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Mechanical spectroscopy study on the $Cu_{54}Zr_{40}Al_6$ amorphous matrix alloy at low temperature

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ABSTRACT

A mechanical spectroscopy study of $Cu_{54}Zr_{40}Al_6$ bulk metallic glasses composites was carried out in the kHz and MHz frequency ranges, by means of flexural and ultrasonic methods, respectively, in the temperature interval 150–300 K. In internal friction and attenuation curves at low temperature were observed peaks which were associated with distortions in the configuration of atomic clusters, which absorbed different quantities of energy due to short and medium order rearrangements. Changes within the clusters or atomic jumps between clusters occurring in the specimen induced the onset of polyamorphic peaks, since electronic interactions and bonding changed abruptly.

1. Introduction

In recent years, the study of multicomponent glass-forming alloys has been of great scientific and technological interest for their unique properties, which arise from the lack of long-range regularity in their atomic structure and their compositional homogeneity similar to the liquid state. These alloys show better mechanical properties, superior corrosion resistance and higher yield stress and fracture toughness than their crystalline counterparts [1–4]

Bulk metallic glasses (BMG) composites, produced by rapid solidification, usually exhibit a non-equilibrium structure formed by quasicrystals and/or metastable crystalline phases embedded in an amorphous matrix [5]. Under heating, below its crystallization temperature, the material undergoes an atomic rearrangement to a more stable state. This phenomenon is known as structural relaxation, which is manifested as a continuous change in some physical properties [6]. Published studies on the mechanical behavior of the BMGs at low temperatures are few and mostly concerned with mechanical testing (tensile and compressive tests) [7–9]. Very little work has been carried out on anelastic properties of BMGs at cryogenic temperatures [10–12].

Anelastic behavior can be studied by mechanical spectroscopy, which can be defined as an energy absorption technique, where waves of mechanical stress interact with the structure of the solid. The absorbed energy spectrum generated is commonly used to study interactions of defects and phase transformations in crystalline alloys [13,14]. Thus, in BMG, this technique can be very useful in the study of the transition from the glassy to crystalline state [15,16], changes in the structural order, as α or β relaxations [17–19], and vibrational characteristics which are inaccessible by other methods [4,20,21].

The literature reports that the mechanical deformations that can occur in the sample depends on the type of applied stress, thus the stress associated to mechanical vibration and waves on the sample may cause rotation or sliding of crystals and a variation in the potential energy between a pair of atoms in metallic materials whereas with transverse waves, a release and accumulation of deformation can be detected that is related to thermal relaxation of squeezed free volume, accompanied by an increase in atomic distance [11].

In this study, anelastic relaxation processes induced in a cast glassy alloy $Cu_{54}Zr_{40}Al_6$ with a very small nanocrystalline fraction were analyzed in terms of frequency, in the temperature range 150–300 K. Internal friction and attenuation were measured by the flexural method in the kHz frequency range and by the ultrasonic pulse-echo method in the MHz frequency range, respectively.

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The main interest was in the anelastic effects on the free volume and structural changes on BMG at cryogenic temperatures.

2. Experimental procedure

An amorphous matrix alloy with nominal composition of $Cu_{54}Zr_{40}Al_6$ was studied by mechanical spectroscopy. The criteria for choosing this material was the synergetic effect between minimal topological instability and average electronegativity difference ($\lambda_{min} \cdot \Delta\bar{e}$) of the elements in the Cu–Zr–Al system, since this criterion is related to the glass-forming ability (GFA) of metallic alloys and also to ranking in metastable phase formation [22–24].

A $Cu_{54}Zr_{40}Al_6$ sample was melted in a quartz nozzle with a high-frequency induction furnace and cast as a rod of 6 mm diameter and 30 mm length into a copper mold [25].

