



Mechanical Relaxation of a $\text{Ti}_{36.2}\text{Zr}_{30.3}\text{Cu}_{8.3}\text{Fe}_4\text{Be}_{21.2}$ Bulk Metallic Glass: Experiments and Theoretical Analysis

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Abstract

The dynamic mechanical relaxation behavior of $\text{Ti}_{36.2}\text{Zr}_{30.3}\text{Cu}_{8.3}\text{Fe}_4\text{Be}_{21.2}$ bulk metallic glass with good glass-forming ability was investigated by mechanical spectroscopy. The mechanical relaxation behavior was analyzed in the framework of quasi-point defects model. The experimental results demonstrate that the atomic mobility of the metallic glass is closely associated with the correlation factor χ . The physical aging below the glass transition temperature T_g shows a non-Debye relaxation behavior, which could be well described by stretched Kohlrausch exponential equation. The Kohlrausch exponent β_{aging} reflects the dynamic heterogeneities of the metallic glass. Both concentration of “defects” and atomic mobility decrease caused by the in situ successive heating during the mechanical spectroscopy experiments.

Keywords Metallic glasses · Mechanical relaxation · Physical aging · Quasi-point defects

1 Introduction

Bulk metallic glasses have attracted extensive investigations for their excellent mechanical and physical properties due to their unique disordered atomic structure [1–3]. In contrast to the conventional amorphous materials (i.e., polymers or oxide glasses, etc.), metallic glasses have a relatively simple atomic packing structure, making them an ideal carrier for studying the origin of the amorphous nature and the phenomenon of glass transition [4, 5]. As metastable materials, the physical process approaching to the stable state always occurs in metallic glasses and then the structural relaxation (i.e., physical aging) becomes one of the most fundamental

issues in metallic glasses [5–7]. However, how to describe the mechanism of mechanical relaxations in metallic glasses by suitable models with clear physical meaning is a challenging issue in glassy materials [8–10]? Mechanical relaxation processes (i.e., secondary β relaxation and main α relaxation) are closely associated with glass transition, diffusion phenomenon and mechanical properties in metallic glasses [5]. Dynamic mechanical analysis is an effective testing tool to study the internal atomic arrangement and microscopic mechanism of mechanical behaviors for amorphous materials [2, 11–13].

Compared with other typical metallic glasses, Ti-based metallic glasses show lower Young’s moduli and good corrosion behavior [14]. Interestingly, Ti-based metallic glasses show formation of a metastable phase after the first crystallization process [15]. As the candidate as application of engineering materials, to well realize the physical and mechanical properties of metallic glass, dynamic mechanical relaxation behavior could be investigated [16, 17].

In the current study, the dynamic mechanical relaxation of the $\text{Ti}_{36.2}\text{Zr}_{30.3}\text{Cu}_{8.3}\text{Fe}_4\text{Be}_{21.2}$ (at.%) bulk metallic glass was investigated, which possesses exceptionally high glass-forming ability (GFA) and remarkable compressive plasticity as reported in the previous literature [18]. Based on the experimental results and theoretical analysis, the relationship between the dynamic mechanical relaxation

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and the atomic mobility as well as the “defects” in metallic glass was established.

2 Experimental Procedure

The sample of $\text{Ti}_{36.2}\text{Zr}_{30.3}\text{Cu}_{8.3}\text{Fe}_4\text{Be}_{21.2}$ (at.%) metallic glass was prepared by arc melting under a Ti-gettered argon atmosphere. The purities of all the elements are higher than 99.6%, and each ingot was re-melted more than five times to ensure the chemical homogeneity. The samples of plate with a dimension of $3\text{ mm} \times 10\text{ mm} \times 80\text{ mm}$ were fabricated by suction casting into a water-cooled copper mold. The amorphous characteristics of specimens were then characterized by X-ray diffraction (XRD, D8, Bruker AXS GmbH, Germany) with $\text{CuK}\alpha$ radiation. As for the thermal properties, both the glass transition temperature T_g and the onset temperature of crystallization T_x of the specimens were measured by differential scanning calorimeter (DSC, Pekin Elmer, DSC-7) under a constant heating rate of 20 K/min. The dynamic mechanical behavior of $\text{Ti}_{36.2}\text{Zr}_{30.3}\text{Cu}_{8.3}\text{Fe}_4\text{Be}_{21.2}$ bulk metallic glass was studied by the commercial dynamic mechanical analysis (DMA, TA Q800) under the single cantilever mode. The specimens for the DMA experiments were prepared by using a low-speed cutting machine with water cooling and then polished surfaces carefully. Typical specimen dimension of the Ti-based metallic glass is approximately $1\text{ mm} \times 3\text{ mm} \times 30\text{ mm}$. A sinusoidal stress was applied on the samples and then by measuring the corresponding strain of the specimens during the DMA experiments, the storage modulus E' and loss modulus E'' as well as the internal friction (mechanical loss) $\tan\delta = E''/E'$ could be obtained.

