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Role of elastostatic loading and cyclic cryogenic treatment on relaxation behavior of Ce-based amorphous alloy

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ABSTRACT

In this work, $Ce_{65}Cu_{15}Al_{10}Ni_8Co_2$ bulk metallic glass was annealed at sub-T_g and then elastostatically and cryogenically was loaded and treated, respectively. X-ray Diffraction (XRD) analysis was carried out to show amorphousness of treated alloys, and enthalpy variations and specific heat capacity were evaluated using differential scanning calorimetry (DSC). The results showed that the designed treatment procedure leads to the recovery of relaxation, i.e. rejuvenation, from the relaxed state to the as-cast condition. It is suggested that the generation of free volume and the structural rearrangements, appeared in the abnormal behavior of DSC curves, are responsible for recovery of relaxation. It is also found that the primary elastostatic loading facilitates the subsequent rejuvenation evolution caused by the cryogenic cycling process.

1. Introduction

Due to their unique thermodynamic and kinetic features, bulk metallic glasses (BMGs) are formed from a non-equilibrium state with a high energy level [1–3]. With decrease in the glass transition temperature (T_g), complex atomic arrangements occur and the material relaxes to an equilibrium structure with an energy state higher than the crystalline counterpart [4,5]. In an amorphous structure, high temperature leads to an aging behavior, i.e. structural relaxation, accompanied with short and medium atomic rearrangements [6,7]. Under this condition, the short- and medium-scale ordered configurations increase so that the BMG tends to have a relaxed structure. On the other side, it is possible to enhance stored energy and excite atomic structure, namely rejuvenation, by some specific treatments [8–10]. In a high-energy amorphous structure, short range order (SRO) and medium range order (MRO) arrangements decrease and plasticity of BMG improves [11]. In recent years, some techniques such as ion irradiation [12–14], laser peening [15,16], mechanical loading [17–20], plastic deformation [21–24], cryogenic cycling and thermal treatments [25–28] have been used to rejuvenate glassy alloys. In all the mentioned techniques, the main objective in a rejuvenation process is to intensify structural heterogeneity and increase possible sites for nucleation of shear bands through annihilating of ordered structures [29,30]. In general, a rejuvenated structure has a weaker backbone with lower number of SRO and MRO configurations providing more nano-scale soft regions for creation of shear transformation zones (STZ). This event leads to multiple shear banding and subsequent improved plastic deformation under an external loading.

In this study, we designed a combinational technique, including mechanical and cryogenic treatments, to rejuvenate a BMG alloy. In this method, the as-cast BMG sample was loaded with a compressive elastostatic loading and immediately exposed to a cryogenic cycling treatment with different number of cycles. The experimental treatment was designed somehow to provide conditions for studying the relaxation behavior of BMG alloy. It should be noted that our rejuvenation process was performed in room temperature. Hence, it is very important to

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1. Introduction

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consider an alloying composition which is possible to show the relaxation behavior at lower temperature. For this purpose, a Ce-based BMG alloy with low-temperature relaxation region and good glass forming ability (GFA) was fabricated and subsequent treatments were performed to investigate the relaxation/rejuvenation behaviors of material.

2. Materials and methods

At the first step, arc re-melting process was applied to prepare $Ce_{65}Cu_{15}Al_{10}Ni_8Co_2$ ingots with high purity constituents (>99.8 %) under a Ti-gettered argon environment. This process was done five times to ensure the elemental homogeneity. The ingots were then cast in the form of 1-mm diameter rods by a water-cooled copper mold casting technique. The samples were cut from the rods with the length of 2 mm. To avoid any extra heat generation during the cutting process, a slow rate water-cooled diamond was applied to cut BMG samples from the reference rod. The as-cast sample was considered as the reference (S1). Other samples were annealed in a vacuum furnace for 3 h at 0.95 T_g. An annealed sample (S2) was kept for the experiment. Afterwards, other annealed samples were elastostatically loaded at 90 % yield strength for 4 h in the room temperature using a compression machine test. It should be noted that previous works confirmed that the compressive elastostatic loading at near yield strength increases the possibility for structural rejuvenation in BMGs [31]. The pure loaded sample was coded as S3 in the paper. The other loaded samples were then treated under a cryogenic cycling treatment procedure with cycle numbers of 20, 40 and 60 (see Fig. 1). The mentioned samples were coded as S4, S5 and S6, respectively. After the mentioned processes, X-ray diffraction (XRD; Rigaku Ultima IV instrument) test with Cu-Kα radiation and $2\Theta = 25-95°$ was used to evaluate amorphousness of treated samples. It should be noted that XRD analysis was done from the cross sections of samples. Differential scanning calorimetry (DSC) experiment was also carried out at 20 K/min under pure argon atmosphere to analyze temperature characteristics of amorphous samples. It is worth mentioning that in order to remove any oxidation and contamination, careful polishing process was done using a double disc polishing machine (MPT RSMP-2S) with 550 RPM prior to the treatments and subsequent experiments.

