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Strain glass-induced switch from transformation plasticity to reversible superelasticity in Ti-Zr-Ni-Cu alloys

Chao Song^{a,d}, Shuai Ren^b, Shaohui Li^{id a}, Shengwei Li^a, Guijun Liu^a, Zhihua Nie^c, Yandong Wang^{a,d} and Daoyong Cong^{a,d}

^aBeijing Advanced Innovation Center for Materials Genome Engineering, State Key Laboratory for Advanced Metals and Materials, University of Science and Technology Beijing, Beijing, People's Republic of China; ^bShenzhen Key Laboratory of High Performance Nontraditional Manufacturing, College of Mechatronics and Control Engineering, Shenzhen University, Shenzhen, People's Republic of China; ^cSchool of Materials Science and Engineering, Beijing Institute of Technology, Beijing, People's Republic of China; ^dInstitute for Materials Intelligent Technology, Liaoning Academy of Materials, Shenyang, People's Republic of China

ABSTRACT

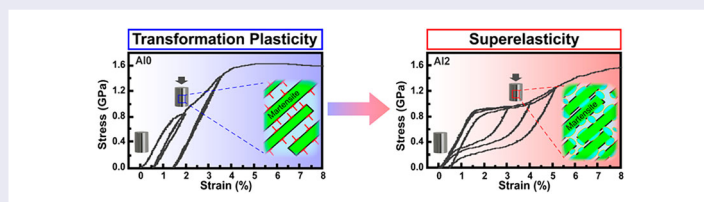
Reversibility of phase transformation, a basic requirement for functional properties of shape memory alloys, such as superelasticity, is severely affected by plastic deformation. Here, strain glass has been utilized to improve the reversibility of phase transformation. The incorporation of Al into Ti-Zr-Ni-Cu induces strain glass and endows the alloy with reversible phase transformation and thus ideal superelasticity. *In situ* x-ray diffraction and dynamic mechanical experiments were utilized to verify the mechanism for the conversion from transformation plasticity to superelasticity. This work provides a new way to improve the reversibility of phase transformation and expands the practical implications of strain glass.

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KEYWORDS

Shape memory alloys; phase transformation; strain glass; superelasticity



IMPACT STATEMENT

Conversion from transformation plasticity to reversible superelasticity is realized by employing the strain glass concept.

1. Introduction

Reversibility of phase transformation is the foundation for functional applications of shape memory alloys (SMAs) such as actuators using shape memory effect, medical implants using superelasticity and solid-state refrigerators using elastocaloric effect [1–4]. These functional properties are all derived from the reversible first-order martensitic transformation (MT) induced by various fields, such as stress and temperature. Nevertheless, the reversibility of phase transformation is influenced by plastic deformation via dislocation slip which occurs when the local stress exceeds the yield strength. This plastic deformation is affected, on the one hand, by the yield strength, and on the other hand, by the compatibility

at interfaces [5,6]. In the case of stress-induced martensitic transformation (SIMT), inferior compatibility leads to stressed transition layer at the interface and could easily result in plastic deformation during SIMT, which is called plasticity accompanying SIMT or, in short, transformation plasticity, while good compatibility reduces or even eliminates the stressed transition layer which helps inhibit plastic deformation and thus leads to superelasticity (SE) as a result of the structural distortion accompanying SIMT.

Frequently, owing to the remarkable structural change from austenite (A) to martensite (M), these two deformation modes (plastic deformation and superelasticity) coexist in one alloy, and a lot of research on SMAs aims

CONTACT Daoyong Cong dycong@ustb.edu.cn Beijing Advanced Innovation Center for Materials Genome Engineering, State Key Laboratory for Advanced Metals and Materials, University of Science and Technology Beijing, 30 Xueyuan Road, Haidian District, Beijing 100083, People's Republic of China

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to inhibit the plastic deformation for optimal superelasticity. The mostly used approach is strengthening the matrix by refining grains and/or introducing precipitates to inhibit dislocation slip [7–9]. Thermal or mechanical cycling can also reduce residual strain by introducing dislocations and redistributing stress fields that impede the generation of new dislocations in subsequent cycles and stabilize the residual martensite [10–12]. Constructing suitable nanocomposite microstructures is also an effective method which can lower the martensite nucleation barrier and decrease the critical stress for SIMT [13,14]. However, these methods usually require intricate processing procedures and also result in some loss of transformation strain due to the increase of the fraction of non-transformable phase.

