Influence of structural relaxation on atomic mobility in a $Zr_{41.2}Ti_{13.8}Cu_{125}Ni_{10.0}Be_{22.5}$ (Vit1) bulk metallic glass

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ABSTRACT

The effect of an annealing at a temperature above or below the glass transition temperature in a $Zr_{41.2}Ti_{13.8}Cu_{125}Ni_{10.0}Be_{22.5}$ bulk metallic glass was investigated using dynamic mechanical analysis. Structural relaxation influences both the storage modulus (elastic component) and the loss modulus (viscoelastic component). Kinetics can be captured by a stretched exponential relaxation function. Experimental results are correctly described using a physical model based on the concept of defects for the mechanical response of amorphous materials and especially for the characteristic time relative to atomic mobility.

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1. Introduction

Mechanical properties of bulk metallic glasses have been the subject of many articles during the last decades [1–17]. At high temperature a homogeneous viscous flow is observed, while at room temperature a localized deformation occurs, with the formation of shear bands. In any case the microstructure of the alloy is a key parameter. Depending on the alloy composition, but also on the thermo-mechanical history of the alloy, very different behaviors have been reported. Nowadays, a lot of studies concern the possibility to reduce initiation or propagation of shear bands, in order to reduce the brittleness of these materials, for instance by introducing 'hard points' which can deviate or even stop the propagation [18–21]. Furthermore, since bulk metallic glasses are thermodynamically out of equilibrium, with a disorder which has been blocked during the rapid cooling, an excess of Gibbs energy exists

and annealing can cause a gradual approach towards their equilibrium state. The ultimate state is the crystalline state, but when annealing is performed below the glass transition temperature T_g, a slight evolution, called structural relaxation is first detected. Structural relaxation induces only local rearrangements and the non-crystalline state is maintained. As it will discussed in the present paper this relaxation induces a decrease of the free volume or of the number of defects (depending on the retained model) existing in the alloy. This reduction limits the atomic diffusion, or in other words the atomic mobility. Due to these local atomic rearrangements, many properties are modified. Some amorphous materials become more brittle, while some soft magnetic properties can be improved. In order to follow this progressive evolution, many techniques can be used: differential scanning calorimetry (DSC), transmission electron microscopy (TEM) or small angle X-ray spectroscopy (SAXS). Wang et al. [22] have shown that during heating, enthalpy can overshoot near the glass transition temperature, as in many other amorphous materials, like polymers. This enthalpy increase ΔH depends on annealing time t_a at a given

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annealing temperature T_a. Wang et al. [22] have proved that this time dependence can be well fitted by a stretched exponential relaxation function, with a Kohlrausch exponent lower than 1. Structural relaxation can increase the shear viscosity by order of magnitude. Furthermore, the current literature indicates that this evolution contain both reversible and irreversible components [23–26]. For instance Bobrov et al. [23] have reported that in the $Pd_{40}Cu_{30}Ni_{10}P_{20}$ metallic glass ability to undergo homogeneous flow during creep decreases strongly as a result of structural relaxation, but part of this decrease can be recovered by a further annealing in the glass transition region.

One of the techniques which is very sensitive to any microstructure evolution is the dynamic mechanical analysis (DMA), which is also sometimes mechanical spectroscopy [27–34]. A low level periodic stress is applied, with a given frequency (f) and the resulting stain is recorded. Both the magnitude of this strain and the phase lag between stress and strain are measured. When a low stress level is applied, only reversible components of the deformation are activated: elastic (instantaneous) and viscoelastic (also called anelastic) (delayed). Any evolution of atomic mobility yields a variation of the relative magnitude of these two components.

Thus, the aim of this paper is to investigate the influence of structural relaxation on the atomic mobility by performing DMA experiments. Structural relaxation is induced by annealing treatments conducted below T_g. The studied alloy is the classical Vit1, well known due to its exceptional glass forming ability and its thermal stability [35–38].

2. Experimental procedure

The investigated BMG $Zr_{41.2}Ti_{13.8}Cu_{125}Ni_{10.0}Be_{22.5}$ alloy obtained in the form of a 3 mm thick plate is supplied by Howmet Corp (USA). The amorphous nature of this alloy is confirmed by X-ray diffractometry (XRD) with $CuK\alpha$ radiation.

