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Isothermal internal friction behaviour of a Zr based bulk metallic glass with large supercooled liquid region

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Abstract

In this paper, the internal friction behaviour of Zr-Ti-Cu-Ni-Be bulk metallic glass (BMG) containing 2 at% Fe at elevated temperatures has been studied in isothermal dynamic mechanical analysis experiments. The experiments lead to the determination of metastable equilibrium internal friction Q_e^{-1} , as a function of temperature, which can be well described by the Maxwell model with viscosity, $\eta(T)$, following a Vogel-Fulcher-Tammann (VFT) relation or Arrehnius law. Comparison with the Zr-Ti-Cu-Ni-Be glass-forming liquid shows that a small addition of Fe results in a stronger liquid behaviour of the alloy, exhibiting a higher strength parameter as well as lower VFT temperature and therefore correlates better glass forming ability and thermal stability. From the isothermal internal friction data, the activation energies for viscous flow and primary crystallization of the Zr based supercooled metallic liquid are also derived. It is found that the former is comparable to the activation energy controlling the diffusion process of atoms, e.g. the Ni element of medium size and higher mobility among the components of this alloy, while the latter to that of atoms, e.g. the Ti element of larger size and lower mobility. Thus, it is proposed that both isothermal viscous flow and primary crystallization of the alloy in the supercooleld liquid region are atom diffusion-controlled processes. However, the dominating atomic species are different from each other in the multicomponent Zr based BMG.

1. Introduction

In the past decades, several multicomponent alloy systems with superior glass forming ability (GFA) such as La–Al–Ni, Mg–Y–Cu, Zr–Ni–Al–Cu and Zr–Ti–Cu–Ni–Be have been found, which enables the synthesis of bulk metallic glasses (BMGs) at very low cooling rates, e.g. $1-10^2$ K s⁻¹ [1–4]. These BMGs typically exhibit a high resistance against crystallization at the glass transition and in the supercooled liquid region. Among these BMG alloys, $Zr_{41.2}Ti_{13.8}Cu_{12.5}Ni_{10}Be_{22.5}$ is the

best glass former, with a critical cooling rate of approximately 1 K s^{-1} [5]. Lately, the experimental results show that the addition of a small Fe element (i.e. substitution of Ni with 2 at% Fe) enhances the GFA of the alloy [6]. Moreover, the Zr-Ti-Cu-Ni-Be BMG containing Fe was found to have a higher thermal stability, revealing a larger supercooled liquid region when heated above the glass transition temperature [7]. The high stability of the Zr-Ti-Cu-Ni-Be BMG with respect to the crystallization allows for the measurements of thermophyscial properties, such as specific heat capacity [8], diffusion [9], viscosity [10], etc, far into the supercooled region over broad

time and temperature range. In addition, it has been well known that the internal friction is very sensitive to the local atomic rearrangement and kinetics of atomic movements in metallic glasses [11-23]. Thus, the study of internal friction behaviours of these novel materials is expected to provide some useful information on the inherent nature of the supercooled metallic liquid. In particular, the detailed internal friction investigations of crystallization and viscous flow of these BMGs in the supercooled liquid region appear to be essential for scientific and technological process of the metallic glasses.

In the present work, the internal friction of the Zr–Ti–Cu– Ni–Be BMG containing 2 at% Fe has been measured by using a dynamic mechanical analyser under isothermal conditions at elevated temperatures. The metastable equilibrium internal friction as well as the onset of crystallization of the Zr BMG is determined as a function of temperature. Then, the viscous flow and crystallization of the supercooled metallic liquid is further studied along with the published data on atomic transport.