3 Results and Discussion

3.1 Structural and Thermal Properties

As demonstrated in previous literature [18], $\text{Ti}_{36.2}\text{Zr}_{30.3}\text{Cu}_{8.3}\text{Fe}_4\text{Be}_{21.2}$ bulk metallic glass processes exceptionally high GFA, and the amorphous structure could be obtained by naturally cooling with an ingot of about 150 g. In the current work, the amorphous nature of the as-cast specimen was confirmed by XRD as shown in Fig. 1a, exhibiting a typical amorphous “hump” in the XRD pattern without any crystalline peaks. The XRD result indicates a fully amorphous alloy of the sample. Figure 1b illustrates the DSC curve of as-cast $\text{Ti}_{36.2}\text{Zr}_{30.3}\text{Cu}_{8.3}\text{Fe}_4\text{Be}_{21.2}$ bulk metallic glass. During DSC measurements, the alloy was heated to 850 K with a constant heating rate of 20 K/min. The glass transition temperature T_g and the onset of crystallization temperature T_x can be measured to be around 581 K and 651 K, respectively. The width of the supercooled liquid region (SLR) defined as $\Delta T = T_x - T_g$ is determined as 70 K, which is relatively comparable with many other good glass formers [19, 20].

3.2 Dynamic Mechanical Analysis

Figure 2 shows variations of the normalized storage modulus E'/E_u and loss modulus E''/E_u during heating with a frequency of 0.3 Hz. Two regions can be observed as marked in Fig. 2, which is very similar to the results reported in other metallic glasses [6, 21–23]: (1) from room temperature to $\sim 500\text{ K}$, the normalized storage modulus E'/E_u is high and keeps a constant while the normalized loss modulus E''/E_u is almost zero and can be ignored. The present metallic glass in this region is dominated by elasticity. As continually heating to $\sim 630\text{ K}$, the normalized loss modulus E''/E_u begins to increase to its maximum value corresponding to the main α

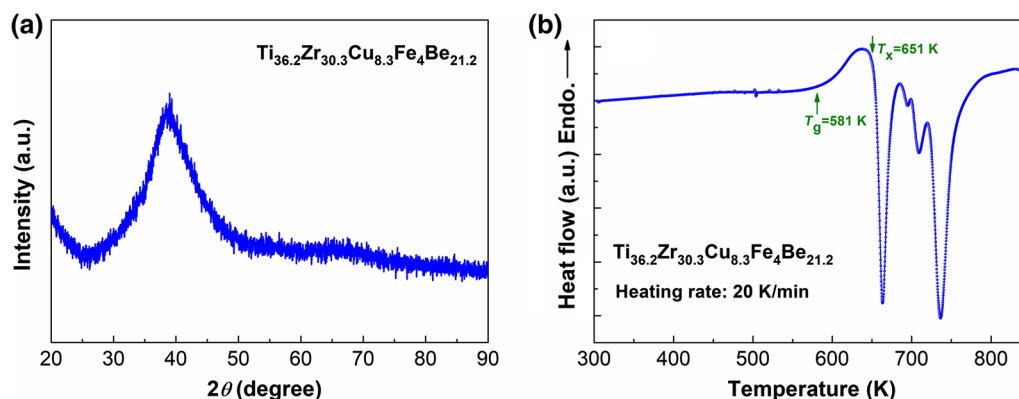


Fig. 1 a XRD pattern of the as-cast $\text{Ti}_{36.2}\text{Zr}_{30.3}\text{Cu}_{8.3}\text{Fe}_4\text{Be}_{21.2}$ bulk metallic glass, which confirms the amorphous structure of the model alloy, b DSC curve of the $\text{Ti}_{36.2}\text{Zr}_{30.3}\text{Cu}_{8.3}\text{Fe}_4\text{Be}_{21.2}$ metallic glass with a heating rate of 20 K/min