3. Results

The XRD patterns for all the samples with different rejuvenation processes are given in Fig. 2. The absence of sharp diffraction peaks suggests that there is no crystalline phase in the samples and the glassy nature remained intact after the certain treatments.

Fig. 3 shows specific heat capacity (C_p) of specimens in different states at the temperature range of glass transition. To evaluate the changes in value of C_p, it is very important to remove experimental errors from the curves. For this purpose, a correction process using an additional base line was done to minimize the eventual errors [32]. As observed, the annealing treatment has a considerable effect on kinetic and thermodynamic evolution in the range of glass transition temperature so that the annealing process weakens exothermic event below the glass transition and intensifies overshoot peak above the glass transition temperature (T_g). A detailed look on Fig. 3 reveals that the overshoot peak at the end of glass transition temperature is accompanied with another abnormal semi peak near the crystallization temperature (T_x). In fact, two semi peaks are located between T_g and T_x, which illustrates the eccentric endothermic behavior of material in super cooled liquid region. This feature is very clear for the annealed sample (S2), which shows a two-step-like glass transition phenomenon [33]. Moreover, one can see that there is no apparent exothermic peak or shoulder below T_g in both of reference (S1) and annealed (S2) samples, which indicates that a low amount of free volume and quench-in defects exist in the liquid glass former in comparison with many other alloys [11,21,30]. In other words, it is concluded that the Ce-based BMG has a strong liquid with considerable amount of atomic clusters penetrating to each other. Comparing S1 with S2, it is suggested that the annealing treatment leads to the structural relaxation; however, it is not very obvious which is due to the inherent features of our strong BMG. Nevertheless, T_g value increases about 18 K (see Table 1) and peak intensification occurs in the super cooled liquid region, which implies higher kinetic stability of annealed sample.

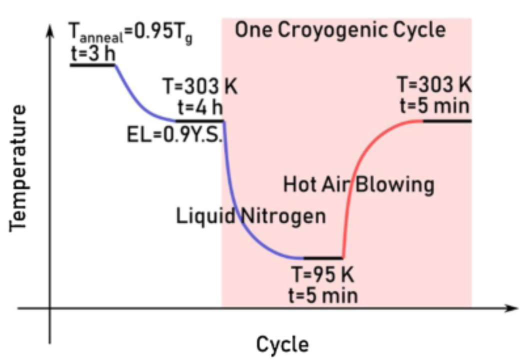

Fig. 1. Schematic of rejuvenation treatment for the BMG samples.

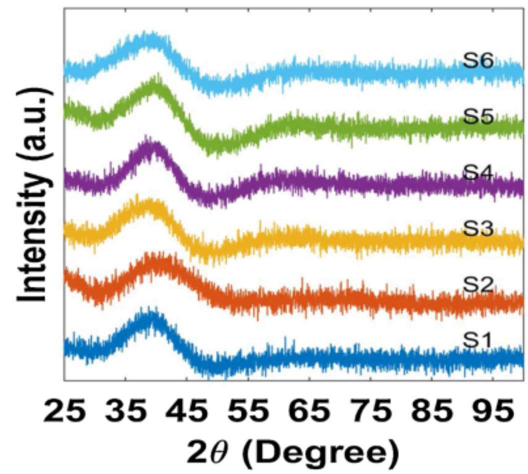

Fig. 2. XRD patterns of all the samples.

