermal stability, and compressive and tensile mechanical properties of the $Zr_{52.5}Ti_5Cu_{18}Ni_{14.5}Al_{10}$ bulk metallic glass has been investigated. The results reveal that cold-rolling does not influence the thermal stability of the material. However, the small plastic deformation induced by cold-rolling is very effective for improving the room temperature plastic strain of the glass: the compressive plastic strain increases from 1.1% for the as-cast material to 2.6% for the cold-rolled sample and the tensile ductility increases from 0% for the as-cast glass to 0.8% for the cold-rolled sample. Hardness measurements indicate that cold rolling creates a heterogeneous microstructure consisting of hard and soft regions. Most likely, the soft regions are preferred locations for shear band formation and propagation, whereas the hard regions may act as obstacles for shear band propagation, effectively limiting shear bands from propagating catastrophically and, consequently, enhancing the room temperature plastic deformation of the glass.

1. Introduction

Plastic flow of bulk metallic glasses (BMGs) generally occurs inhomogeneously and is localized into narrow shear bands, which results in an early and catastrophic failure [1,2]. As a result, under tensile loading, most BMGs show little macroscopic plasticity (<1%) at room temperature [1,2]. However, plastic deformation of metallic glasses under compressive loading can be improved by the creation of heterogeneous microstructures through the use of the proper mechanical pre-treatments. For example, the application of lateral pre-compression on the $Zr_{52.5}Cu_{17.9}Ni_{14.6}Al_{10}Ti_5$ glass creates soft and hard regions and induces the formation of a heterogeneous stress field, as observed by hardness measurements and finite element analysis [3]. As a result, plastic deformation increases from 1% for the as-cast rod to about 10% for the pre-deformed material [3]. Similar results have been observed for the $Zr_{41.2}Ti_{13.8}Cu_{12.5}Ni_{10}Be_{22.5}$ pre-deformed by channel-die compression [4]. This mechanical pre-treatment also creates soft and hard regions, effectively enhancing the room temperature plastic strain ability of the metallic glass [4]. Cold-rolling is also very useful for improving the plastic deformation of BMGs [5] through the creation of deformation-induced microstructural heterogeneities, as observed by transmission electron microscopy, remarkably enhancing the plastic deformation from 0.5% for the as-cast material to about 15% for the cold-rolled material [5]. As well, cold-rolling has positive effect on the tensile ductility of Zr-based BMGs, as recently reported by Cao et al. [6]. Along this line, this work examines the effect of cold-rolling on the room temperature mechanical properties of the $Zr_{52.5}Ti_5Cu_{18}Ni_{14.5}Al_{10}$ bulk metallic glass. Compressive and tensile tests reveal that cold-rolling is very effective in improving the plastic deformation of the glassy material.

2. Experimental

An ingot with nominal composition $Zr_{52.5}Ti_5Cu_{18}Ni_{14.5}Al_{10}$ (at.%) was prepared by arc melting in a titanium-gettered argon atmosphere. The ingot was remelted several times in order to achieve a homogeneous master alloy. From this ingot, plates with dimensions $1.7 \times 40 \times 80\,mm^3$ and cylindrical rods with 3 mm diameter and 80 mm length were prepared by copper mold casting. Plates were used for tensile tests, while cylindrical rods were used for compression tests. As-cast plates and rods were cold-rolled at room temperature to thickness and diameter reduction of 5 and 3%, respectively, using a laboratory rolling mill. As-cast as well as cold-rolled samples for tensile tests were prepared into dog-bone geometry with length of about 40 mm and width of the testing gauge of 2 mm by wire erosion. Cylindrical specimens with length/diameter ratio of 2.0 (6 mm length and 3 mm diameter) were prepared from the as-cast and cold-rolled samples. Both ends of the specimens were carefully polished to make them parallel to each other prior to the compression test. Tensile as well as compressive tests were carried out using an INSTRON 8562 testing facility with a strain rate of $1 \times 10^{-4}\,s^{-1}$. The strain during mechanical tests was measured directly on the specimen using a Fiedler laser-extensometer. At least four specimens for each condition were tested to ensure reproducibility of the results. The amorphous nature of the specimens was verified by X-ray diffraction (XRD) using a Siemens Micro-diffractometer ($\lambda = 0.1542$ nm), and the surface mor-