Thermal stability of this metallic glass was examined by differential scanning calorimetry (Perkin–Elmer DSC-7) at a heating rate of 3 K/min under an argon atmosphere. The DSC curve for this alloy exhibits a distinct glass transition, followed by a wide supercooled liquid region before crystallization at higher temperatures. Glass transition temperature (T_g), onset crystallization temperature (T_x) and extend of the supercooled liquid region ($\Delta T_x = T_x - T_g$) are 650 K, 729 K and 79 K, respectively. Isothermal measurements have also been carried out.

Dynamic mechanical measurements were carried out in inverted torsion mode using a mechanical spectrometer described by Etienne et al. [39]. In experiments performed under sine sinusoidal stress, the relationship between stress and strain can be written as follows: $G^* = G' + iG''$, where G' and G'' are the storage and loss modulus, respectively. Internal friction or loss factor, defined as $\tan\delta = G''/G'$, gives information on the atomic mobility and its variation with structural evolution. Measurements were performed as a function of frequency in the range from 10^{-4} to 1 Hz with a resolution in $\tan\delta$ better than 2×10^{-4}. The specimens used had dimensions of around $40 * 4 * 1$ mm^3. All measurements were performed in high vacuum. Measurements of the dynamic properties have first been carried out with a constant driving frequency of 0.3 Hz and a constant heating rate of 3 K/min or vs time at a given temperature.

3. Results

3.1. Thermal stability detected by DMA

Fig. 1 shows the relative temperature dependence of the storage modulus G'/G_u and the loss factor $\tan\delta$ when a constant frequency
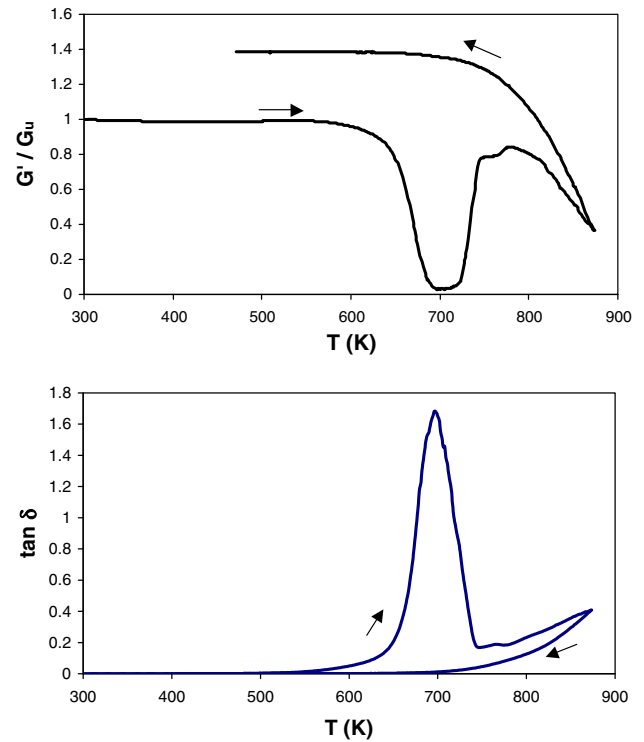

Fig. 1. Temperature dependence of storage modulus, G'/G_u, and loss factor, $\tan\delta$, during continuous heating. Frequency: 0.3 Hz; heating rate: 3 K/min.

(0.3 Hz) and a constant frequency (0.3 Hz) are used. Curves related to modulus have been normalized in order to enable a better comparison (G_u is the storage modulus for the as-received sample at room temperature). During heating, a very large increase of the loss factor is observed above about 573 K and very large values of this parameter are recorded in the supercooled liquid region, where the storage modulus is very low. Crystallization induces a decrease of the loss factor and an increase of G', due to the decrease of atomic mobility [33].

