Reversible anelastic deformation mediated by β relaxation and resulting two-step deformation in a La$_{60}$Ni$_{15}$Al$_{25}$ metallic glass

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The stress relaxation dynamics of a La$_{60}$Ni$_{15}$Al$_{25}$ metallic glass were studied in ribbon and bulk samples. In both tensile and single cantilever testing modes, it is observed that the stress decay of deep glass is mediated by the β relaxation, which contributes about 5% to the total stress. The characteristic time of stress relaxation near the glass transition coincides with that of the α relaxation, indicating that the two-step stress decay may correspond directly to the two dynamic relaxations. A possible atomic mechanism involving both relaxation and deformation is proposed based on the evolution of shear transition zone, which enables reconstruction of the two-step deformation theoretically. The present experimental and theoretical protocol further provides a strategy to detect the β relaxation associated phenomena at room temperature and even lower, circumventing interference from physical aging or the α relaxation. These findings clarify the elusive roles of relaxation modes in the nonelastic deformation of amorphous matters and reveal the interrelationships between them.

I. INTRODUCTION

The relaxation dynamics and nonelastic deformation mechanisms of glasses pose one of the most challenging, unsolved scientific problems in condensed matter physics and solid mechanics [1–5]. As the glass-forming liquid is cooled down to about $1.2\,T_g$ (with T_g the glass transition temperature), the single-relaxation mode splits into α and β processes [6–9]. When the α relaxation becomes slow below T_g, the β relaxation is then the principal source of atomic dynamics in the deep glassy state [10–12]. Previous studies have shown that the β relaxation is essential for understanding the glass transition [13], structural heterogeneity [14], and deformation [15–17] of metallic glasses (a category of closely packed glasses with metallic constituents), although it should be acknowledged that there is still considerable controversy in the role of the β relaxation played in dynamics of the generic glass-forming systems.

How the β relaxation is related to deformation is also an important issue worthy of further discussion. Glasses are believed to deform through atomic rearrangement in local soft regions involving from tens to hundreds of atoms which are termed shear transformation zones (STZs) or flow units [18–20]. Yu *et al.* [21] estimated the potential energy barrier of STZ activation based on the cooperative shear model (CSM),

pointing out that it is nearly equivalent to the activation energy of the β relaxation. By probing the changes of potential energy, shear modulus, and anelastic strain recovery, Johnson and Samwer [22] proposed that the isolated shear transformations confined in the elastic matrix could be associated with the β relaxation, while percolation of atoms in STZs is associated with the α relaxation. Unfortunately, up to now, there is no experimental observation of the β relaxation directly involved in deformation of metallic glasses. Luo *et al.* [23,24] reported an intriguing two-step deformation phenomenon in several metallic glasses by monitoring the stress decay under constant strain. However, calculation of the characteristic time suggests that the fast process observed is not related to the β relaxation. Regarding recent findings [25], the temperature derivative of the stress–decay amplitude displays a profile similar to the dynamic relaxation curve, which is more likely to be attributed to the competition between physical aging and thermal activation, rather than the β relaxation effect. We believe that the detection of deformation mediated by the β relaxation should follow the following principles: (1) Observations at lower temperatures. On the one hand, low temperature avoids interference from physical aging or α relaxation. On the other hand, the characteristic time of the β relaxation increases at low temperature, which is conducive to detecting on the experimental time scale. (2) More precise measurement systems. The deformation caused by the β relaxation may be relatively small, and the use of more precise deformation measurement can prevent such small deformations from being overlooked.

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In this paper, we monitored the stress decay of a $La_{60}Ni_{15}Al_{25}$ glass under constant strain across a broad range of temperatures, to room temperature and even lower. Deformation experiments were performed on TA DMA Q850, which can start to record data at the rate of ten points per second just 0.5 s after loading. Compared with previous works, the current experimental method enables us to observe the exact evolution of stress within tens of seconds after loading. Direct experimental evidence is provided to support a scenario that the β relaxation only partially controls the reversible anelastic deformation, while the rest of the stress decay can be considered as the transition from anelasticity to plasticity controlled by the α relaxation, which provides understanding of glass relaxation dynamics and deformation. Another significance of the current work is to provide an effective method for detecting the β relaxation and the related properties at low temperature without interference from physical aging or α relaxation. Finally, a physical picture involving multiple scales is further constructed, which is of great significance for understanding the complex dynamics and deformation mechanisms of glassy materials.