The phase analysis of the as cast sample, was performed by X-ray diffraction (XRD) using a Rigaku Geigerflex Diffractometer with Cu Kα radiation and the transmission electron microscopy (TEM) were performed with an acceleration voltage of 200 kV (Tecnai G^2 HR-TEM (FEI/Philips)). Thermal properties of the alloy was characterized by differential scanning calorimetry (DSC) in a Netzsch DSC 404, at a heating rate of 40 K/min. From the original rod, the samples were cut in appropriate formats for mechanical spectroscopy studies in different frequency ranges, in order to characterize the anelastic behavior over a wide interval of frequencies. The mechanical spectroscopy study was carried out by the flexural vibration method operating at kHz frequencies, and the ultrasonic pulse-echo method, at MHz frequencies. In the ultrasonic method, the wave mode can be selected in the measurements using X-cut or Y-cut ultrasonic transducers, whereas the flexural vibration method excites both transversal and longitudinal strains.

Flexural vibration was applied in clamped-free mode, exciting the first tone of a rectangular sample cut to size $16 \times 6 \times 0.7\ mm^3$, in an acoustic elastometer system (Vibran Technologies, AE 102 model). The operating details of this equipment were given in our previous paper [26]. The internal friction (Q^{-1}) was determined by the free decay of sample oscillations, δ, using the following equation $Q^{-1} = \delta/\pi$ [13].

For ultrasonic pulse-echo measurements, a cylindrical sample was cut of 9.84 mm height and 6 mm diameter. The measurement faces were mechanically polished to a parallelism of approximately 10^{-4} radians, necessary for accurate ultrasonic measurements [27]. The ultrasonic wave velocity through the sample was measured by the conventional pulse-echo technique [28]. A MATEC ultrasonic system was used with Y-cut quartz transducer of fundamental frequency 5 MHz, to generate transversal waves of 5 MHz. To bond the transducer to the measurement face of the sample, water-free oil with a low solidification temperature was used. The round trip time for the wave was accurately determined by the pulse-echo overlap technique (Papadakis) applied to the transit time due to diffraction [29]. Ultrasonic attenuation was obtained by conventionally monitoring two echoes, and diffraction corrections were also carried out. Measurements were taken at a frequency of 5 MHz, with transversal waves, while the material was cooled from room temperature to 150 K and then heated to 300 K, at a controlled rate of 1 K/min.

3. Results and discussions

3.1. Characterization of the as cast alloy

Fig. 1(a) shows the differential scanning calorimeter (DSC) traces of the $Cu_{54}Zr_{40}Al_6$ alloy obtained at a heating rate of 40 K/min. The material under study, a typical metal/metal alloy, has a high glass-forming ability with a large super-cooled liquid region (ΔT_x), showing glass transition temperature (T_g) at 730 K and onset crystallization temperature (T_x) at 807 K.

Fig. 1(b) displays the X-ray diffraction (XRD) pattern of the alloy which exhibits a broad diffuse peak characteristic of the amorphous structure without any visible peak corresponding to crystalline phases, this result is in good agreement with the TEM images and diffractions electron patterns. However, in the high resolution images (HRTEM) of the sample (Fig. 2) is observed a region with totally amorphous matrix (Fig. 2(a)) and other with composite-like structure (Fig. 2(b)). The regions indicated by arrows, in Fig 2(b), show small crystals embedded in amorphous matrix with average size about 2–4 nm. The inset of Fig. 2(b) shows the diffraction pattern of the region with crystal-like order. It is worth noting that size of crystals are very small and highly dispersed in the matrix, which lead to no observation in the XRD pattern.