3.2. Evolution during isothermal annealing

Isothermal experiments were carried out in a large temperature range (573–683 K), both below or above the glass transition temperature ($T_g = 650$ K), in order to detect both structural relaxation and onset of crystallization. Fig. 2 displays this evolution during annealing at various temperatures. $G'(t = 0)$ corresponds to the initial value of G' at the corresponding annealing temperature. Kinet-
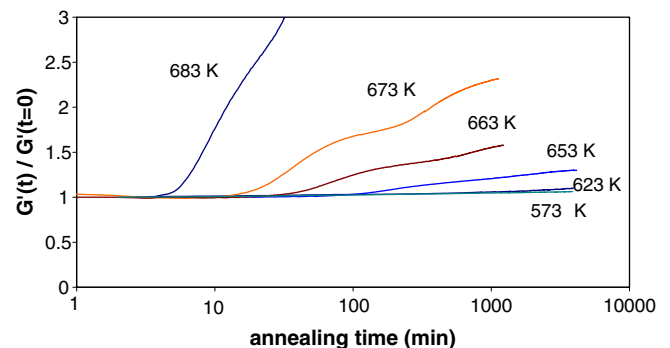

Fig. 2. Relative variation of the storage modulus vs annealing time at various temperatures.

ics and magnitudes are strongly temperature dependent and different stages appear. Fig. 3 illustrates in a different way this evolution during an annealing at 646 K, with an expanded scaling. The slow initial evolution of both G'/G_u (with $G_u = G'(T = 293$ K)) and $\tan\delta$ corresponds to the structural evolution, while after about 1500 s the rapid evolution can be associated to the onset of crystallization, in agreement with previously reported data. [33].

Let us now investigate in more details the kinetics of the structural relaxation. To avoid this crystallization phenomenon, it is better to conduct annealing at lower temperatures, for instance 573 K. Let us consider the parameter:

$$\Delta = \frac{\tan\delta(t) - \tan\delta(t \to \infty)}{\tan\delta(t = 0) - \tan\delta(t \to \infty)}$$

and its variation vs annealing time t_a. Fig. 4 indicates that a plot of $\ln(\ln(-\Delta))$ vs $\ln(t_a)$ is a straight line. Therefore the dependence of the loss factor on time can be well fitted by a stretched exponential relaxation function

$$\tan\delta(t) - \tan\delta(t = 0) = A\left(1 - \exp\left[-\left(\frac{t}{\tau}\right)^{\beta}\right]\right)$$

where A is the maximum magnitude of the relaxation, τ is the characteristics structural relaxation time, β is the Kohlrausch exponent whose value is between 0 and 1, reflecting a broad distribution of relaxation time and not a single Debye relaxation time. From this plot we deduce: $\beta = 0.61$. These results are in very good agreement with those reported by Wang et al. in an other Zr-base bulk metallic glass [22]. They observed that the enthalpy recovery during annealing was well captured by a similar expression. Agreement concerns not only the qualitative description, but also the quantitative value. Indeed, they obtained also $\beta = 0.61$. Therefore this kind of evolution appears like a general phenomenon in bulk metallic glasses. More generally it has been recognized in the literature that the glass transition regime of supercooled fluids is characterized by a number of typical kinetic phenomena. For instance, in his 1983 paper, Hodge [40] interpreted
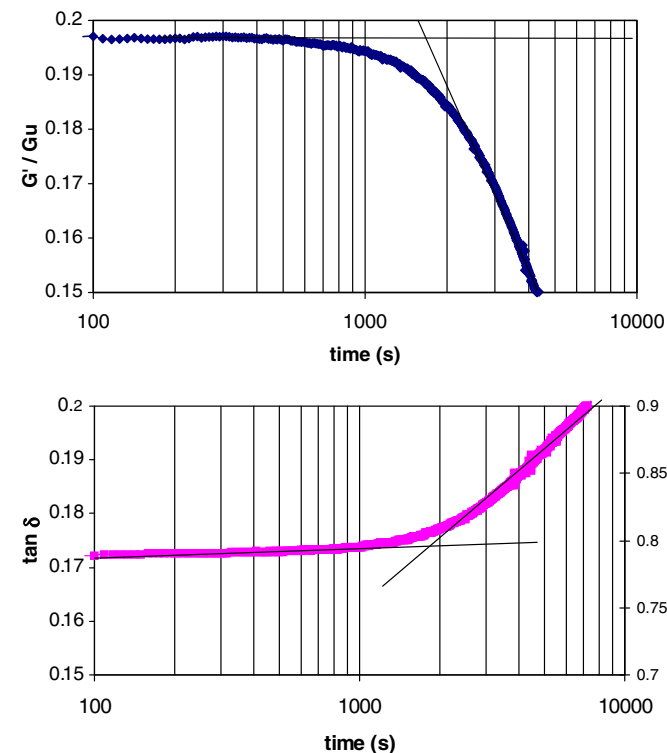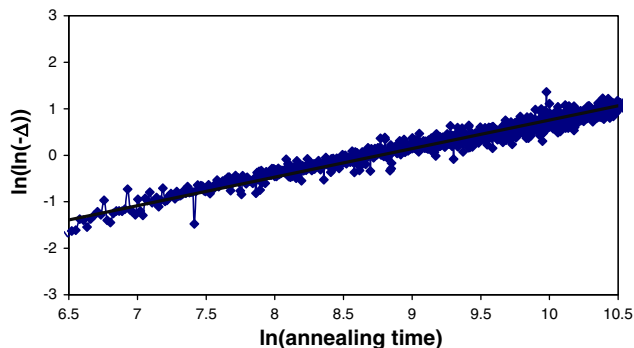