II. MATERIALS AND METHOD

The selected compositions for this study are two representative metallic glasses, $La_{60}Ni_{15}Al_{25}$ and $Zr_{50}Cu_{40}Al_{10}$, which exhibit different β relaxation behaviors, with glass transition temperatures of 461 and 692 K (see Fig. S1 of the Supplemental Material [26] for their differential scanning calorimetric curves), respectively. Bulk samples (30 mm × 2 mm × 1 mm) and glassy ribbons with a uniform thickness of 30 μm were used for the stress relaxation measurements. More technical details about the sample preparation and experimental information are available in the Supplemental Material [26]. Figure 1(a) shows the stress relaxation curves at a constant strain of 0.6% at various temperatures with a step of 5 K. Stresses are normalized by their initial value σ_0. After reaching each test temperature, the samples were stabilized for 30 min to ensure the thermal stability of the clamps and furnace. Interestingly, the stress decay exhibits a two-step behavior in the temperature range 315 ($0.68T_g$)–360 K ($0.78T_g$). For clarity, the successive offset curves are shown in Fig. 1(b), where a transition from the two-step to the single-step decay upon heating can be clearly observed.

III. RESULTS AND DISCUSSION

In the light of current knowledge, glasses have two main relaxation processes reflecting their atomic dynamics: The fast secondary β relaxation and the slow primary α relaxation [27]. It is natural to ask whether the fast stress decay observed here can be attributed to the β relaxation, and whether the slow decay is related to the α relaxation. To confirm the relationship between the fast stress decay and the β relaxation, the temperature dependence of their characteristic time should be compared. The characteristic time τ_1 of the fast decay process can be identified by the derivative of the normalized stress σ/σ_0 with respect to the logarithmic time $\ln t$, as shown in Fig. 2(a). The characteristic time τ_β of β relaxation is
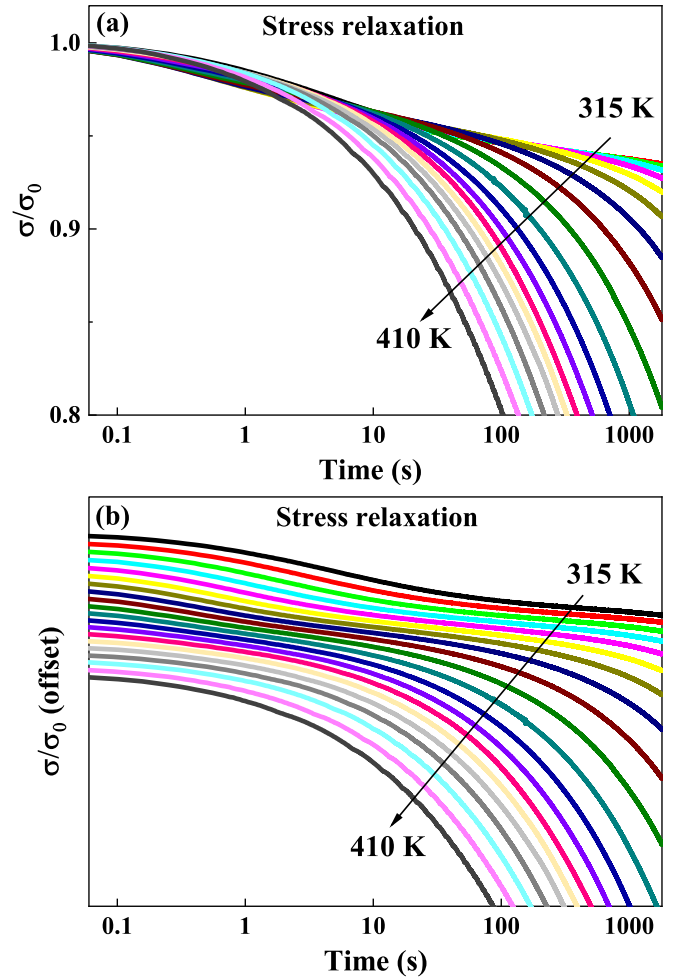

FIG. 1. A two-step stress decay phenomenon in the $La_{60}Ni_{15}Al_{25}$ metallic glass. (a) Representative stress relaxation at temperatures from 315 to 410 K. Successive offset of the curves is further shown in (b).