Theoretically, improving the crystallographic compatibility through modifying crystal structure can reduce plastic deformation. Nonetheless, modifying the structure of austenite and/or martensite without introducing other unfavorable influence poses a formidable challenge. The R phase transformation has been used to enhance the cyclic stability of Ni-Ti alloys. The B2 $\leftrightarrow$ R transformation exhibits high cyclic stability due to smaller transformation strain ($\sim 1\%$) and the complex stacking sequence of R phase impedes the formation of stacking faults [15–17]. Moreover, step-wise phase transformation is anticipated to reduce the lattice mismatch at the interface between the transforming phases. Therefore, it is reasonable to expect that constructing an intermediate structure between austenite and martensite can improve the compatibility between the transforming phases and thus enhance the reversibility of the phase transformation. The strain domains in strain glass, which has been found in various shape memory alloys [18–24], are likely to be a suitable choice of the intermediate structure. In strain glass alloys, only local symmetry breaking occurs while macroscopic symmetry or average structure remains unchanged [18], and the strain domains can be considered to bear an intermediate structure between austenite and martensite. Elemental doping is a simple and effective way to induce strain glass. In this work, we introduced strain glass into the Ti-Zr-Ni-Cu shape memory alloy by doping Al and greatly enhanced the reversibility of phase transformation. Consequently, we successfully turned the transformation plasticity into reversible superelasticity with this strategy.

2. Materials and methods

Alloys with nominal compositions of Ti₂₅Zr₂₅Ni₃₅Cu₁₅ and (Ti₂₅Zr₂₅Ni₃₅Cu₁₅)_{99.8}Al_{0.2} (at.%) (hereafter denoted as Al0 and Al2 alloys, respectively) were prepared by arc melting. The ingots were homogenized

at 1223 K for 24 h followed by water quenching. The phase transformation temperatures were determined by differential scanning calorimetry (DSC, NETZSCH 214 Polyma) at a cooling/heating rate of 10 K/min. Microstructure and orientation analyses were performed using a scanning electron microscope (SEM, Zeiss Supra 55) equipped with electron backscatter diffraction (EBSD) detector (Oxford HKL SEM system) and a transmission electron microscope (TEM, FEI Tecnai G2 F20). The storage modulus and internal friction were measured with a dynamic mechanical analyzer (DMA, TA Q800). Uniaxial compression tests were conducted at various temperatures using a universal testing machine (INSTRON-5966) equipped with a temperature control chamber. The specimens of $\Phi 2 \times 4 \text{ mm}^3$ for compression tests were cut from the ingots with axial direction along the solidification direction (SD). The structure evolution during loading and unloading was studied by *in situ* x-ray diffraction (XRD, Rigaku SmartLab 9 kW, CuK α) experiments using a custom-made loading apparatus. The *in situ* experiments were performed in reflection geometry using specimens of $2 \times 4 \times 1.2 \text{ mm}^3$ with the loading direction parallel to the diffraction plane.

3. Results and discussion

The phase transformation behavior of Al0 and Al2 alloys was characterized by DSC measurements. As indicated from Figure 1(a), the Al0 alloy shows a thermoelastic martensitic transformation, with martensitic and austenitic transformation start and finish temperatures (M_s , M_f , A_s and A_f) being 231, 208, 247 and 276 K, respectively. The thermal hysteresis (defined here as $A_f - M_s$) is 45 K. This wide thermal hysteresis typically implies a large $|\lambda_2 - 1|$ [6], where λ_2 is the middle eigenvalue of the transformation stretch matrix and it is usually used to quantify the crystallographic compatibility: the greater the deviation of λ_2 from 1, the more severe the lattice misfit [25,26]. A striking contrast is that no peaks were observed in the DSC curve of the Al2 alloy (see Figure 1b). This implies that the martensitic transformation, as observed in the Al0 alloy, is suppressed in the Al2 alloy.

Microstructure and orientation characterizations were performed to study the influence of doping. Figure 1(c and d) show the backscattered electron (BSE) images of Al0 and Al2 alloys. Similar distribution and morphology of Ti₂Ni-like precipitates are observed in both alloys. Figure 1(e and f) present the EBSD orientation maps of Al0 and Al2 alloys. Both alloys show similar coarse grains and strong texture with [001] parallel to the solidification direction (SD). These results suggest that there is negligible microstructural difference between the undoped and doped alloys at the micrometer scale.