2. Experimental

Bulk $Zr_{41}Ti_{14}Cu_{12.5}Ni_8Be_{22.5}Fe_2$ metallic glasses were prepared by water quenching the molten mixture of the elements in an evacuated silica tube. The amorphous nature of the as-quenched samples was verified by x-ray diffraction (XRD) with Cu K α radiation. The measurements of high resolution transmission electronic microscopy (HRTEM) and selected area diffraction were performed at 200 kV in a JEOL 2010F microscope to examine the microstructures of the annealed sample. A differential scanning calorimeter (Perkin-Elmer DSC7) was applied for the thermal analysis of the as-received Zr based BMG at a constant heating rate of 50 K min⁻¹ under a continuous argon flow.

A Dynamic Mechanical Analyser (DMA 2980 TA Instruments) was used to measure the in-phase and out-ofphase dynamic mechanical response of the samples subjected to forced oscillation in a single cantilever bending mode. The dynamic mechanical quantity, e.g. internal friction, Q^{-1} , was therefore inferred. For all isothermal measurements at elevated temperatures, samples with dimensions of $1 \times 3 \times 30$ mm³ were heated to the selected temperature at the same rate as used in the DSC scan and then maintained for the tests.

3. Experimental results

Figure 1 shows a typical DSC trace obtained from the $Zr_{41.2}Ti_{13.8}Cu_{12.5}Ni_8Be_{22.5}Fe_2$ BMG at a heating rate of 50 K min⁻¹. The DSC curve exhibits a distinct endothermic reaction due to glass transition, followed by a wide supercooled liquid region before several exothermic crystallization peaks at higher temperatures. The glass transition temperature, T_g , onset crystallization temperature, T_x and supercooled liquid region defined as the temperature interval $\Delta T_x = T_x - T_g$ are determined to be 641 K, 727 K and 86 K, respectively. This indicates that the Zr based BMG has high thermal stability, which enables the measurement of internal friction far into the supercooled liquid.

Figure 2 shows the internal friction data obtained by DMA during isothermal annealing at several elevated temperatures



Figure 1. DSC trace obtained at a heating rate of 50 Kmin^{-1} for the $Zr_{41.2}Ti_{13.8}Cu_{12.5}Ni_8Be_{22.5}Fe_2$ BMG.



Figure 2. The variation of internal friction with time during isothermal annealing at different temperatures for the $Zr_{41.2}Ti_{13.8}Cu_{12.5}Ni_8Be_{22.5}Fe_2$ BMG. The driving frequency is 1 Hz.

near T_g, i.e. 662, 657, 648, 646 and 635 K. A constant driving frequency of 1 Hz is used. It is apparent from these data that the internal friction is nearly constant immediately after the annealing temperature is reached. No notable relaxation process occurs in the beginning of the measurements, implying that the relaxation time is short for the metastable equilibrium supercooled liquid at the given temperatures. In figure 2, significant deviations from the initial metastable equilibrium internal friction (Q_e^{-1}) begin to be observed at times (hereafter defined as incubation time, τ_c) marked by the arrow, and the material then undergoes a drop in internal friction, e.g. from 0.39 to 0.096 between approximately 540 and 13000 s at 622 K. Clearly, remarkable changes in the alloy's structure or composition must occur during the course of each isothermal measurement, which results in the considerable decrease in internal friction with annealing time. Furthermore, it is noticed that while the metastable equilibrium internal friction, $Q_{\rm e}^{-1}$, of the original alloy is reduced markedly with decreasing annealing temperature, the incubation time, τ_c , becomes longer. For instance, $Q_{\rm e}^{-1}$ and $\tau_{\rm c}$ are 0.39 and 540 s, respectively, when the isothermal test is performed at 662 K, 0.29 and 1080s at 657 K, 0.17 and 2340s at 648 K and 0.15 and 3360 s at 646 K. The value of τ_c for the lowest temperature 635 K cannot be determined by means of the tangent method precisely enough to be presented in this paper

Figure 3 shows the results of HRTEM observation of the Zr based BMG annealed at 662 K for 5400 s (> τ_c of 540 s for this temperature). Obviously, the microstructure of the annealed sample is composed of nanocrystals surrounded by