standardly determined from the dynamic mechanical spectra [Figs. 2(b) and 2(c)]. It is obvious that the β relaxation shifts to higher frequencies (temperatures) with the increase of test temperature (frequency), implying the gradual decrease of its characteristic time. The τ_β can be deduced from the relation $2\pi f_{peak}\tau_\beta \approx 1$ [28]. As shown in Fig. 2(d), τ_1 and τ_β have the same Arrhenius-type temperature dependence, indicating that the fast decay process is equivalent to the β relaxation. The Arrhenius fit to the data yields an activation energy $E_\beta = E_1 = 83$ kJ/mol. For comparison, we also conducted stress relaxation tests on $Zr_{50}Cu_{40}Al_{10}$ metallic glass without clearly separated β relaxation [Fig. 2(e)] in the temperature range 450 ($0.65T_g$)–550 K ($0.79T_g$). The test temperature range is approximately below $0.75T_g$, corresponding to the relative temperature range of the two-step decay in $La_{60}Ni_{15}Al_{25}$ metallic glass. It can be observed from Fig. 2(f) that the stress relaxation response does not exhibit the two-step decay phenomenon, which further indicates the consistency between the fast stress decay process and the β relaxation.

Furthermore, it can be expected that the fast decay process in the stress relaxation experiment at lower temperature will
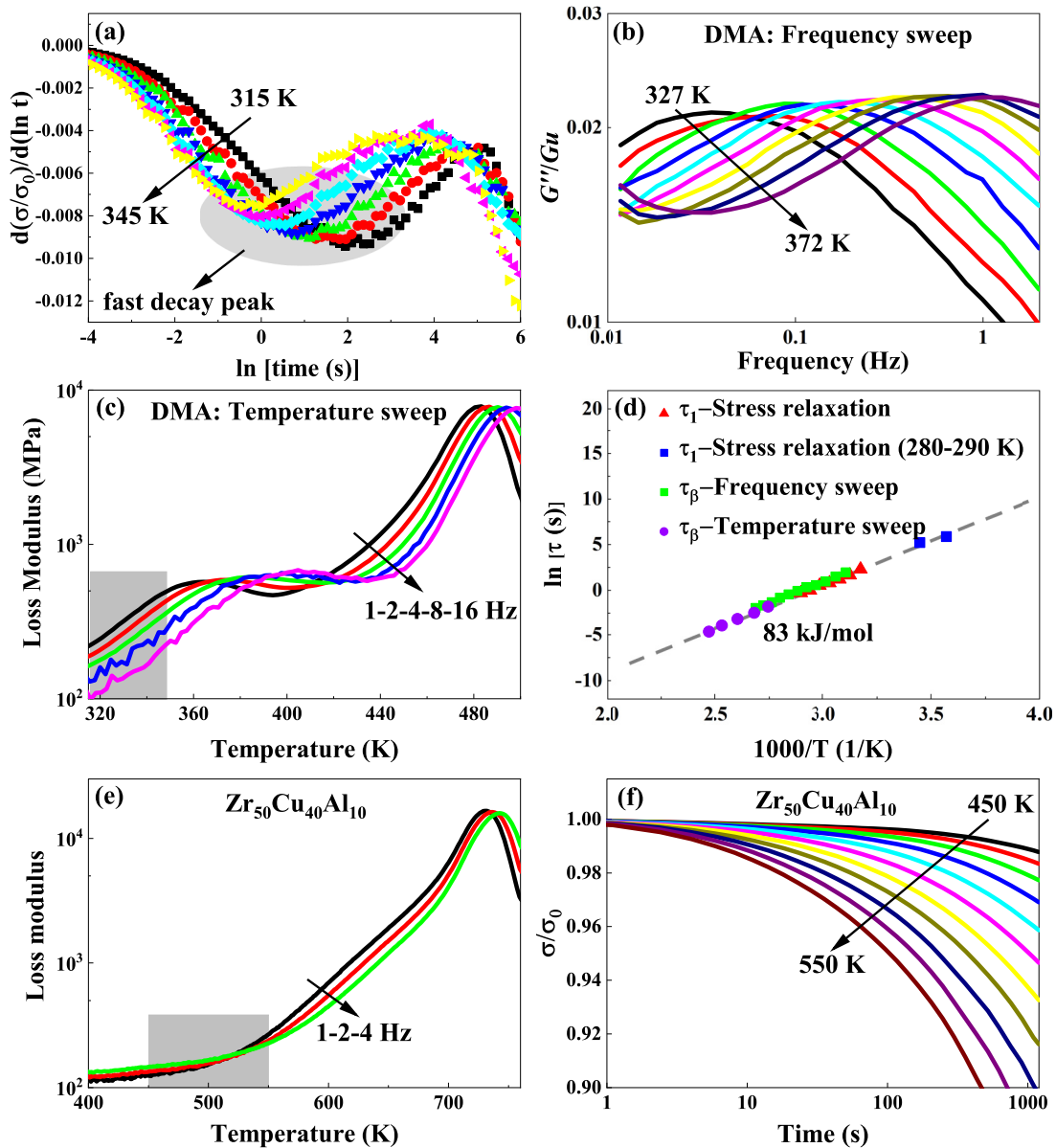
FIG. 2. Consistency between fast stress decay and the β relaxation. (a) Identification of characteristic time τ_1 of the fast decay process from the stress decay rate versus time. (b) Normalized loss modulus spectra G''/G_u as a function of frequency at various temperatures. (c) Temperature dependence of loss modulus on frequency with constant heating rate of 3 K min^{-1}. Light gray area corresponds to the temperature range of two-step stress decay. (d) Temperature dependence of the stress relaxation characteristic time τ_1 and that of the β relaxation τ_β, respectively. (e) Temperature dependence of loss modulus of a $Zr_{50}Cu_{40}Al_{10}$ metallic glass without clear β relaxation. (f) Stress relaxation of $Zr_{50}Cu_{40}Al_{10}$ at temperature range shaped in (e).