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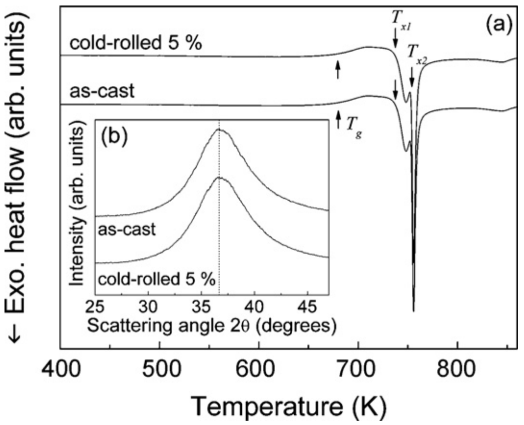

Fig. 1. (a) DSC scans (heating rate 40 K/min) and (b) XRD patterns (Cu K_α radiation) for the as-cast and cold-rolled $Zr_{52.5}Ti_5Cu_{18}Ni_{14.5}Al_{10}$ glassy plates.

phology was evaluated by scanning electron microscopy (SEM) using a HITACHI TM 1000 tabletop microscope. The thermal stability of the samples was investigated by differential scanning calorimetry (DSC) with a PERKIN-ELMER DSC7 calorimeter at 40 K/min heating rate under a continuous flow of purified argon. Hardness maps were done on the cross-section of both the as-cast and the cold-rolled plates using a computer-controlled STRUERS DURAMIN 5 Vickers hardness tester. Indentations were carried out every 50 μm with an applied load of 0.1 kg and dwell time 10 s.

3. Results and discussion

As a typical example of the effect of cold-rolling on the thermal stability of the glass, Fig. 1(a) shows the constant-rate heating DSC scans (40 K/min) of the as-cast and cold-rolled $Zr_{52.5}Ti_5Cu_{18}Ni_{14.5}Al_{10}$ plates. Heating the as-cast material from room temperature up to elevated temperatures reveals a distinct endothermic event characteristic of the glass transition (T_g) at 677 K. With increasing temperature the DSC curve exhibits two crystallization events with onsets T_{x1} and T_{x2} (738 and 753 K, respectively). The supercooled liquid region, defined as $\Delta T_x = T_{x1} - T_g$ and corresponding to the stability of the supercooled liquid against crystallization, is 61 K. The enthalpy of crystallization related to the exothermic DSC peaks is 50.7 J/g. The same thermal stability data are observed for the cold-rolled glass, which indicates that the small deformation induced by cold-rolling does not lead to any significant variation of the thermal stability of the $Zr_{52.5}Ti_5Cu_{18}Ni_{14.5}Al_{10}$ BMG. As well, cold-rolling does not induce any measurable change in the XRD diffraction patterns (Fig. 1(b)). The patterns reveal the typical broad maximum characteristic for amorphous materials for both as-cast and cold-rolled plates and no distinct crystalline peaks are detected within the sensitivity limits of XRD. Similar results (not shown here) have been observed for cold-rolling of the cylindrical specimens.

Fig. 2(a) and (b) shows the shear band morphology of the free surface (i.e. *z*–*y* surface, normal to the rolling direction) of the cold-rolled samples. Plastic deformation during rolling induces the formation of several shear bands in both the cylindrical rod (Fig. 2(a)) and plate (Fig. 2(b)). Besides shear banding, cold rolling creates a heterogeneous microstructure in the samples, as shown by the hardness maps taken on the free surface of the plates (Fig. 3(a) and (b)). The as-cast material displays a narrow range of hardness values ranging between 500 and 540 HV (Fig. 3(a)), which implies a rather homogeneous microstructure. Conversely, the cold-rolled sample (Fig. 3(b)) is much more heterogeneous and is characterized by a wider range of hardness values (440–550 HV). These results indicate that strain-induced softening occurs during cold-rolling, in agreement with the results on indentation of other BMGs [7,8].