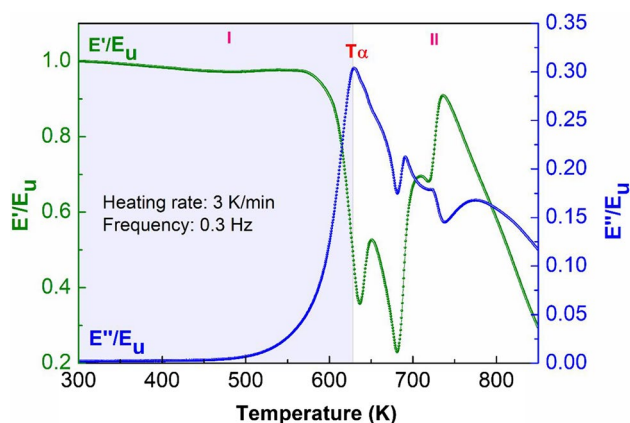


Fig. 2 Temperature dependence of the normalized storage modulus E'/E_u and loss modulus E''/E_u of the $\text{Ti}_{36.2}\text{Zr}_{30.3}\text{Cu}_{8.3}\text{Fe}_4\text{Be}_{21.2}$ bulk metallic glass. The driving frequency is set to be 0.3 Hz, and the heating rate is 3 K/min. The unrelaxed modulus E_u is estimated as the storage modulus at ambient temperature

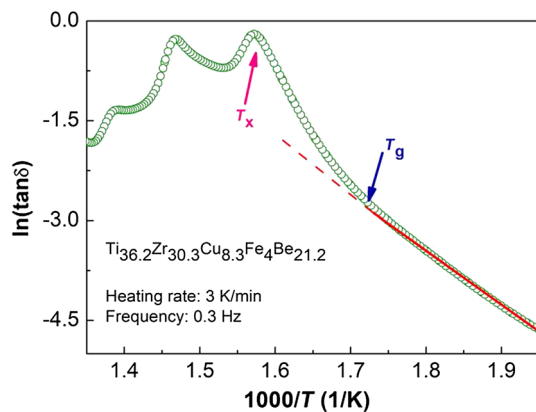


Fig. 3 Variation of $\ln(\tan\delta)$ as a function of $1000/T$ of $\text{Ti}_{36.2}\text{Zr}_{30.3}\text{Cu}_{8.3}\text{Fe}_4\text{Be}_{21.2}$ bulk metallic glass

relaxation. Meanwhile, the normalized storage modulus E'/E_u also drops as the temperature increases, and the viscoelastic component reaches its maximum value; (2) when the temperature is above 630 K, it is found that both the storage modulus and loss modulus show an evident temperature dependence. There are several crystallization events based on the DMA measurement, which is in good accordance with the DSC result.

Figure 3 shows the natural logarithm of $\tan\delta$ as a function of the reciprocal of temperature $1000/T$. A linear relationship can be observed at the low temperature region, and it is deviated near the T_g as temperature increases. The characteristic temperature at the turning point of the curve is marked as T_g in Fig. 3, which is in good agreement with the DSC results.

In order to better understand the dynamic mechanical relaxation process, the quasi-point defect (QPD) model proposed by Perez [24, 25] is adopted. In the framework of QPD model, the micro-domains corresponding to the fluctuations in enthalpy or entropy in glass-forming liquids are defined as quasi-point defects [26]. In the framework of the QPD model, the characteristic time τ^* corresponds to the average time required for the structural units to move over its own distance, which is expressed as [27]:

$$\tau^* = t_0 \left(\frac{\tau_\beta}{t_0} \right)^{\frac{1}{\chi}}, \quad (1)$$

where τ_β represents the mean duration required for the thermal-mechanical driven transitions of structural units, and t_0 is a pre-exponential factor, which reflects the time scale. It should be mentioned that τ_β can be described by an Arrhenius law:

$$\tau_\beta = \tau_0 \exp\left(\frac{E_a}{kT} \right)^{\frac{1}{\chi}}, \quad (2)$$