be slower than that at higher temperature, because the τ_β follows an Arrhenius law. This is also confirmed by the blue squares in Fig. 2(d), which represent the characteristic time of fast decay in the stress relaxation measurements of the single cantilever mode at 280 K ($0.61T_g$) and 290 K ($0.63T_g$) (see Fig. S2 for their stress relaxation curves [26]). Compared with the literature, the current result is of considerable significance for further study of the β relaxation and its related physical properties. As the temperature decreases, the interference of aging and α relaxation on the measurement of β relaxation may decrease or even be negligible.

The above results have shown that the fast stress decay process can be attributed to the localized atomic motion origi-

nating the β relaxation observed by mechanical spectroscopy. However, the dynamic process of the deep glass state appears to be rather intricate, as suggested by the stress relaxation process identified by Luo *et al.* [23,24] over a time duration of approximately 1000 s. Therefore, it is necessary to further clarify the mechanism of relaxation dynamics of the glass state. It is generally believed that the β relaxation is related to local and reversible atomic motion in metallic glasses [29,30]. In this context, one may wonder whether the fast decay process in the stress relaxation measurements is also reversible. Therefore, a set of successive loading/unloading experiments were performed isothermally at 315 K, starting after 10 min of thermal equilibration at this temperature. The schematic
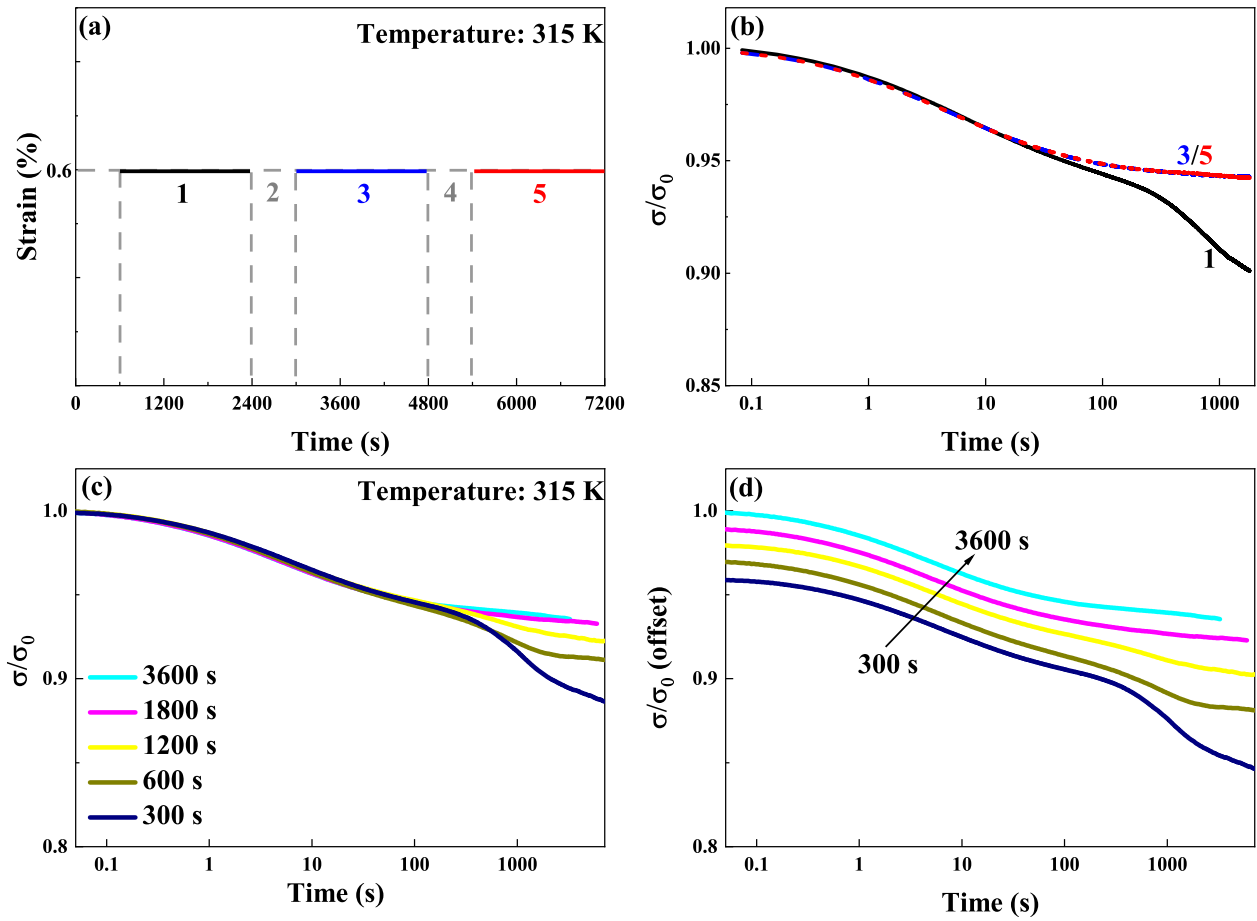