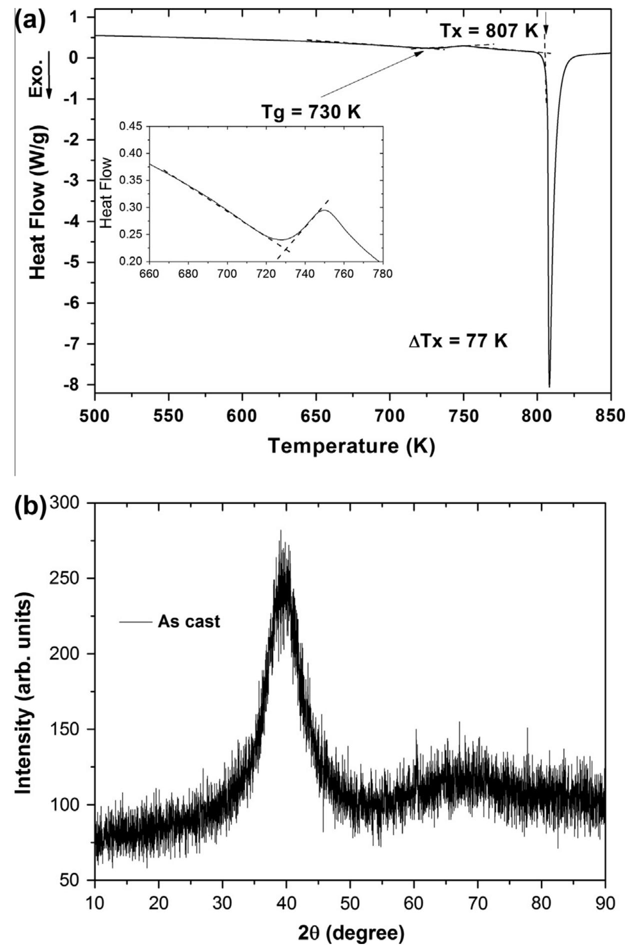

Fig. 1. (a) DSC thermogram obtained at a heating rate of 40 K/min and, (b) X-ray diffraction pattern of $Cu_{54}Zr_{40}Al_6$ alloy in as cast condition.

3.2. Characterization in kHz frequency range by flexural vibrations

Fig. 3 shows the changes in the anelastic relaxation spectra observed by flexural measurements at kHz bandwidth frequency, in the temperature range 150–300 K. The thermal cycle shows changes occurring in the internal friction (Q^{-1}) spectrum of the sample (Fig. 3(b)). During the cooling, the internal friction shows an anomaly in the form of a very broad peak, which begins at 276 K and finishes around 175 K. Superimposed to this broad anomaly two evident sharp peaks are observed at 163 K and 186 K, here and after namely peak 1 and 2, respectively. It is interesting to note that a slight anomaly in the frequency curve (Fig. 3(a)) is present at the temperature of peak 2. During the initial heating, the internal friction decreases, the peak 1 disappear while the peak 2 appears again but with less intensity. The broad peak apparently shifts to a higher temperature, or is reduced in the intensity and thus not observed.

The flattening of the broad peak observed in the heating arm of the thermal cycle in Fig. 3(b) and the higher frequency observed in the heating run (Fig. 3(a)), could indicate that a reduction in atomic mobility and a consequent reduction in the interatomic distance between the clusters at low temperature is produced during the cooling run. The contraction of the clusters results in a redistribution of the free volume, leading to changes in the spectra. Peaks 1 and 2 may be related to changes (relaxation process) that probably occur within some specific cluster, probably involving Cu or Zr elements in its shell, since mechanical waves promote strong changes in the bonding forces among the constituent elements that lead to