where E_a is the activation energy of the process, k is the Boltzmann constant, τ_0 is a scaling factor. It should be noted that the correlated parameter χ , which is closely associated with the concentration of quasi-point defects (C_d) in glassy materials [25]. According to the QPD theory, atomic or molecular mobility of the glassy materials can be described by the correlation factor χ : for the correlated parameter $\chi = 0$, it can be considered as a perfect crystal where any movement of a structural unit is constrained by other structural units, and the atoms are at the maximum degree of order. On the other hand, when the correlated parameter χ equals to 1, the system stays in totally disordered, which corresponds to the ideal gas where any structural movement is independent. As described above, the correlation factor χ ranges from 0 (fully constrained) to 1 (constraint free). On the basis of the QPD model, the concentration of these defects is strongly temperature dependent. Below the glass transition temperature T_g , the amorphous material is frozen in an iso-configuration state where the concentration of quasi-point defects remains constant since the correlation factor χ keeps constant below the T_g .

The dynamic mechanical relaxation process is sensitive to the driving frequency. In general, the loss modulus will move toward the higher temperature region as the frequency increases. Such changes can be easily interpreted as the raising frequency is equivalent to reduce the temperature from the perspective of time-temperature equivalence principle [28]. As illustrated in Fig. 4, the temperature dependence of loss modulus E'' of $\text{Ti}_{36.2}\text{Zr}_{30.3}\text{Cu}_{8.3}\text{Fe}_4\text{Be}_{21.2}$ metallic glass varies with

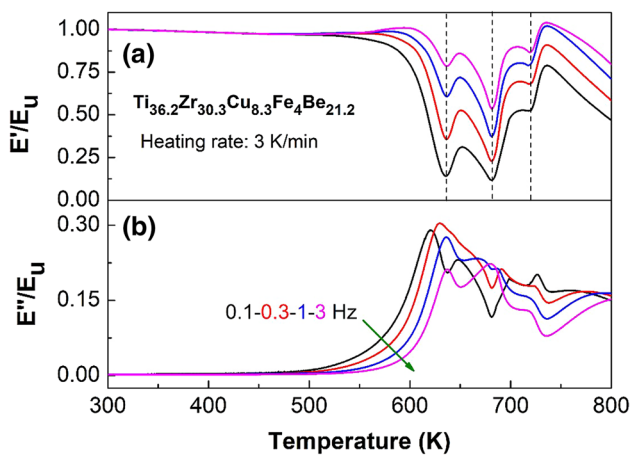


Fig. 4 Normalized storage modulus E'/E_u and loss modulus E''/E_u as functions of temperature with frequency ranges from 0.1 to 3 Hz for the $\text{Ti}_{36.2}\text{Zr}_{30.3}\text{Cu}_{8.3}\text{Fe}_4\text{Be}_{21.2}$ bulk metallic glass

different driving frequencies. Similar to other metallic glasses [7], the main α relaxation (the first peak in E'') gradually shifts toward the high temperature region as the driving frequency raised from 0.1 to 3 Hz. The amplitude of the offset depends on the activation energy of the corresponding relaxation event. Importantly, the variations of the storage modulus E' at different driving frequencies exhibit the same feature. The location of valley marked with dotted lines do not change as frequency increases, which may be related to the fast crystallization events of the Ti-based metallic glass at certain temperatures.