FIG. 3. Reversibility of the stress decay process mediated by the β relaxation. (a) Schematic illustration of the successive loading/unloading experiments at 315 K after 10 min thermal equilibration. (b) Stress relaxation curves corresponding to stages 1, 3, and 5, respectively. (c), (d) Stress relaxation curves and their offsets starting after different duration of isothermal annealing.

illustration is shown in Fig. 3(a). Stages 1, 3, and 5 are stress relaxation processes, while stages 2 and 4 are unloading processes. The three stress relaxation curves are shown in Fig. 3(b), where the last two stress relaxation curves coincide, reflecting a reversible deformation. However, another stress decay process is observed in the first stress relaxation curve in the time range 100–1000 s, which appears to be consistent with the dynamic process identified by Luo *et al.* [23,24].

The influence of the duration of the preannealing on the stress relaxation response is shown in Figs. 3(c) and 3(d). It is observed that this stress decay process disappears after a preannealing of duration 1800 s. This irreversible behavior suggests that this is not an intrinsic relaxation process, but it is related to the evolution of the glassy state during annealing. However, the β relaxation mediated stress decay is an intrinsic dynamic behavior which leads to reversible, anelastic deformation. It can be observed that the amplitude of stress decay caused by β relaxation is about 5%, which means that the contributions of different relaxation processes to deformation seem to be separated. We believe that this result is crucial to understand the intrinsic relationship between relaxation dynamics and deformation.

After determining that the first intrinsic fast decay of stress relaxation is a reversible process corresponding to the β re-