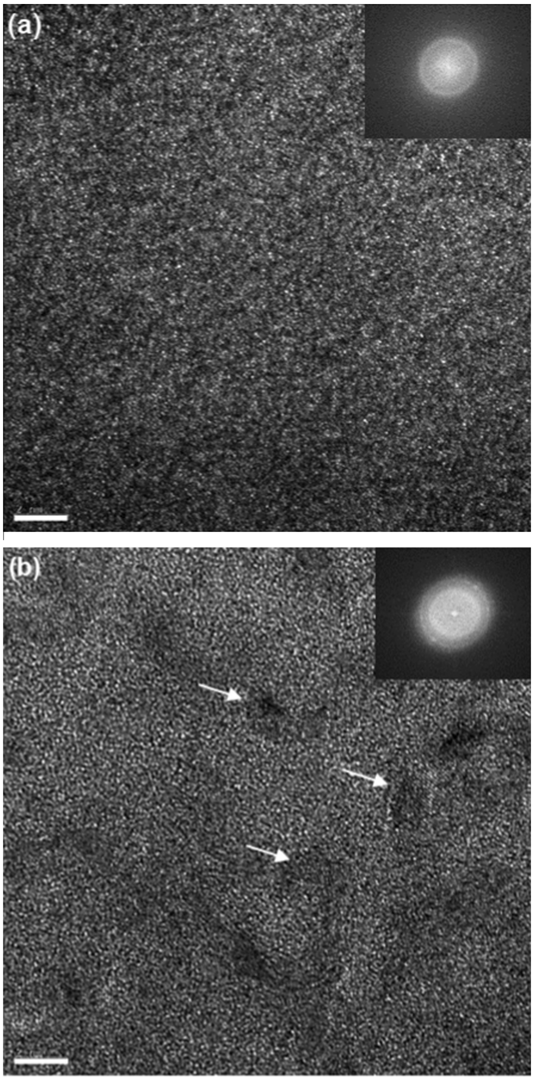

Fig. 2. (a) HRTEM image from the fully amorphous region and, (b) HRTEM image from composites-like region, on the inset figures shows the Fourier transformation pattern. (scale bar 2 nm).

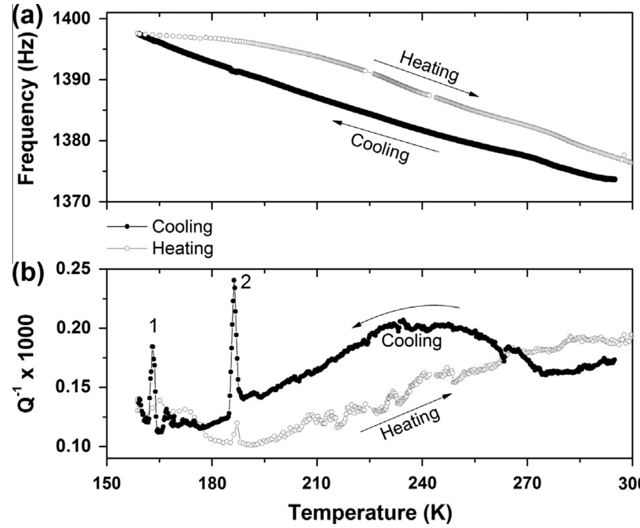

Fig. 3. Changes in the anelastic spectra that occur in the sample $Cu_{54}Zr_{40}Al_6$. (a) Behavior of the frequency during a cooling-heating cycle. (b) Internal friction curve showing a broad peak during the cooling, followed by intense peaks around 163 K and 186 K.

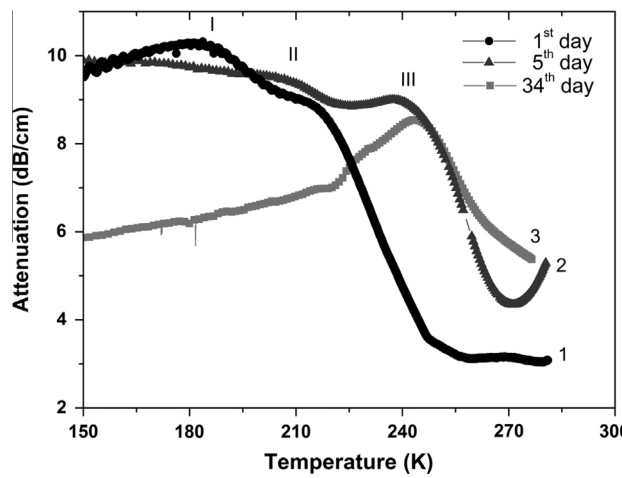

Fig. 4. Spectra of transversal wave attenuation of 5 MHz, plotted against temperature during three heating runs (1 K/min). The first run shows a broad peak near 180 K that is lower in next run, possibly due to interactions caused by atomic rearrangement. The peak at 238 K in the second run shifts to 245 K in the third.

the sharp increase in internal friction. Internal friction changes during heating are expected in BMG alloys, since the migration of free volume induces changes in the sample, leading to more stable states [26,30]. During the thermal cycle, it is suggested that both the creation and annihilation of free volume occurs and can be followed by rotation some types of clusters, causing hysteresis in the frequency (Fig. 3(a)).