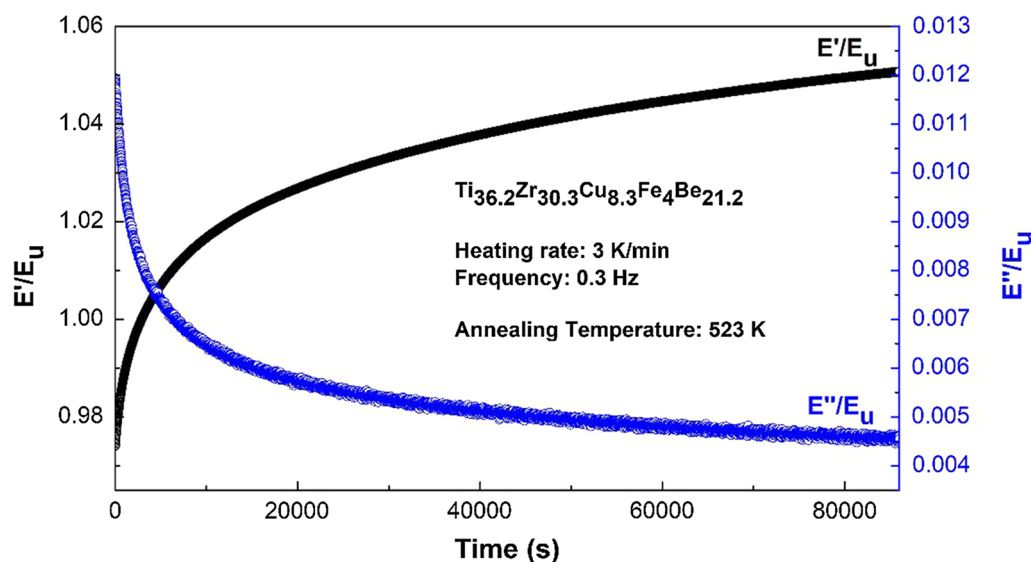


Fig. 5 Time dependence of the normalized storage modulus E'/E_u and loss modulus E''/E_u at the aging temperature of 523 K for 24 h

3.3 Heat Treatment on the Mechanical Relaxation

In order to further probe the influence of heating treatments on the dynamic mechanical relaxation behavior of the $\text{Ti}_{36.2}\text{Zr}_{30.3}\text{Cu}_{8.3}\text{Fe}_4\text{Be}_{21.2}$ metallic glass, the sample was annealed under constant temperature during the DMA equipment. To avoid crystallization, the annealing temperature was set below the T_g . As shown in Fig. 5, the isothermal treatment at 523 K results in the increase in the storage modulus and the decrease in the loss modulus, respectively, which indicates that physical annealing increases the elastic component and reduces the viscoelastic component for metallic glasses. The results are in good agreement with other reports [21, 29].

Figure 6 exhibits the evolution of internal friction $\tan\delta$ over the annealing time for $\text{Ti}_{36.2}\text{Zr}_{30.3}\text{Cu}_{8.3}\text{Fe}_4\text{Be}_{21.2}$ metallic glass. From the perspective of the QPD model, the physical aging would cause the reduction of the concentration of defects [30]. As for the annealing experiment, the decrease of defect concentration under the physical aging will lead to the reduction of the atomic mobility and then reduce the internal friction $\tan\delta$ of the present metallic glass. The evolution of the loss factor $\tan\delta$ could be well described using the Kohlrausch–Williams–Watts (KWW) type of equation [31, 32]:

$$\tan\delta(t) - \tan\delta(t = \infty) = A \cdot \exp\left[-(t/\tau)^{\beta_{\text{KWW}}}\right], \quad (3)$$

where τ is the characteristic relaxation time required for the system to reach an equilibrium state, which is governed by the atomic mobility during the structural relaxation of amorphous alloys. The parameter A denotes the maximum