Figure 3. The high resolution microscopy image and selected area electronic diffraction pattern for the $Zr_{41.2}Ti_{13.8}Cu_{12.5}Ni_8Be_{22.5}Fe_2$ BMG annealed at 662 K for 5400 s.

the amorphous matrix. In the inset of figure 3, electronic diffraction pattern of the selected area consists of some Bragg diffraction spots superimposed on the amorphous diffuse rings. These results demonstrate that the Zr based BMG crystallizes on a nanometre scale during isothermal annealing at the elevated temperature. Similar crystallization behaviour has been found in the Fe-free Zr_{41.2}Ti_{13.8}Cu_{12.5}Ni₁₀Be_{22.5} BMG and has been extensively investigated by means of various analytical methods [25-32]. Previous studies reveal that the formation of nanocrystals during isothermal annealing of the Zr_{41.2}Ti_{13.8}Cu_{12.5}Ni₁₀Be_{22.5} alloy involves preceding decomposition on a nanometre scale, which initiates an abrupt onset of nucleation within one of the decomposed phases but however limits the grain size and density of the crystalline precipitates. Considering the fact that Fe addition does not affect the phase separation of the Zr-Ti-Cu-Ni-Be BMG [33], the Zr based BMG with a small Fe addition has an isothermal crystallization mechanism similar to the Zr_{41,2}Ti_{13,8}Cu_{12,5}Ni₁₀Be_{22,5} BMG in the supercooled liquid region. Consequently, it is believed that the decrease in internal friction during isothermal annealing at the given temperatures is closely associated with the phase separation and subsequent nanocrystallization of the alloy.

4. Discussion

As Sinning and Haessner have suggested [34], a Maxwell model could be applied to describe the low frequency internal friction behaviour of a glass above T_g , which is not of anelastic but of viscoelastic origin. Generally, the behaviour of a material is called 'viscoelastic' if its stress–strain relationship contains a non-elastic, time-dependent component in addition to the instantaneous elastic deformation. If this time-dependent component (which is responsible for internal friction) is completely recoverable and linear, it is called 'anelastic', which is then a special case of 'viscoelastic'. However, the term 'viscoelastic' in this paper is restricted to the non-anelastic contribution. The internal friction is then in general a sum of an anelastic and a viscoelastic component [34].



Figure 4. The temperature dependence of metastable equilibrium internal friction of the $Zr_{41.2}Ti_{13.8}Cu_{12.5}Ni_8Be_{22.5}Fe_2$ BMG in an Arrhenius plot and fits to the experimental data using the functions as described in the text.

(This figure is in colour only in the electronic version)

Therefore, the internal friction can, in the simplest case, be related to viscosity and approximated as

$$Q_{\rm e}^{-1} = G_{\rm u}/\omega\eta,\tag{1}$$

where G_u is the unrelaxed modulus, ω the circular frequency and η the viscosity. In addition, the equilibrium viscosity of the supercooled liquid is usually reproduced with the Vogel– Fulcher–Tammann (VFT) relation [35]

$$\eta = \eta_0 \exp\left(\frac{D^* T_0}{T - T_0}\right),\tag{2}$$

where η_0 is the high temperature limit of viscosity, D^* corresponds to the strength parameter and, T_0 the VFT temperature, at which the barriers with respect to flow would go to infinity. By introducing expression (2) into (1), one obtains

$$Q_{\rm e}^{-1} = A \exp\left(-\frac{D^*T_0}{T - T_0}\right).$$
 (3)

In this formula, $A = G_u / \omega \eta_0$.

Figure 4 shows the metastable equilibrium internal friction of the initial $Zr_{41.2}Ti_{13.8}Cu_{12.5}Ni_8Be_{22.5}Fe_2$ alloy in an Ahrrenius plot. We note that fitting the metastable equilibrium data using equation (3) yields $D^* = 22.3$ and $T_0 = 311.6$ K, which are of the same order of magnitude as those determined from the viscosity measurements for other Zr–Ti–Cu–Ni–Be BMG forming liquids. Bush *et al* [35] have reported the VFT relation for $\eta(T)$ with D^* of 18.5 and T_0 of 412.5 K for Zr_{41.2}Ti_{13.8}Cu_{12.5}Ni₁₀Be_{22.5} BMG and 22.7, 372 K for Zr_{46.75}Ti_{8.25}Cu_{7.5}Ni₁₀Be_{27.5} BMG.