laxation, the other question is whether the slow stress decay is controlled by the stress-assisted activated α relaxation. The answer is probably yes. As shown in Fig. 4(a), the pink area shows the characteristic time of the α relaxation (τ_α) and the slow stress decay (τ_2) near the glass transition. The experimental curves used to identify the characteristic time are shown in Figs. S3 and S4 [26]. It can be observed that τ_α and τ_2 merge, which indicates that the overall stress relaxation may be closely related to the α relaxation. It should be noted that τ_β extrapolated from the Arrhenius law at such high temperatures ($0.93--1.01T_g$) would be about 10^{-4} s, so the fast stress decay process can be completely ignored in our mechanical experiments as it is off the experimental time window. Therefore, the stress relaxation response obtained at this time is the single slow decay process. The other reason is that the stress relaxation method is equivalent to dynamic mechanical spectroscopy. They are, respectively, the time and frequency domain responses of the same phenomenon, and the magnitude of loss modulus is proportional to the rate of stress decay. From this perspective, the β and α relaxations are one-to-one corresponding to the two stress decay processes. Whether the two stress decay processes or two relaxations can be decoupled to a recognizable degree in the experiments is generally related to temperature, and this
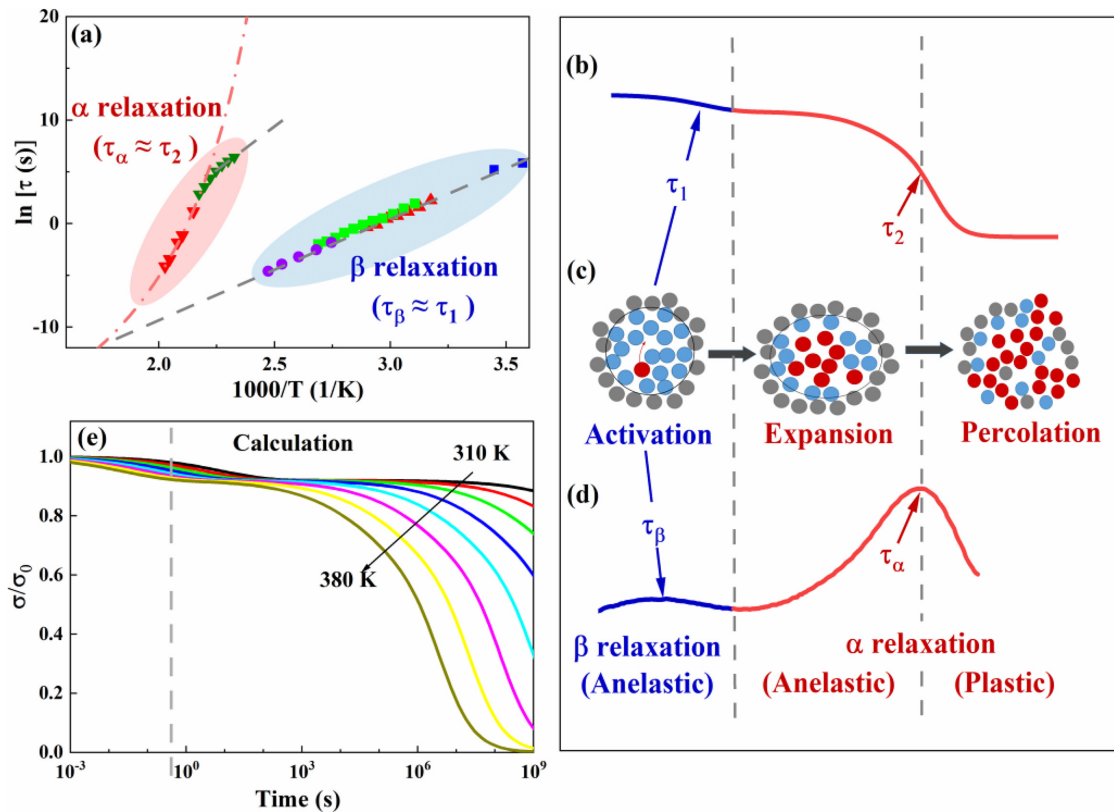
FIG. 4. Correlation between deformation and relaxation based on the evolution of STZs. (a) Temperature dependence of the two-step stress relaxation characteristic times (τ_1 and τ_2) and that of the dynamic relaxations (τ_β and τ_α). (b) Two-step decay of stress at a constant strain. (c) Activation (elementary motion), expansion (atomic motions localized in the interior of STZ due to hard boundary confinement), and annihilation (percolation of atoms due to boundary collapse) of STZ. (d) The α and β relaxation processes identified by DMA. (e) Stress relaxation curves from 310 to 380 K calculated by Eq. (2).