Fig. 4. (a) Double logarithmic plot of the variation of $\tan\delta$ vs logarithm of the annealing time at 573 K.

the nonexponentiality as being a consequence of cooperativity in the motion of the rearranging structural units within the glass former. Böhmer et al. [41] reported a very interesting review on nonexponential relaxations in about 70 strong and fragile glass formers. A lot of experimental methods have been used to determine the coefficient β. These methods include dielectric and specific heat spectroscopies, viscoelastic modulus measurements in the shear and tensile modes as well as shear compliance investigations, quasi-elastic light scattering experiments and others. Numerous theoretical approaches have been developed to find a physical meaning to this parameter. But this is still an open question and the microscopic interpretation of the stretching parameter β varies from one theory to another. Experimental values of β are in the range (0.24–1). For polymers, values extend from 0.24 (PVC) to 0.55 (polyisobutylene), for alcohols from 0.45 to 0.75, while for orientational glasses and networks values are up to 1 (for GeO$_2$). Therefore, bulk, metallic glasses appear to be in the middle of this range. Furthermore this review by Böhmer et al. [41] provides strong evidence for a general correlation of the nonexponentiality of the structural response (quantified using this parameter β) with the fragility. This last parameter proposed by Angell [42] is directly connected to the viscosity variation in the glass transition temperature range. Bulk metallic glasses are known to intermediate materials, between strong materials (like oxide glasses) and fragile materials (like polymers) [43]. As a conclusion, present results are in good agreement with a lot of results observed in various amorphous materials: polymers, organic liquids, oxide glasses.

3.3. Reversibility and irreversibility of the phenomenon

As shown in the previous section, a thermal treatment performed at a temperature lower than the glass transition one induces a significant evolution of both modulus and loss factor. But the question is as follows: are these variations reversible or not? To get an answer, series of thermal treatment have been conducted, as indicated in Fig. 5. First a continuous heating up to 683 K ($T > T_g$) with a fixed heating rate (3 K min^{-1}) is carried out, then a rapid cooling, a subsequent annealing at 623 K ($T < T_g$), during 30 min, a rapid cooling and, finally, a second continuous heating up to 683 K. Fig. 5 displays the results. Annealing at 623 K induces a storage modulus increase and a loss factor decrease. However the differences disappear as soon as a sufficient temperature is reached, schematically as soon as the glass transition temperature is obtained. Thus, the evolution induced by the structural relaxation is reversible, at least for a major part.