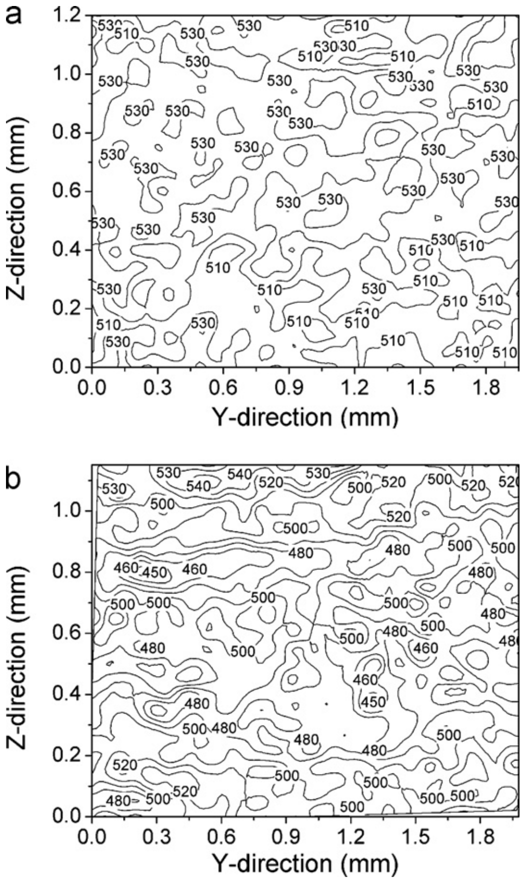

Fig. 3. Hardness maps of the free surface for the (a) as-cast and (b) cold-rolled glassy plates.

The room temperature tensile and compressive stress–strain curves for the as-cast and cold-rolled glassy samples are shown in Fig. 4. The as-cast specimen tested under tension exhibits a

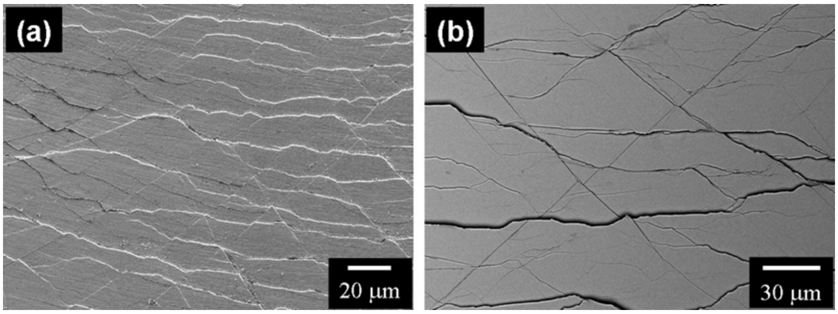

Fig. 2. Cross-section of the cold-rolled $Zr_{52.5}Ti_5Cu_{18}Ni_{14.5}Al_{10}$ rod (a) and plate (b), revealing the formation of several shear bands.

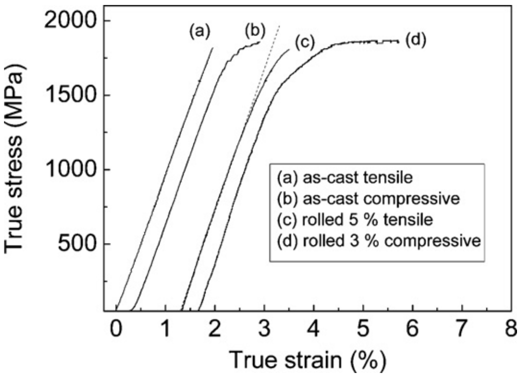

Fig. 4. Room temperature true stress–true strain curves for the $Zr_{52.5}Ti_5Cu_{18}Ni_{14.5}Al_{10}$ metallic glass: (a) as-cast tested in tension, (b) as-cast tested in compression, (c) cold-rolled tested in tension and (d) cold-rolled tested in compression.

perfect elastic regime of 2.0 ± 0.1% before fracture, which occurs at about 1780 ± 40 MPa without any visible macroscopic ductility. Such a brittle behavior is similar to what observed for bulk metallic glasses tested in tension [1,2,9,10]. On the other hand, the compressive curve of the as-cast material exhibits an elastic regime of 1.8 ± 0.2% before yielding, which occurs at about 1600 ± 50 MPa. After yielding the stress increases with strain reaching a compressive strength of 1850 ± 50 MPa. Fracture occurs at 2.9 ± 0.2% strain, therefore, giving a plastic deformation of about 1.1 ± 0.3%.

