Journal of Alloys and Compounds 586 (2014) S36–S40

Contents lists available at SciVerse ScienceDirect

Journal of Alloys and Compounds

journal homepage: www.elsevier.com/locate/jalcom

Effects of shot peening on the nanoindentation response of $Cu_{47.5}Zr_{47.5}Al_5$ metallic glass

J. Fornell [a], A. Concustell [b], A.L. Greer [c], S. Suriñach [a], M.D. Baró [b], J. Sort [d,*]

[a] Departament de Física, Facultat de Ciències, Universitat Autònoma de Barcelona, E-08193 Bellaterra, Spain
[b] La Farga Lacambra, Ctra. C-17, Km. 73,5, 08508 Les Masies de Voltregà, Barcelona, Spain
[c] Department of Materials Science and Metallurgy, University of Cambridge, Pembroke Street, Cambridge CB2 3QZ, United Kingdom
[d] Institució Catalana de Recerca i Estudis Avançats (ICREA) and Departament de Física, Universitat Autònoma de Barcelona, E-08193 Bellaterra, Spain

ARTICLE INFO

Article history:
Available online 20 December 2012

Keywords:
Metallic glasses
Shot peening
Microstructure
Mechanical properties

ABSTRACT

The effects of shot peening (SP) on the nanoindentation response of $Cu_{47.5}Zr_{47.5}Al_5$ metallic glass, both in the as-cast state and after thermally induced structural relaxation (i.e., after annealing close to the glass transition), are investigated. As expected, annealing causes an increase of hardness attributable to annihilation of free volume. Conversely, nanoindentation tests, carried out on a transverse section at different distances from the SP surface, reveal that SP induces mechanical softening. Both the hardness and the reduced Young's modulus progressively decrease as the peened surface is approached. Finite-element simulations of the nanoindentation curves indicate that the cohesive stress and the Mohr–Coulomb friction coefficient also decrease after SP, an observation which is in line with the induced mechanical softening. Differential scanning calorimetry measurements reveal an increase of the relaxation enthalpy, particularly when SP is performed on the previously annealed metallic glass, consistent with deformation-induced creation of free volume.

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1. Introduction

Traditionally, physical and chemical treatments of the surface of metallic alloys have been performed with the aim of controlling friction and wear, improving corrosion resistance, altering the final appearance (e.g. colour, lustre) of the material, or promoting the change of a physical property such as conductivity or reflectivity. Among the investigated treatments, shot peening (SP), laser peening or ion irradiation have been used to alter the surface of polycrystalline alloys through the creation of structural disorder and reduction of the grain size or, in bulk metallic glasses, to induce residual stresses and increase the amount of free volume within the amorphous structure. In all cases, this leads to significant changes in the resulting static and dynamic mechanical properties as well as in the corrosion behaviour [1–7]. SP treatments have been performed on bulk metallic glasses, for example, to improve the overall macroscopic compression plasticity from about 10% (as-cast state) to roughly 25% [1] or to increase the spontaneous passivity during corrosion tests [8]. The plastic flow associated with SP typically brings about a decrease of hardness (i.e. softening) at distances of up to tens of μm from the peened surface [1,6]. Strain softening has been often reported in metallic glasses during macroscopic compression experiments or nanoindentation, eventually leading to the so-called "indentation size effect" [9–13].

Plastic flow in metallic glasses is accompanied by dilatation, i.e., creation of free volume which is strain-rate dependent [9,10,12,14]. The net increase of free volume due to the applied shear stresses can be qualitatively assessed from differential scanning calorimetry curves, where an increase of the relaxation enthalpy is usually observed during heat treatments of a previously deformed metallic glass. The strain softening associated with generation of free volume leads to sharp localization of plastic deformation in the form of shear bands. As a consequence, stress serrations are observed in the compression stress–strain curves or during nanoindentation loading [12].

Because of the peculiar mechanisms that govern deformation behaviour of metallic glasses, yielding in these materials cannot be simply described by the conventional von Mises or Tresca criteria, as in crystalline metals but, instead, the normal stress components acting on the shear plane need to be also taken into account [12,15,16]. The Mohr–Coulomb yield criterion can be expressed as follows:

$$\tau_y = c - \beta_{MC}\sigma_n \tag{1}$$

where τ_y is the shear stress on the slip plane at yielding, c is the shear strength in pure shear (termed the *cohesive stress*), β_{MC} denotes the in the *Mohr–Coulomb friction coefficient* of the glass and σ_n is the normal stress acting on the shear plane. In turn, β_{MC}

* Corresponding author. Tel.: +34 935812085; fax: +34 935812155.
E-mail address: jordi.sort@uab.cat (J. Sort).