decoupling is due to the different temperature dependence of the characteristic time of the two relaxation processes. what we understand as decoupling between β and α relaxation processes is the recognizable separation of the contributions of two relaxation modes in the relaxation spectrum or deformation process. Taking the $La_{60}Ni_{15}Al_{25}$ glass system in this work as an example, although the relaxation process is already split above T_g in Fig. 4(a), the stress decay under constant strain near the glass transition is observed as a single process. This is because the two relaxation behaviors have quite broad relaxation time distributions and they can only be observed as separate processes if their time scales are very different. As the temperature decreases, the degree of coupling between the two relaxations decreases, and the separation becomes obvious. In $La_{60}Ni_{15}Al_{25}$, the two-step stress decay phenomenon is only observed below 360 K ($0.78T_g$). However, we also noticed that in the $Zr_{50}Cu_{40}Al_{10}$ metallic glass, there is no two-step stress decay phenomenon within the temperature range $0.65-0.79T_g$. This single-step stress decay of $Zr_{50}Cu_{40}Al_{10}$ metallic glass in the deep glass state is likely due to its Johari-Goldstein (JG) β relaxation exhibiting an excess wing rather than a distinct peak in the dynamic mechanical spectrum. According to previous literature [31], the characteristic time of the excess wing, also interpreted as inherent JG β relaxation, always appears to closely follow the α-relaxation time, exhibiting super-Arrhenius temperature

dependence, and never seems to strongly decouple from the α relaxation.

One potentially controversial point is whether there is a correlation between the atomic rearrangement processes involved in the two relaxation processes. However, based on the two-step deformation process and its corresponding relationship with β and α relaxation, we believe that whether β relaxation is responsible for elementary motion is not the focus of current work. There is a sequence between the two deformation processes, and their contributions can be separated in the low-temperature region, so it is highly likely that there is a correlation between them. Therefore, we agree that β relaxation is the precursor of α relaxation, and α relaxation is composed of a group of consecutive Johari-Goldstein β relaxations.

After determining the corresponding relationship between the two relaxation processes and the two-stage phenomenon, the microscopic atomic mechanism can be further discussed. This atomic mechanism should be closely related to the deformation process, involving reversible or irreversible atomic rearrangements. It is well known that under external stress, the deformation of glassy materials may be elastic, anelastic, or plastic, implying different types of atomic motion [32]. The plastic deformation can be viewed as the stress-driven α relaxation in which global irreversible atomic rearrangements occur. Such primary α relaxation can be interpreted as

consisting of a group of consecutive β relaxations involving local reversible atomic arrangements [33]. As claimed by Johnson and Samwer [22], the isolated STZ transitions confined in the elastic matrix are associated with the β relaxation, while percolation of these transitions leads to the collapse of the confining matrix.

The structure of metallic glasses is considered to be heterogeneous, including solid- and liquidlike regions at the nanoscale, as confirmed by experiments [34–36] and simulations [37–39]. The interaction between the two regions before yielding, that is, the elastic back stress applied by the solidlike regions on the liquidlike ones, leads to the anelastic response. This picture has been rationalized by the concept of deformation units, such as the core-shell concept [40] or STZ theory [20]. From the perspective of viscoelastic mechanics, the stress relaxation method used in this paper is a typical transition from reversible deformation to irreversible deformation [41]. Therefore, in addition to the demonstrated relationship between the two-step stress relaxation and the two dynamic processes, it is possible to further establish their correlation with atomic motions related to STZ evolution during nonelastic deformation. The above results in Fig. 3 have shown that the β relaxation mediated fast-decay process is indeed a reversible anelastic process at the macroscopic level. Its characteristic time decreases following the Arrhenius law as temperature increases, eventually becoming undetectable at the experimental time scale. We also observed that the stress decay caused by the β relaxation during deformation is only about 5%, which is far less than the anelastic component ($>30\%$) of the amorphous solids in some previous literature [29,42]. Therefore, part of the anelastic component should be derived from the contribution of the second deformation process controlled by the α relaxation.

The analysis of the deformation mechanism yields a physical picture that once again highlights the correspondence between the two relaxation processes and the two-step decay. Initially, under the external thermomechanical stimuli, the system undergoes local atomic rearrangements, which are macroscopically reversible and exhibit anelastic characteristics. This initial process is controlled by the β relaxation, which may be interpreted as the activation of STZ deformation units. This point has been justified by Yu *et al.* [21] showing that the β relaxation and STZ activation have the same activation energy. For this process to be possible in a glassy solid, the atoms localized in an STZ site should be the ones that can move or jump more easily. It should be noted for STZ the volume character is less important, but local shear modulus is the main characteristic [18,43]. Subsequently, the atomic cluster within the STZ expands further, producing a larger anelastic component. This expansion may occur through continuous β relaxation transitions, and then it can be viewed as part of the α relaxation rather than β relaxation. This is slightly different from the view of Johnson *et al.* [22]. Last, the expansion of STZs may be such that their boundaries impinge, canceling the elastic energy of the boundaries and resulting in macroscopic irreversible deformation.