In addition, even if this point is not shown on this figure, it is observed that evolution of G', G'' or $\tan\delta$ vs temperature beyond 683 K is not modified by an annealing performed at 623 K. In other words, crystallization kinetics during a continuous heating is not affected by a structural relaxation, since this relaxation is canceled
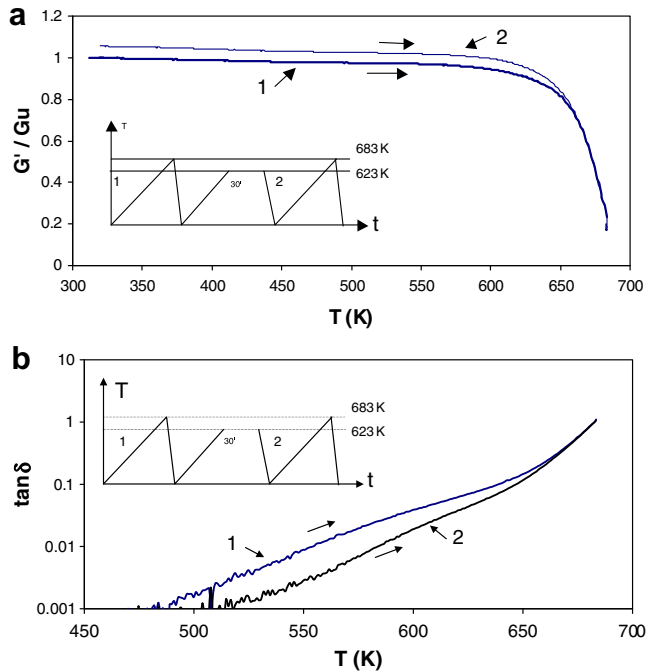

Fig. 3. Storage modulus and loss factor evolution vs annealing time at 646 K.

Fig. 5. Temperature dependence of (a) storage modulus and (b) loss factor during successive continuous heatings.

when the specimen goes through the glass transition temperature range, before the onset of this crystallization process.

4. Discussion

Experimental results indicate that aging at a temperature below the glass transition one induces a reversible evolution of both elastic and viscoelastic features of the bulk metallic glass. Structural relaxation enhances the elastic component and decreases the viscoelastic one. Thus, a decrease of the atomic mobility is induced by this treatment. This mobility in amorphous materials, which is connected to the viscoelasticity, has been the subject of many developments in the literature. To understand the relaxation mechanism various theoretical models, such as free volume theory [43–47], Adam and Gibbs theory [48], coupling model [49–50], thermodynamic approach [51], energy landscape sampling [52] or mode coupling theory [53] have been proposed. They relate the complex phenomenology to molecular-scale events. Recently Langer et al. [4] have introduced in bulk metallic glasses the concept of effective disorder temperature and they have used this parameter as an essential dynamical state variables. This concept was originally introduced in the non-equilibrium theory of non-crystalline materials and has emerged in the physics literature and was also developed to explain for instance the Newtonian–non-Newtonian transition observed in the viscosity of oxide glasses [54]. However, many authors used the concept of free volume or defects in amorphous materials. Local transformations (diffusion, shear,...) are nucleated under the applied temperature gradient or the applied stress and the assistance of thermal fluctuations in regions around the free volume sites of the glassy structure. Granato [55] claimed that instead of using the free volume concept, all the phenomena can be easily explained using the interstitial model. Argon et al. [56] and Perez et al. [57–61] proposed to introduce a more general concept of defect: either the flow defect [46,56] or the quasi-point defect [57–61]. In these theories, microregions with density nanofluctuations in amorphous materials are associated with an excess of enthalpy and entropy and are called

defects. Their existence has been indirectly demonstrated through measurements of density fluctuations by small angle Synchrotron X-ray scattering [61–62]. It is believed that the molecular movements within these defects, which are responsible for the main mechanical relaxation, are hierarchically correlated, but related to the elementary molecular motion. The defect concentration is temperature dependent and is given by the statistics of Boltzman. Furthermore, the interest of this approach is to relate the degree of cooperativity, given by a parameter χ, to the concentration of enthalpy fluctuations at a nanometric scale (C_d) in the glassy system. This concentration depends on material, temperature and thermal history. By using this model Perez et al. [59] have demonstrated that the loss factor can be expressed by the following equation:

$$\ln(\tan \delta) = \frac{U_\beta}{kT} - \chi \cdot \ln(\omega) - \chi \cdot \ln(\tau^*) - \ln(\delta),$$

where U_β is the apparent activation energy for the elementary atomic movement, k the Boltzman constant, T the absolute temperature, ω the angular frequency, τ^* and δ are constant values. χ is the correlation factor, which traduces the interdependency of the different movements, which are assumed to be hierarchically correlated, according to the model initially proposed by Palmer et al. [63]. χ is between 0 and 1: $\chi = 0$ corresponds to the maximum degree of order (i.e. the perfect solid), in which any atomic movement required the movement of all the atoms in the specimen, while $\chi = 1$ corresponds to the maximum degree of disorder (i.e. the perfect gas), when all the movements are independent.

Thus, for a given temperature and a given frequency, the loss factor is directly connected to this correlation factor, i.e. to the state of order. Therefore, evolution shown in Fig. 5 can be easily understood: the structural relaxation induces a decrease of the loss factor since it produces a decrease of the defect concentration and then an increase of the local order. This reversible change could be due to short range chemical or topological ordering. A lot of experiments (e.g. electrostatic levitation and calorimetric methods, small neutron angle scattering, small angle X-ray scattering experiments, thermoelectric power measurements) have shown that in Vit1 the primary crystallization is preceded by phase separation [25,64–66]. This phase separation results in two supercooled liquid phases with two different compositions. During this decomposition, diffusion occurs, leading to a new short range order. Wang et al. [22] have shown that the decomposition has significant effect on the chemical and topological configuration of the amorphous alloy and, at least, Be and Zr arrangements are largely rearranged after the phase separation and that the decomposed two amorphous phases have different short range order. Van de Mortèle et al. [26] also observed, by TEM and nanoanalysis, that one of the phases is Zr and Be rich. Concerning the type of decomposition, Schneider et al. [62] indicated that the phase separation process shows many features of a spinodal decomposition, with a spinodal temperature of 671 K. This critical temperature is close to the value that we observe in Fig. 5: the loss factor evolution vs temperature merge nearly in this temperature range. So it could be assumed that part of the evolution observed during structural relaxation is correlated to this decomposition. Furthermore, as pointed out by many researchers, nanoparticle toughening is one potential direction to improve mechanical properties of bulk metallic glasses. So, understanding the structural evolution during devitrification is of essential significance for this purpose, and then, different investigations have been reported in the literature on the onset of crystallization, in many alloys. For instance, Fan et al. [67] have shown in $Zr_{60}Cu_{20}Pd_{10}Al_{10}$ that the small angle X-ray scattering shows increased periodic composition fluctuations before crystallization, providing evidence for the formation of (Zr–Pd) rich domains of short-range order in the liquid. Evidence for decomposition was also obtained in $Zr_{52.5}Cu_{17.9}Ni_{14.6}Al_{10}Ti_5$ bulk metallic glass [68].

In contrast, in $Cu_{43}Zr_{43}Al_{l7}Ag_7$, crystallization is a single step phase transformation [69,70], while in $Mg_{65}Cu_{25}Tb_{10}$, crystallization proceeds in three stages [71]. During stage I, icosahedral clusters are formed that transforms to a quasi-crystalline approximant during stage II and finally the orthorhombic Mg_2Cu phase is obtained. Consequently the microstructure evolution occurring in bulk metallic glasses during annealing at high temperature is a complex phenomenon, and its relation with a preliminary structural relaxation has to be investigated in details. Small angle scattering experiments have to be conducted. They are now in progress.

5. Conclusion

Atomic mobility in the investigated bulk metallic glass (Vit1) is strongly affected by annealing treatments. Dynamic mechanical analysis shows that the structural relaxation induced by these treatments increases the storage modulus but decreases the loss modulus and the loss factor. During annealing at a given temperature, the kinetics of loss factor evolution, which is directly connected to this atomic mobility, is correctly fitted by a stretched exponential relaxation function, which a Kohlrausch exponent close to 0.6. This evolution is reversible and has no clear influence on the further kinetics of crystallization.

Physical models based on the concept of defects give a good description of these phenomena.

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