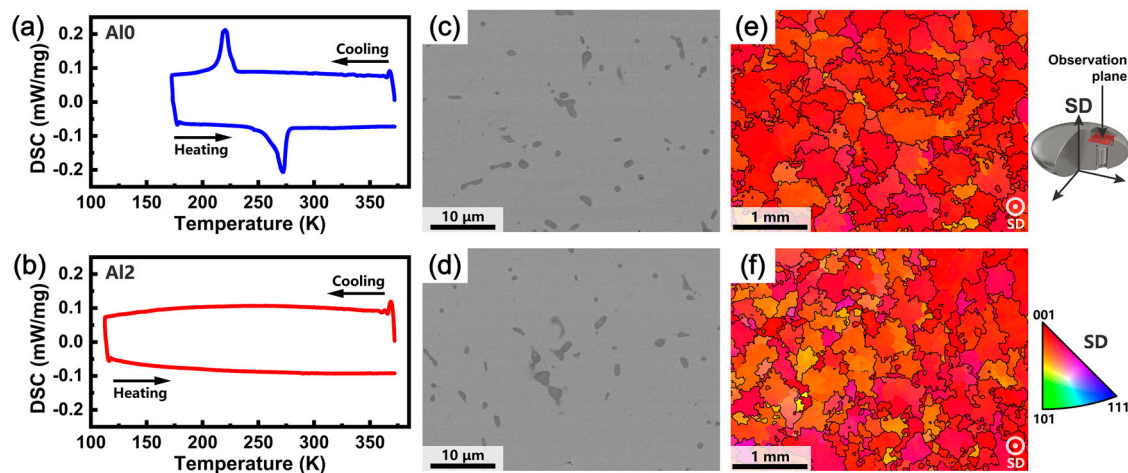


Figure 1. (a, b) DSC curves for the (a) Al0 and (b) Al2 alloys. (c, d) BSE images of the (c) Al0 and (d) Al2 alloys. (e, f) EBSD orientation maps for (e) Al0 and (f) Al2 alloys with the sample geometry and the legend of a stereographic triangle (parallel to the SD) shown beside.

To investigate the mechanical response associated with stress-induced phase transformation, the alloys were subjected to compressive mechanical tests. As shown in Figure 2(a) measured at room temperature (RT), a distinct phase transformation plateau can be observed on the curve of Al0 alloy (as indicated by the arrow), and the critical stress of SIMT (σ_{SIM}) is ~ 780 MPa. Significant plastic deformation is observed under loading-unloading conditions with varying degrees of deformation, and only minor superelastic strain can be obtained. In stark contrast, the Al2 alloy shows ideal superelasticity (see Figure 2b) with a higher σ_{SIM} of 900 MPa. This implies the higher stability of the austenite phase in Al2 alloy, which is consistent with the DSC results. Different from the cyclic deformation behavior of Al0, no residual strain was observed in Al2 when the applied strain did not exceed 4% and ideal superelasticity was achieved. When the applied strain reached 5%, there exists a small residual strain after unloading, yet a large recoverable strain of 4.6% can still be obtained. The obvious stress plateau and stress hysteresis (see Figure 2b) are consistent with the characteristics of first-order martensitic transformation [27,28].

Figure 2(c and d) display the stress-strain curves for compression up to failure measured at room temperature and 423 K, respectively. As indicated from Figure 2(c), the full loading process at room temperature for each alloy consists of four stages: (1) elastic deformation of initial phase, (2) phase transformation, (3) elastic deformation of transformation products and (4) plastic deformation until fracture. As indicated from Figure 2(a), significant plastic deformation occurs during SIMT in Al0 and the introduced defects hinder the phase transformation, which account for its smaller transformation strain and higher work hardening rate at stage (2) as

compared with Al2. Compression tests at a higher temperature (423 K) were performed to investigate the deformation behavior of austenite. As seen from Figure 2(d), both alloys display two deformation stages, i.e. (1) elastic deformation and (2) plastic deformation until fracture, with no SIMT, at 423 K. The yield strength of austenite (σ_A) (Figure 2d) is significantly higher than σ_{SIM} at room temperature (Figure 2a), yet the Al0 alloy shows substantial plastic deformation during SIMT. This suggests that the local stress concentration during the phase transformation process is responsible for the plasticity accompanying SIMT in the Al0 alloy. In addition, σ_A of Al2 is higher than that of Al0, which might be due to the strengthening effect as a result of doping.