Fig. 3 also determines that the elastostatic loading (EL) and cryogenic cycling (CC) affect the kinetic and thermodynamic features of Ce-based samples. In comparison with sample S2, the EL process weakens the peak intensities in super cooled liquid region. Furthermore, with addition of CC process into the treatment, the mentioned peaks were further weakened. It is worth-mentioning that T_g decreases with application of EL/CC processes and moves to the reference sample (S1) trend. This trend continues with the increase in the number of cycles so that sample S6 has the lowest T_g (See Table 1). Hence, one can conclude that the enthalpy of rejuvenated samples rises in comparison with the annealed and as-cast samples.

Integrating the C_p values from DSC results, it is possible to evaluate the energy state of samples with different treated conditions [33]. Fig. 4 shows enthalpy curves of samples, which include the glass, crystal and liquid enthalpy regions. Considering the enthalpy trend attained from the reference sample, the enthalpy of liquid (H_L) can be fitted as follows:

$$H_L = -14.19 + 0.051T - 1.85 \times 10^{-5} + 7.8 \times 10^{-9}$$

While the enthalpy of crystallization is fitted by a linear equation:

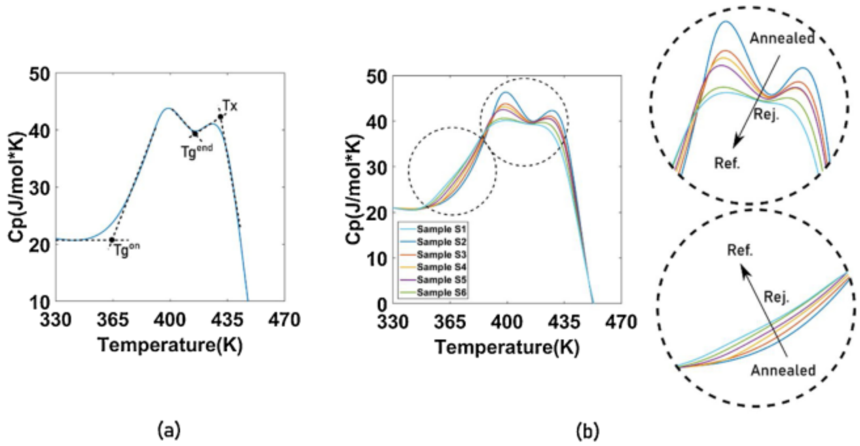

Fig. 3. C_p-T curves at the temperature range of glass transition and super cooled liquid region for all the samples.

Table 1
Temperature characteristics of samples obtained from the DSC results.

Temp. features (K)	S1	S2	S3	S4	S5	S6
T_g^{on}	358	376	373	370	368	361
T_g^{end}	413	422	421	419	418	415
T_X	426	432	432	430	429	427

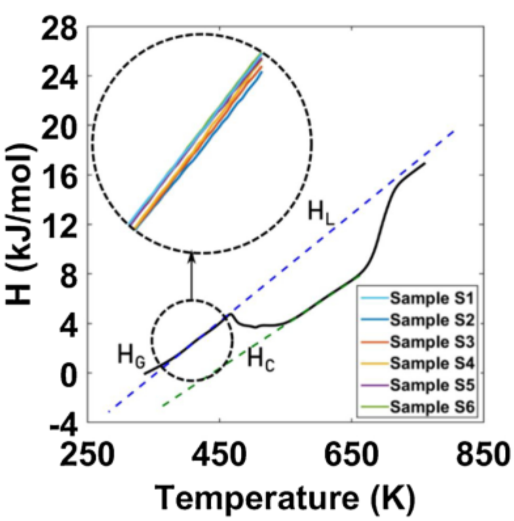

Fig. 4. The enthalpy of samples in different states.

$H_C = -11.91 + 0.029T$

Fig. 5 presents the enthalpy curves of samples relative to H_C trace. Two turning points in each curve indicate that a two-step enthalpy change dominates on the energy trend of amorphous samples as a function of temperature. The first step occurs in the temperature between T_g^{on} and T_g^{end}, while the second step is located in the temperature range of T_g^{end} and T_x. As can be seen, the enthalpy values for rejuvenated treated samples at super cooled liquid and glassy states is higher than the relaxed one (S2). However, it does not go beyond the enthalpy of reference sample (S1).