In previous studies, Angell [36] has proposed the fragility concept to describe the different temperature dependences of the viscosity. The liquids are therefore classified in terms of their strong-fragile behaviour. Strong liquids with high values of D^* show small changes in kinetic properties near T_g and the slowdown of mobility in the supercooled melt. The fragile ones possessing a low D^* value, on the other hand, show an abrupt change of the kinetics and the speedup of mobility. According to the classification scheme, the Zr-Ti-Cu-Ni-Be BMGs with and without Fe show behaviour closer to the strong ones. Within the fragility concept, the viscosity of liquids has been further connected to their GFA for BMGs with all metallic constituents [10,37,38], despite the behaviour of metal-metalloid BMGs such as Pd-Ni-Cu-Ni-P, not following the same trend due to the difference in their liquid structure [39, 40]. In the recent investigations by Mukherjee et al [38], the trends in glass forming among BMG-forming alloys have been investigated within the framework of viscosity and specific volume measurement. They found that the BMG forming liquids are relatively strong, revealing a very small change in volume upon crystallization and a high viscosity at melting temperature. This indicates little free volume and correspondingly denser packing density and lower atomic mobility in these strong metallic liquids. As a consequence, the strong liquid behaviour of BMGs is believed to be an important contributing factor to their superior GFA as well as a large suprecooled liquid region, since it implies sluggish kinetics in the entire supercooled liquid region and thus retards kinetically the nucleation and growth of crystallites. Furthermore, GFA is found to be better for the stronger liquid, usually exhibiting a higher value of D* and smaller VFT temperature far below glass transition.

Here, it is worth noting that while the VFT temperature of Zr–Ti–Cu–Ni–Be BMG is lowered by 80.9 to 331.6 K; the D* value increases from 18.5 to 22.3 due to the addition of 2 at% Fe. This means that with the addition of Fe, the Zr based alloy becomes stronger than the Fe free alloy, which correlates with its better GFA and larger supercooled liquid region.

Nevertheless, often over narrower temperature range, especially near T_g , the viscosity can be also approximated very well with an Arrhenius equation:

$$\eta = \eta'_0 \exp\left(\frac{E_{\rm v}}{kT}\right),\tag{4}$$

where the fitting parameters, η'_0 and E_v are the pre-exponential factor and activation energy for viscous flow of supercooled liquid, respectively. Accordingly, the relationship between metastable equilibrium internal friction and temperature in the supercooled liquid region is again expressed as

$$Q_{\rm e}^{-1} = B \exp\left(-\frac{E_{\rm v}}{kT}\right),\tag{5}$$

where $B = G_u / \omega \eta'_0$.

In figure 4, an Arrhenius fit of metastable equilibrium internal friction data yields E_v of 2.1 eV for a viscous flow of the alloy containing Fe above T_g . A more interesting finding is that the activation energy for viscous flow agrees well with a diffusion activation energy of about 2.0–2.2 eV for the Ni atom in the supercooled liquid of $Zr_{41.2}Ti_{13.8}Cu_{12.5}Ni_{10}Be_{22.5}$ BMG [41]. It is implied that the viscous flow of the Zr based alloy in the supercooled liquid region might be controlled mainly by the faster diffusion of the atom of medium size among the components of this alloy, e.g. Ni, which probably follows the collective hopping mechanism [41].

Note that there is no distinct difference between the best fitting results by using either the VFT-like equation or the Arrehnius law within the temperature range above T_g . At temperatures below T_g , the measured internal friction seems to be larger than the viscoelastic contribution as expected



Figure 5. Arrhenius plot of $\ln \tau_c$ versus 1/T for the $Zr_{41.2}Ti_{13.8}Cu_{12.5}Ni_8Be_{22.5}Fe_2$ BMG.

by equations (3) or (6), implying a more important amount anelasticity towards lower temperatures.