According to the above analysis, we give a possible physical picture [Figs. 4(b)–4(d)] involving relaxation, deformation, and their potential microscopic atomic mechanisms. The β relaxation and its induced stress decay corresponds

to the activation of STZs, at which time the deformation is reversible. However, the α relaxation and its mediated deformation correspond to the expansion and percolation of STZ. The expansion of STZs is responsible for the anelastic and still reversible deformation, while the percolation of STZs drives the transition from anelasticity to plasticity. It should be noted that, considering the microscopic heterogeneity of metallic glasses, the characteristic time corresponding to any of the above processes is widely distributed, and the characteristic time observed in the experiments has to be regarded as a statistical (Boltzmann) average of the distribution of times involved in the processes.

According to the literature [22,44], the evolution of an isolated STZ may involve local rearrangement of dozens or even hundreds of atoms. The β relaxation related to the activation of STZs should then involve localized jumps of only a few atoms. As envisaged by Palmer *et al.* [45], these atomic motions should be hierarchically constrained. Perez [46] investigated the hierarchical dynamics and relaxation behavior of amorphous polymers and established a correlation between the two relaxation processes:

$$\tau_\alpha = \left(\tau_\beta / t_0^{1-\chi}\right)^{1/\chi}, \tag{1}$$

where t_0 is the scaling parameter that is related to the shift between two relaxations. χ is a correlation factor ranging from full order ($\chi = 0$) to full disorder ($\chi = 1$). Equation (1) is formally identical to the basic formula originally proposed by Ngai [7] in the coupling model. According to relevant descriptions, in polymeric systems the two-step relaxation consists of a short-term exponential relaxation and long-term stretched exponential relaxation, and this behavior is related to the transition from monomer relaxation to multibody relaxation. The two-step decay phenomenon demonstrated in metallic glasses in this paper is primarily a result of atomic rearrangement-induced deformation. As such, theoretical calculations of this behavior must consider the contributions from various deformation components associated with the evolution of STZs. In our recent work [32], by analyzing the different deformation components caused by the β and α relaxation, the macroscopic deformation of amorphous solids can be calculated by the following equation:

$$J(t) = \frac{1}{E_{\text{el}}} + A_\beta \left[1 - \exp\left(-\frac{t}{\tau_\beta}\right) \right]$$
$$+ A \left\{ 1 - \exp\left[-\frac{1}{\chi}\left(\frac{t}{\tau_\alpha}\right)^\chi \right] \right\} + A\frac{t}{\tau_\alpha}, \tag{2}$$

where $J(t)$, which is the ratio of strain to stress, can be expressed as the sum of elastic deformation, deformation controlled by β relaxation, anelastic deformation, and plastic deformation. Using the parameter values identified in Ref. [32], we are able to calculate the stress response for a given strain step. As demonstrated in Fig. 4(e), the two-step stress relaxation behavior can be well reconstructed. By specifying an appropriate cutoff time (shorter than this time the relaxation process may not be accurately identified), the transition from two-step relaxation to one-step relaxation can be observed.

IV. CONCLUSION

In summary, by identifying about 5% of stress decay mediated by the β relaxation in stress relaxation experiments of a $La_{60}Ni_{15}Al_{25}$ metallic glass at room temperature or lower, we separate the respective contributions of the β relaxation and α relaxation. The β relaxation corresponds to the activation of STZs, which is a macroscopic reversible process. On the other hand, the α relaxation mediated deformation corresponds to the expansion and percolation of STZs, where the former is responsible of the anelastic deformation and the latter involves the transition from anelasticity to plasticity. This provides a bridge between the relaxation dynamics and the macroscopic deformation. Through theoretical analysis of possible atomic mechanisms involving dynamic relaxation and deformation, we reconstruct this two-step decay curve during stress relaxation and its transition to the one-step decay. Another significance of the current work is to provide a method for investigating β relaxation at room temperature or even lower. Under this condition, the characteristic time of the β relaxation can reach hundreds or even thousands of seconds and the interference of structural relaxation can be almost avoided.

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