Cold-rolling remarkably affects the tensile as well as the compressive mechanical properties of the glass. The cold-rolled sample tested in tension displays a clear yield, which occurs at 1.4 ± 0.1% strain and at about 1350 ± 30 MPa. The stress then increases with strain up to 1800 ± 40 MPa where fracture occurs at 2.2 ± 0.1% strain, which corresponds to a tensile plastic deformation of about 0.8 ± 0.15%. Similarly, the compressive yield strength of the cold-rolled BMG decreases to 1450 ± 50 MPa with respect to the as-cast material (1600 MPa), whereas the compressive strength is unchanged (1850 MPa). Most important, the plastic deformation increases to about 2.6 ± 0.3%, which is more than two times larger than the as-cast material.

In general, monolithic BMGs exhibit only limited macroscopic plasticity at room temperature due to the formation of highly localized shear bands [1,2]. Spreading of such localized shearing events occurs around shear transformation zones (STZs) and creates free volume [1,2]. Accumulation of free volume inside the shear bands decreases the viscosity, which induces strain/thermal softening [11]. Due to local softening, the region within the shear band deforms more easily than the rest of the sample and, as a result, only a few shear bands are activated, leading to catastrophic failure soon after yielding [11]. However, early fracture of BMGs can be delayed by controlling two factors: shear band formation and propagation [2]. The formation of multiple shear bands permits the distribution of the plastic strain over several bands [1], whereas limiting/arresting shear band propagation avoids the autocatalytic softening that would lead to failure along a single shear band [2].

This behavior can be induced by manipulating the microstructure through the creation of heterogeneous stress fields and the generation of soft and hard regions [3]. The soft regions may become preferred locations for subsequent deformation [8,12] and may assist further shear band initiation, while the hard regions can effectively impede shear bands from propagating catastrophically by branching/arresting of multiple shear bands [3,12].

The present cold-rolled samples can be analyzed in a similar way. The cold-rolled material contains a large amount of shear bands (Fig. 2) and can be considered as a network of hard and soft regions (Fig. 3(b)). Because of this heterogeneous microstructure, different areas in the glass may require a different critical stress to initiate deformation during subsequent compressive and tensile tests. Most likely, the pre-existing shear softened areas are reactivated at low stress explaining the low yield strength of the cold-rolled samples [1,2]. The hard regions may then act as obstacles for shear band propagation and, as a result, plastic deformation within the pre-existing shear bands stops [2]. After the shear bands at the weakest sites are arrested, new shear bands have to be formed to accommodate the applied strain. The critical stresses needed to generate the new shear bands gradually increases from easy to difficult nucleation sites [2], explaining the apparent work hardening behavior visible in Fig. 4. Upon continued straining, this propagation-arrest mechanism would accommodate an increasing amount of deformation, leading to the larger plastic strain observed for the present cold-rolled material with respect to the as-cast samples.

4. Conclusions

The influence of cold-rolling on structure, thermal stability, and room temperature compressive and tensile mechanical properties of the $Zr_{52.5}Ti_5Cu_{18}Ni_{14.5}Al_{10}$ bulk metallic glass has been investigated. Cold-rolling does not affect the thermal stability of the glass. However, it induces the formation of several shear bands and creates a heterogeneous microstructure consisting of hard and soft regions, as demonstrated by the hardness measurements. As a result of the generation of such structural heterogeneities, the room temperature plastic deformation of the metallic glass is significantly improved: the compressive plastic strain increases from 1.1% for the as-cast material to 2.6% for the cold-rolled sample. As well, the tensile ductility increases from 0% for the as-cast glass to 0.8% for the cold-rolled sample.

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