Through the internal friction measurements, we have also investigated the temperature dependence of the onset of crystallization of the Zr based alloy under isothermal conditions in the supercooled liquid region.

Figure 5 shows that the temperature variation of incubation time, τ_c , for isothermal primary crystallization of the alloy, can be described with an Arrhenius-type equation:

$$\tau_{\rm c} = \tau_0 \exp\left(\frac{E_{\rm c}}{kT}\right),\tag{6}$$

where τ_0 is a constant and E_c is the activation energy for isothermal crystallization. By plotting $\ln \tau_c$ versus 1/T, an approximately straight line is obtained. The activation energy, E_c , derived from this plot is 3.96 eV, which is, however, comparable to that reported on atomic mass transport in the Zr_{41.2}Ti_{13.8}Cu_{12.5}Ni₁₀Be_{22.5} BMG, e.g. $Q_{\rm Ti} = 4.09(\pm 0.46) \, {\rm eV}$ for the diffusion of the Ti element in the alloy [30]. In fact, it has been revealed in previous studies [24, 25, 29-31] that the preceding decomposition of Zr_{41,2}Ti_{13,8}Cu_{12,5}Ni₁₀Be_{22,5} BMG in the supercooled liquid region is controlled by the diffusion of atoms. Phase separation with respect to slower moving species determines the time scale of the primary crystallization of the supercooled Moreover, it has been testified that metallic liquid. upon isothermal annealing in the supercooled region, the Zr_{41.2}Ti_{13.8}Cu_{12.5}Ni₁₀Be_{22.5} alloy decomposes with respect to Ti (and presumably Be) on the nanometre scale, triggering the formation of Ti-rich nanocrystals [24, 25, 30]. These results, taken together, suggest that the isothermal crystallization of the supercooled metallic liquid of the studied Zr based BMG is also a diffusion-controlled process, in which the Ti element undoubtedly plays a key role, taking into account the coincidence of activation energies between the isothermal crystallization and diffusion of Ti.

Based upon the above analysis of isothermal kinetics data, we propose that both viscous flow and isothermal primary crystallization in the supercooled liquid region of the multicomponent Zr based BMG are atom-diffusion controlled processes. Nevertheless, the kinetics of the former is dominated by a faster diffusion of the medium sized atom, e.g. the Ni element, while that for the latter is closely related to

5. Conclusions

The isothermal measurements of internal friction have been performed using the dynamic mechanical analyser on the Zr-Ti-Cu-Ni-Be BMG former with 2 at% Fe addition at elevated temperatures. Above T_g , the metastable equilibrium internal friction data are well described by the Maxwell model with a viscosity, $\eta(T)$, following a VFT relation or Arrehnius law. A VFT-like fit to the metastable equilibrium internal friction yields a strength parameter of 22.3 and a VFT temperature of 311.6 K. When compared with the Zr-Ti-Cu-Ni-Be BMG, these results show that a small addition of Fe leads to a stronger liquid behaviour and therefore correlates with the better GFA and thermal stability of the alloy studied. From the isothermal internal friction data, the activation energies for viscous flow and primary crystallization of the supercooled metallic liquid are also determined to be 2.1 and 3.96 eV, which are comparable to those for the diffusions of atoms of different size and mobility, i.e. the Ni and Ti element, respectively. Thus, it is proposed that both isothermal viscous flow and primary crystallization of the alloy in the supercooled liquid region are atom diffusion-controlled processes. Nevertheless, the kinetics of the former is dominated by the faster diffusion of the medium sized atom, e.g. the Ni element, while that of the latter is closely related to the relatively slow movements and arrangements of atoms of larger size, e.g. the Ti element. That is, the dominating atomic species are different from each other in the multicomponent Zr based BMG.

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