3.3. Characterization in MHz frequency range by ultrasonic attenuation

Fig. 4 shows attenuation spectra collected with transversal ultrasonic waves from a Y-cut transducer at 5 MHz, while heating the sample from 150 K to 300 K. Three measurement runs were carried out: (1) as received, (2) 5 days after the first measurement run and (3) 34 days after the first run. Between measurements, the sample was kept at ambient pressure and temperature, free of stress. Run 1 shows a broad peak (I) near 180 K and a shoulder around 215 K (II). Peak (I) becomes flatter in the second (2) measurement run and disappears in the third (3). In run 2, a reduction is seen in the intensity of peak (II) and a new peak (III) of attenuation emerges around 238 K. Peak (III) is more evident in run 3 and shifts to 245 K, while peak (II) disappears. These changes in the attenuation spectra may be caused by interactions due to atomic rearrangement, similar to the broad peak observed in the internal friction measurements.

For first measurement, the attenuation starts to rise from approximately 220 K, showing a broad peak at higher temperatures, which seems composed of at least two major peaks centered at approximately 183 K and 218 K, respectively. It is interesting to note in Fig. 4 the existence of a peak at approximately 245 K (namely peak III), which appears after the first cycle of measurement and remains almost constant in shape and intensity in the following cycles. At the same time, the peaks I and II become weakly if compared with the first cycle. This different behavior between peaks I and II and peak III is significant, and will be analyzed in the next section comparing them with those observed in low frequencies.

In order to compare the microstructure of amorphous sample before and after mechanical spectroscopy measurements, Fig. 5

shows the TEM images obtained after anelastic studies, which present in bright (a) and dark field (b) images.

From Fig. 5 it is observed that mechanical measurements at low temperature lead to changes in the microstructure of the sample. In this figure is observed an increase in both the size and the volumetric fraction of nanocrystals in comparison with the sample of the as cast condition (Fig. 2). The average size of nanocrystals, after mechanical measurements, was is about 20 nm. The selected area electron diffraction pattern (SAED) in the inset at Fig. 5(b) shows rings of the $Cu_{10}Zr_7$ and small spots of the CuZr nanocrystals, respectively.

3.4. Relations between anelastic and attenuation spectra

The peaks at 163 K and 186 K in flexural measurements (sharp~~s~~ peaks 1 and 2 in Fig. 3(b)) corresponds the peaks at 186 K and 220 K in attenuation, respectively (peaks I and II in Fig. 4). These peaks, may be connected with the same process, they are not only due to defects induced by strain, but can be associated with polyamorphic changes [31] that are more evident at kHz frequencies. These peaks indicates changes in structure due to a sudden break in atomic bonds or the rotation, followed by a rapid rearrangement of a cluster that undergoes irreversible changes when exposed to frequencies in both kHz and MHz range [11]. These peaks may depend on the density, distribution and extent of clusters that interact with the mechanical waves and can be related to thermally activated rearrangement of atoms caused by the external elastic field (strain), as described by Bakai et al. [32].