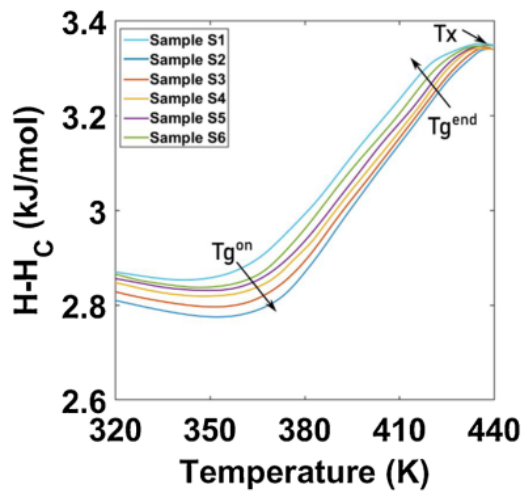

Fig. 5. relative enthalpy of samples attained by subtracting H_C.

Using the following equation, it is possible to estimate the rejuvenation degree of treated samples relative to the annealed one:

$$\frac{H_G^{treated} - H_G^{annealed}}{H_G^{reference} - H_G^{annealed}}$$

In fact, the recovery of H_G determines the effects of EL and CC processes on atomic rearrangements and enhancement of free volume in the BMG structure. Considering enthalpy of samples S1 and S2 as the highest and lowest limits, respectively, a rejuvenation degree of 18 % occurs for the elastostatic loaded sample (S3) (see Fig. 6). This improvement continues with the application of CC process so that with the increase in number of cycles, the rejuvenation degree enhances and reaches the reference sample. Moreover, it is observed that a saturation trend in rejuvenation degree takes place, which shows that structural rejuvenation is not simply an indefinite evolution extending the BMG structure into the highest theoretical energy level.

4. Discussion

The rejuvenation behavior of a glassy alloy strongly relies on primary structural features such as chemical heterogeneity, free volume distribution and internal stress domains [29]. In order to rejuvenate a BMG, it

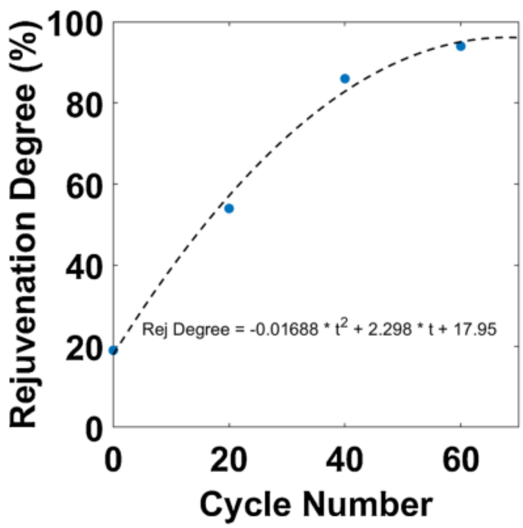

Fig. 6. The rejuvenation degree of samples as a function of cycle number.

is required to amend one of the mentioned features. In general, the sub-T_g annealing treatment provides the essential energy for atomic configuration to form a new arrangement in the microstructure. Moreover, the annealing treatment along with a common air cooling process leads to the increase in SRO and MRO arrangements in the backbone of material. Hence, one can see that sample S2 is energetically more stable than S1. On the other side, EL process increases the level of internal stresses in the material. In general, a time-dependent loading intensifies the hydrostatic stress components in the atomic structure and weakens the ordered arrangements, i.e. SRO and MRO, in the matrix so that the short range clusters tend to break or isolate from each other [18]. Moreover, when the cryogenic cycling process comes into play, the rejuvenation degree dramatically increases in the microstructure. In fact the difference between the rejuvenation mechanism of EL and CC processes is the main reason for the enhancement of rejuvenation during the cryogenic treatment. Based on pair distribution function results in ref. [9], EL process tends to decrease SRO and MRO arrangements in the atomic configuration, while CC treatment increases high energy SRO clusters and enhances loose packing MRO arrangements. In other words, energy landscape of the glassy structure is inherently rugged, so the possibility for sequential atomic rearrangements and cluster-to-cluster rearrangement increases [9]. Using Mossbauer spectroscopy for Fe-based amorphous alloys, researchers also found that the structural heterogeneity and atomic packing density are strongly affected by the types of defects distribution in the material [34,35]. Hence, it is derived that the companionship of CC and EL processes intensifies the structural rejuvenation in the annealed.