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http://dx.doi.org/10.1016/j.jallcom.2012.12.051

is related to the angle between shear bands and the applied stress direction, θ_C, through:

$$\beta_{MC} = \frac{\cos 2\theta_C}{\sin 2\theta_C} \quad (2)$$

Interestingly, both c and β_{MC} can be quantitatively determined from finite-element simulations of the nanoindentation curves [12]. Although variations of such parameters have been quantified for the case of compressed or annealed specimens [12,17], the possible influence of SP on the cohesive stress and friction coefficient has so far been largely overlooked.

In this work we investigate the effects of SP on the structure and mechanical properties of a $Cu_{47.5}Zr_{47.5}Al_5$ metallic glass sheet. No crystallization occurs at the surface of the metallic glass as a result of SP, but there are structural changes leading to mechanical softening detectable by nanoindentation. Indeed, on a section transverse to the SP surface, nanoindentation reveals reductions in hardness and reduced Young's modulus, of up to 25% within 60–80 μm from the SP surface. This effect is particularly pronounced after performing SP on the sample previously relaxed by thermal annealing.

2. Experimental details

Ingots with nominal composition $Cu_{47.5}Zr_{47.5}Al_5$ (at.%) were prepared by arc-melting the pure elements (99.9%) under an argon atmosphere. Sheets of 3 mm in thickness were obtained from the arc-melted alloy by copper mould casting in a purified argon atmosphere. The as-prepared sheets were polished with grinding paper and diamond paste to achieve a mirror finish. Subsequently, SP treatments of 30 s duration were performed at 298 K using glass beads 300–400 μm in diameter, accelerated by a gas pressure of 6 bar. These treatments were carried out both on the as-cast alloy and on a sample annealed for 5 min at 700 K, about 25 K below the glass transition (i.e. after structural relaxation).

The as-cast and shot-peened samples were structurally characterized by X-ray diffraction (XRD) using a Philips X'Pert instrument with Cu-Kα radiation. The thermal stability of the as-cast, relaxed and shot peened samples was investigated by differential scanning calorimetry (DSC) at a heating rate of 20 K/min using a Perkin-Elmer DSC7 instrument. In all cases, DSC measurements were performed from thin slices obtained from the $Cu_{47.5}Zr_{47.5}Al_5$ sheets. On SP samples a surface layer approximately 100 μm deep was sliced off and used for the subsequent characterization.

Nanoindentation measurements were performed in the load-control mode, using a UMIS instrument from Fischer-Cripps Laboratories equipped with a Berkovich pyramidal-shaped diamond tip. The maximum applied load was 10 mN. The thermal drift was always kept below ±0.05 nm s^{-1}. To study the effects of SP, the samples were cut perpendicular to the SP surface and nanoindentation tests were performed on the transverse section at different distances from the surface. From the load–displacement (P–h) curves, the hardness and reduced Young's modulus values were evaluated at the beginning of the unloading segments using the method of Oliver and Pharr [18,19]. Proper corrections for the contact area (previously calibrated using a fused quartz specimen), initial penetration depth and instrumental compliance were applied.

The nanoindentation curves were modelled using finite-element simulations (Strand7 software, developed by G+D Computing Pty Ltd.). A value of Poisson's ratio $\nu = 0.37$ [20] was used in the simulations. A frictionless contact between the indenter and the specimen was assumed. The Berkovich pyramidal indenter was treated as a conical tip with a cone half-angle of 70.3° that provides the same area-to-depth relationship as the ideal Berkovich indenter. This permits the use of axisymmetric elastic equations. It should be noted that although contact solutions for pyramidal indenters have been occasionally reported in the literature, the conversion to an equivalent axisymmetric solution is widely used and accepted [18]. Furthermore, in the axisymmetric geometry, only a half-sectional plane has to be considered in the design of the mesh. A very high Young's modulus (1100 GPa), a Poisson's ratio of 0.07 and a pressure-independent, isotropic behaviour were assumed for the diamond tip. Both the elastic and elasto-plastic responses, employing the conventional Tresca and the pressure-dependent Mohr–Coulomb yield criteria — with variable cohesive stress (c), friction coefficient (β_{MC}), and Young's modulus (E), were numerically calculated and the results compared with the experimental load–displacement indentation data. While variations in E modified the slope of the unloading curve, c and β_{MC} played a main role in the average slope and the curvature of the loading curve. The simulations also provided the stress and displacement contour mappings of the deformed region underneath the indentation imprint.