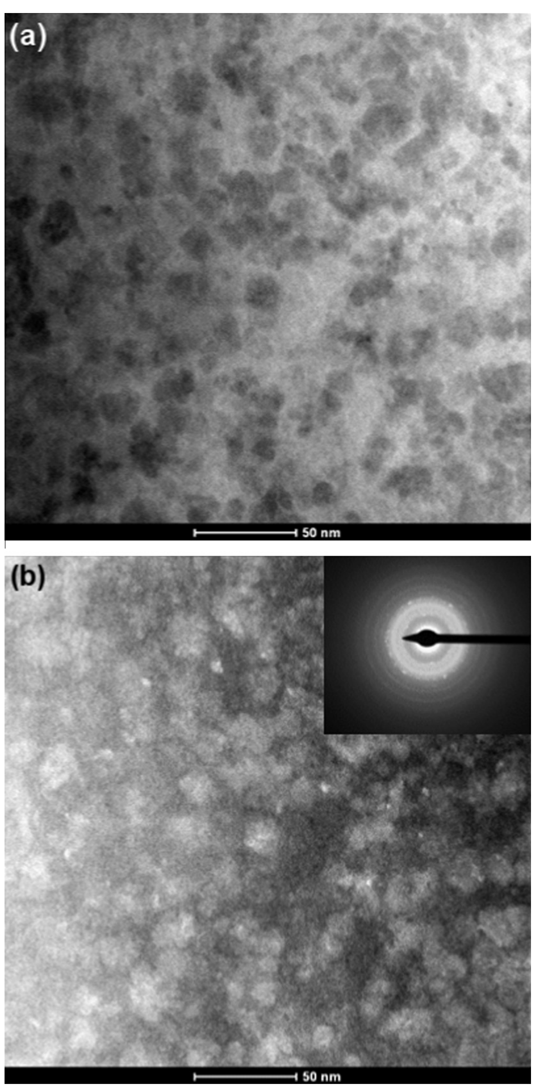

Fig. 5. (a) BF-TEM and (b) DF-TEM image, after mechanical measurements, with the SAED pattern from the nanocrystals in the inset.

Correlating the observed peaks at 1 and 2 , for kHz frequencies, and I and II, for MHz ones for the $Cu_{54}Zr_{40}Al_6$ amorphous alloy, the underlying mechanisms has an activation energy of $\sim$0.85 eV and a difference of $\sim$30 K between peak temperatures at both different frequencies. This behavior, observed in flexural and transversal waves in the studied sample, appears as being similar to some peaks described in the literature for samples of slight different composition [10].

Studies carried out by Lekka et al. [33,34], involving density functional theory simulation in Cu–Zr–Al alloys showed that the higher energy states were due to Cu–Cu atom bonding while the Zr–Zr interactions become to be close to the Fermi level. Peaks I and II in Fig. 4 (peaks 1 and 2 in Fig. 3(b)) could be associated with changes that occur due to annihilation of the less stable clusters, possibly those surrounded by Zr or Cu atoms in the nearest-neighbor shell, and the annihilation and recreation of these structures leads to considerable reduction of these peaks from the first to the third run in attenuation.

In this study is suggested that the narrow peaks, shown in Fig. 3(b) and peaks I and II in Fig. 4, are associated with changes in both the electronic and atomic configuration of the clusters which was stabilized by random short and medium-range rearrangement during each measurement, probably involving Cu and Zr atoms. These changes may lead to formation of new structures, since clusters can be interpenetrating forming supercluster [33,34].

In the literature, it is reported that some amorphous materials exhibit an arrangement of octahedral clusters around a nanocrystalline structure [35] then, during thermal cycles, it is suggested that this arrangement can lead to relaxation processes in which interatomic changes within the clusters or atomic jumps between clusters can occur. The orbital hybridization between the electrons of Al(*sp*) and Cu(*d*) causes the polarization and shortening of interatomic bonds [36] and when the material is excited by mechanical energy, in the MHz range, this can be favors the formation of new Cu-centered and Al-centered icosahedral structures which may be indicates a rearrangement of this structures around 245 K which is evidenced by the arise of peak III in the attenuation spectrum. This behavior may occur in these specimens, inducing the appearance of peaks that can be related to polyamorphic transformation [31], where electronic interactions change abruptly in the MHz bandwidth frequencies. The peaks seen at kHz frequencies have too low energy and have small contribution to form new icosahedral structures, but cause irreversible distortions in some types of cluster leading to a reduction in internal friction.

TEM images shown in Fig. 5, reinforce these transformations that occur in the sample, after mechanical measurements at low temperatures, indicating an increase in the size and number of nanocrystals.

4. Conclusions

$Cu_{54}Zr_{40}Al_6$ bulk metallic glasses were studied by mechanical spectroscopy, employing the flexural (kHz) and ultrasonic (MHz) methods. The anelastic spectra changes at each measurement run for both methods. The lower temperature peaks were associated with the change in the configuration of the atomic clusters which absorbed a different quantity of energy after the random rearrangement of short and medium-range order during each measurement. The relaxation mechanisms of the peaks at higher temperatures are related to interatomic changes within the clusters or atomic jumps between clusters, due to the electron orbital hybridization between Al (*sp*) and Cu (*d*). In addition, this behavior can be intensified by mechanical squeezing applied to the specimen, indicating the appearance of polyamorphic peaks, since electronic interactions and bonding starting to change in the kHz range and are intensified at MHz frequency.

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