Moreover, it is very important to consider the role of structural heterogeneity on evolution of rejuvenation in the BMGs [30]. A heterogeneous structure is capable to create more residual stresses in material. According to DSC results, the presence of two semi peaks in the super cooled liquid region of our Ce-based alloy indicates that a considerable heterogeneity exits in the microstructure. However, it is crucial to note that the semi peaks in DSC curves are signs of compositional heterogeneity and it is not possible to directly confirm the level of topological heterogeneity in the microstructure. As mentioned before, this alloy is in the classification of strong BMGs and has a considerable amount of SRO and MRO arrangements. Therefore it can be estimated that topological heterogeneity is not much enough to be a driving force for the rejuvenation. While the chemical heterogeneity facilitates the cluster-to-cluster transitions taken place in the CC process.

In addition to the mentioned descriptions, researchers showed that two semi-peaks in the super cooled liquid region indicates the intensification of MRO arrangements in the microstructure [33]. In general, string-like arrangement of SRO structures leads to the formation of MRO configurations in the backbone of material. This abnormal behavior in the super cooled liquid region implies that different MRO configurations exist in the microstructure. Hence, it is concluded that the recovery of relaxation, i.e. rejuvenation, from the annealed situation is accompanied with the uniformity of MRO structures so that the semi peaks in DSC curves of treated samples pale into insignificance.

In another viewpoint, some works have been revealed that existence of T_g^{on} and T_g^{end} in a glassy alloy relates to the formation of atomic clusters in super cooled liquid region [36]. However, the considerable amount of SRO and MRO structures in Ce-based alloys suggests that there is no chance for the formation of more ordered clusters in the super cooled liquid region and it is derived that another event may be associated to the appearance of sub-T_x semi peak. With the evaluation of ordered network in the BMG alloys, it is found that the energy level of SRO is lower than the liquid-like structures [37]. Moreover, the energy level of SRO arrangement decreases with the formation of MRO networks. Hence, it can be concluded that sub-T_x endothermic event in the Ce-based alloy may be due to the rearrangement and decomposition of MRO configurations to the isolated SRO clusters with higher energy level.

The results also showed that the cryogenic treatment significantly changes the structural heterogeneity and energy landscape in the BMG; however, the rejuvenation mechanism is unclear and vague. In a work done by researchers, it was revealed that the cryogenic treatment induces non-affine atomic rearrangements in the BMG, which may be related to a low temperature event. i.e. gamma relaxation at 0.2–0.3 T_g, affecting the local atomic structures [38]. In this work, the CC process includes the temperature range of 0.2–0.3 T_g and consequently the excitation of microstructure and recovery of relaxation enthalpy can be related to the low temperature events. Moreover, it is suggested that application of EL process before CC treatment leads to the weakening of ordered backbone and the annihilation of SRO and MRO networks and provides the primary driving force, i.e. intensification of heterogeneity, for rejuvenation evolution. The results also indicated that the recovery of relaxation enthalpy is saturated so that the rate of rejuvenation recovery decreases with the rise in the number of cryogenic cycles. The decrease in the rejuvenation rate strongly depends on number of potential sites for the structural rearrangement. Hence, with the rise in number of cycles, the internal stress domain are weakened and the certain atomic spaces for atomic rearrangement decline.

5. Conclusion

This work aimed to show that how elastostatic loading and cryogenic cycling processes recover the relaxation energy of annealed $Ce_{65}Cu_{15}Al_{10}Ni_8Co_2$ bulk metallic glass. The results indicated that the two abnormal peaks appeared in the DSC curve of amorphous alloys in the range of super cooled liquid region, showing the compositional heterogeneity and MRO variations in the structure. With application of mentioned treatments, the BMG energetically becomes more unstable and moves from the relaxed state to the as-cast condition. With the increase in the number of cryogenic cycles, the rejuvenation enhances in the microstructure and more internal stress domains and free volume are generated in the material. Moreover, it is revealed that the primary elastostatic loading facilitates the subsequent rejuvenation evolution caused by the cryogenic cycling process.

Declaration of Competing Interest

There is no conflict of interests.

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