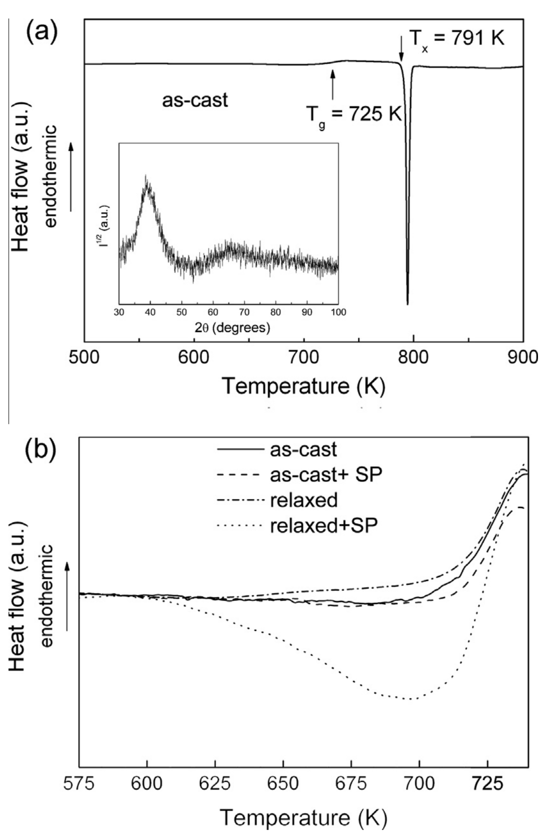

Fig. 1. (a) Differential scanning calorimetry (DSC) curve for as-cast $Cu_{47.5}Zr_{47.5}Al_5$ glass heated at 20 K/min. The inset shows the X-ray diffraction (XRD) pattern of the glass and (b) enlarged view of the DSC scans of the $Cu_{47.5}Zr_{47.5}Al_5$ in the as-cast state, after shot peening of the as-cast sample, after annealing for 5 min at 700 K (relaxed state), and after annealing-induced relaxation followed by shot peening.

3. Results and discussion

Fig. 1(a) shows the DSC scan corresponding to the as-cast $Cu_{47.5}Zr_{47.5}Al_5$ alloy. The curve reveals the existence of a broad exothermic halo in the temperature interval from 650 to 725 K, which corresponds to the enthalpy release during structural relaxation. For the heating rate of 20 K/min, the glass transition temperature is $T_g = 725$ K and the onset of crystallization occurs at $T_x = 791$ K. The relaxation and crystallization enthalpies are $\Delta H_{rel} = 9$ J/g and $\Delta H_{cryst} = 55$ J/g, respectively. The XRD pattern of the $Cu_{47.5}Zr_{47.5}Al_5$ alloy (inset in Fig. 1(a)) consists of broad halos with absence of well-defined peaks, indicating the amorphous character of the as-cast specimen. Analogous DSC curves with similar values of T_x and ΔH_{cryst} and slightly larger ΔH_{rel} are obtained after subjecting the as-cast alloy to shot peening (see Table 1). Short-term annealing at $T_{ANN} = 700$ K brings about a small decrease in ΔH_{cryst}, although no change in T_x is observed. Remarkably, ΔH_{rel} increases

Table 1
Summary of the values of relaxation enthalpy (ΔH_{rel}), crystallization enthalpy (ΔH_{cryst}) and temperature of the crystallization peak ($T_{x,p}$) obtained by differential scanning calorimetry, DSC, at 20 K/min, from the as-cast, as-cast + shot-peened, thermally relaxed, and relaxed + shot-peened samples. In all cases, DSC measurements were performed on thin slices obtained from the $Cu_{47.5}Zr_{47.5}Al_5$ sheets (for the SP samples, the slices were the top 100 μm of the shot-peened surfaces).

Sample	ΔH_{rel} (±2 J/g)	ΔH_{cryst} (±2 J/g)	T_x (±1 K)
$Cu_{47.5}Zr_{47.5}Al_5$ as-cast	9	55	791
$Cu_{47.5}Zr_{47.5}Al_5$ as-cast + SP	11	54	792
$Cu_{47.5}Zr_{47.5}Al_5$ annealed	8	51	790
$Cu_{47.5}Zr_{47.5}Al_5$ annealed + SP	18	55	789

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