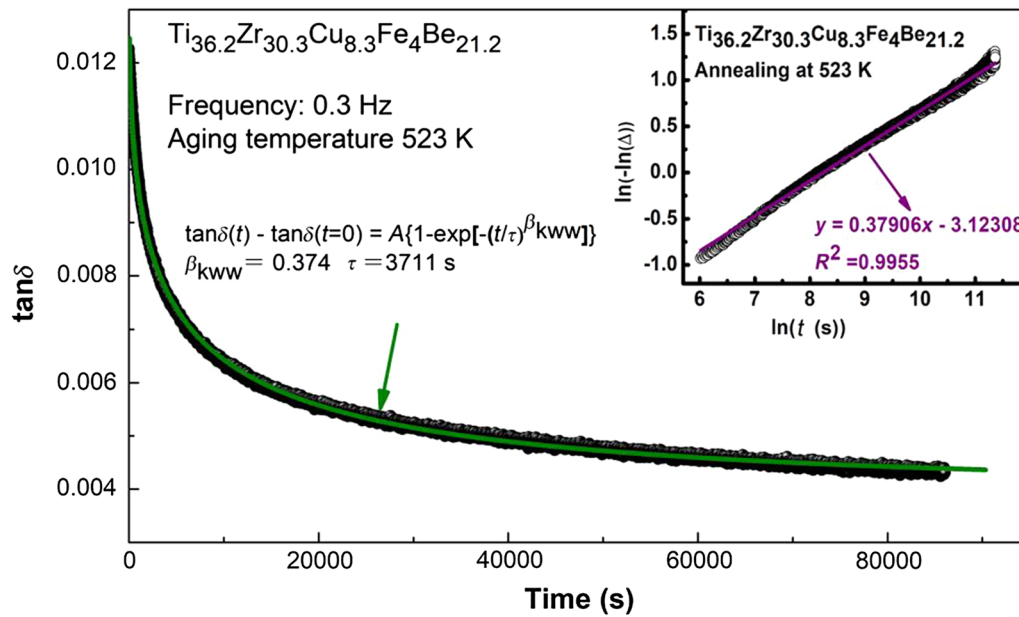


Fig. 6 Variation of internal friction $\tan\delta$ as a function of annealing time at isothermal temperature 523 K for $\text{Ti}_{36.2}\text{Zr}_{30.3}\text{Cu}_{8.3}\text{Fe}_4\text{Be}_{21.2}$ metallic glass

magnitude of the relaxation. β_{KWW} is the Kohlrausch exponent with the value ranges from 0 to 1, reflecting the distribution shape of the relaxation time. Based on Eq. (3), the value β_{KWW} of this curve could be determined to be 0.374 (as shown in Fig. 6). In such a case, another parameter Δ changes with annealing time could be defined as follows [33]:

$$\Delta = \frac{\tan \delta(t) - \tan \delta(t \rightarrow \infty)}{\tan \delta(t=0) - \tan \delta(t \rightarrow \infty)}. \quad (4)$$

The inserted figure in Fig. 6 shows the correlation between $\ln(-\ln(\Delta))$ and logarithm of annealing time $\ln(t)$, from which a good linear relationship could be observed. The Kohlrausch exponent β_{KWW} could be adopted to describe the distribution of the relaxation time considering the relaxation in metallic glasses is non-exponential. The value of β_{KWW} reflects the deviation degree of the relaxation behavior away from a single Debye relaxation for which this value would be equal to 1.

In amorphous materials, atoms or molecules in different regions have various ways to move to reach their equilibrium states, which leads to different relaxation time for different micro-domains, and then such phenomena is defined as dynamic heterogeneity [34]. Dynamic heterogeneity in amorphous materials has been observed from experimental studies [35] and could also be reflected by computational simulations on supercooled liquids [34]. The cooperative movement of rearranging structural units, resulting in the non-exponential feature in glass, reflects its wide distribution of the relaxation time and the degree of dynamic

heterogeneity. Physical aging at different temperatures will result in different degrees of the structural relaxation. Figure 7 illustrates the physical aging experiments of $\text{Ti}_{36.2}\text{Zr}_{30.3}\text{Cu}_{8.3}\text{Fe}_4\text{Be}_{21.2}$ BMG with different annealing temperatures below T_g . The amplitude of the relaxation of $\tan\delta$ is strongly related to the annealing temperature, while higher annealing temperatures lead to greater drops in the internal friction $\tan\delta$. In addition, the variations of $\tan\delta$ with annealing time at different annealing temperatures could also be well fitted by Eq. (3). As a result, the values of β_{KWW} are given in Fig. 7 for samples annealed at different temperatures. The Kohlrausch exponent β_{KWW} ranges from 0 to

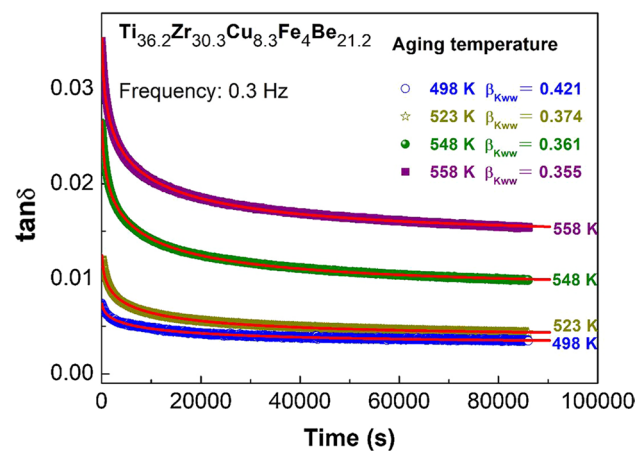


Fig. 7 Time dependence of internal friction $\tan\delta$ under different annealing temperatures, and the red lines are the fitting curves

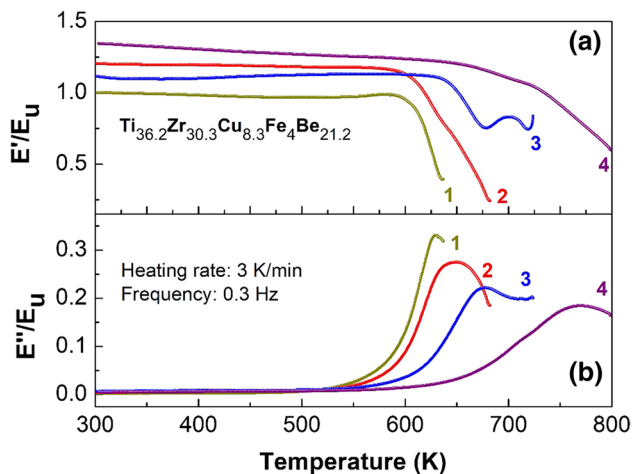


Fig. 8 Temperature dependence of the normalized storage modulus E'/E_u and loss modulus E''/E_u in $\text{Ti}_{36.2}\text{Zr}_{30.3}\text{Cu}_{8.3}\text{Fe}_4\text{Be}_{21.2}$ metallic glass during successive heating to 800 K (640–685–725–800 K). The frequency is 0.3 Hz, and the heating rate is 3 K/min

1. $\beta_{\text{KWW}} = 1$ indicates a single Debye relaxation time rather than a distribution of relaxation time. A smaller β_{KWW} value corresponds to a greater deviation from a single exponential decay and then a broader distribution of the relaxation time. As a consequence, the smaller β_{KWW} generally represents the greater dynamic heterogeneity.

To study the effects of thermal history on the dynamic mechanical relaxation of $\text{Ti}_{36.2}\text{Zr}_{30.3}\text{Cu}_{8.3}\text{Fe}_4\text{Be}_{21.2}$ metallic glass, experiments of progressive DMA heating and cooling cycles were carried out. There are totally four heating processes until finally heating to 800 K, each heating was performed after the cooling to room temperature. During the test, the DMA sample was cooled to the room temperature after each heating procedure, then reheated in the next run. Generally, successive heat treatment causes the increase in the storage modulus and the decrease in the loss modulus [36]. As shown in Fig. 8, the normalized loss modulus E''/E_u increases progressively while the peaks of loss modulus shift to higher temperatures simultaneously after previous heating. That implies that the heating and cooling cyclic treatment, similar to the effect of annealing, could lead the reduction of the quasi-point defects. Consequently, the atomic mobility in $\text{Ti}_{36.2}\text{Zr}_{30.3}\text{Cu}_{8.3}\text{Fe}_4\text{Be}_{21.2}$ metallic glass decreases drastically during the progressive heating process. The result is consistent with previous investigations [36, 37].

4 Conclusion

The dynamic mechanical behavior of $\text{Ti}_{36.2}\text{Zr}_{30.3}\text{Cu}_{8.3}\text{Fe}_4\text{Be}_{21.2}$ metallic glass has been studied by mechanical spectroscopy. The main α relaxation shown in the loss modulus is well associated with the glass transition

phenomenon in metallic glasses. In the framework of QPD model, the relationship between atomic mobility and the concentration of defects has been established by incorporating the correlated factor χ . In addition, the structural relaxation in $\text{Ti}_{36.2}\text{Zr}_{30.3}\text{Cu}_{8.3}\text{Fe}_4\text{Be}_{21.2}$ metallic glass has been investigated during annealing below the glass transition temperature T_g . The structural relaxation under annealing results in the increase in the storage modulus and the decrease in the loss modulus in metallic glasses, respectively. The variations of $\tan\delta$ as a function of annealing time could be well fitted by the stretched exponential relaxation equation, and the Kohlrausch exponent β_{aging} can reflect the degree of the dynamic heterogeneity in metallic glasses.

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