The *in situ* loading-unloading XRD measurements were performed in compression mode with a maximum strain of 3.1%, and the experimental setup is shown in Figure 3(c). Figure 3(a) shows the evolution of XRD patterns for the Al0 alloy; the diffraction peaks can be indexed according to B2 austenite and B19' martensite. Before loading, only the austenite diffraction peaks can be observed. As the strain increases to 2.2%, the martensite diffraction peaks, such as $(101)_M$ and $(020)_M$, are clearly observed and the austenite diffraction peak $(300)_A$ disappears, which indicates the occurrence of SIMT. With further loading, the intensities of martensite diffraction peaks increase and more martensite peaks appear. During the unloading process, the $(101)_M$ and $(020)_M$ peaks gradually weaken. Notably, there is still a large residual strain even when the sample was unloaded to zero stress. The martensite diffraction peaks remain after unloading, indicating the presence of residual martensite. As indicated from the zoomed-in view of the austenite diffraction peak $(110)_A$ (see the right panel of Figure 3a), $(110)_A$ remain throughout the whole loading-unloading

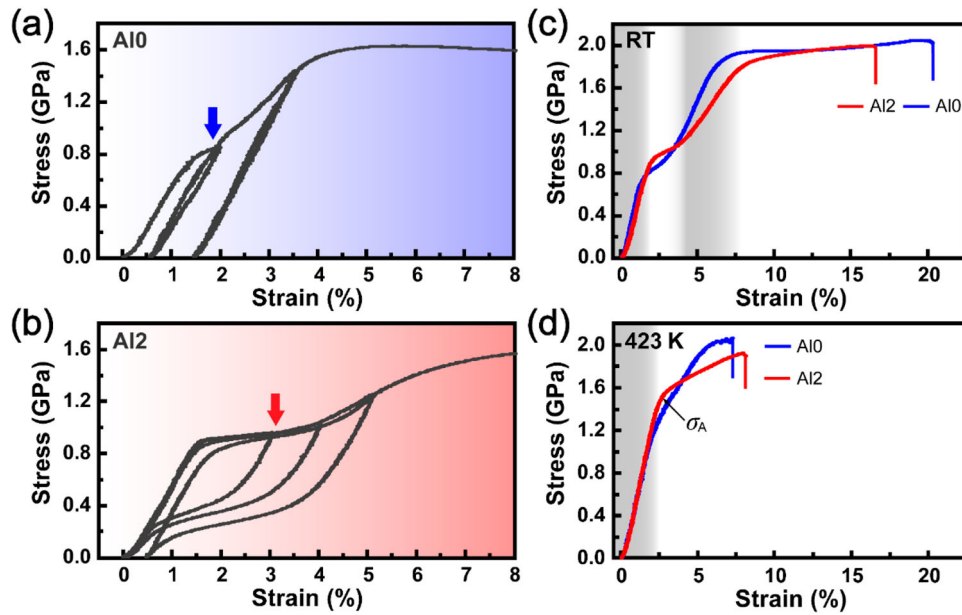


Figure 2. Compressive stress-strain curves of Al0 and Al2 alloys. (a, b) Cyclic compressive stress-strain curves of (a) Al0 and (b) Al2 at room temperature. (c, d) Compressive stress-strain curves of Al0 and Al2 alloys at (c) room temperature and (d) 423 K.

process, suggesting that the stress-induced transformation is incomplete. The non-coincidence of the $(110)_A$ peak positions before loading and after unloading is due to the accumulation of plastic deformation. It is worth mentioning that the intensity of $(110)_A$ exhibits an unusual increase during loading, which might be attributed to grain rotation during plastic deformation. Figure 3(d) shows the evolution of the lattice strain for $(110)_A$ and $(220)_A$ during loading and unloading. As can be seen, the lattice strain cannot be restored to initial state before loading due to the obstruction of dislocations. All these XRD results indicate the apparent irreversibility of the stress-induced transformation in the Al0 alloy, which is consistent with the stress-strain curves in Figure 2(a).

The evolution of XRD patterns for the Al2 alloy is shown in Figure 3(b). The peak positions of Al2 are almost identical to those of Al0, indicating that the austenite and martensite crystal structures of the two alloys are almost the same, with $a_A = 3.132 \text{ \AA}$ for austenite before loading and $a_M = 3.111 \text{ \AA}$, $b_M = 4.170 \text{ \AA}$, $c_M = 4.755 \text{ \AA}$ and $\beta = 98.9^\circ$ for martensite at the maximum strain. Before loading, only austenite diffraction peaks are present. When the sample was loaded to 1.9%, martensite diffraction peaks indexed as $(020)_M$ and $(040)_M$ have appeared and SIMT has started. Upon further loading, the intensity of $(020)_M$ and $(040)_M$ increases and more peaks of martensite emerge. Unlike the Al0 alloy, the evolution of XRD patterns during loading and unloading for the Al2 alloy is fully reversible. During unloading, the intensity of martensite peaks decreases gradually, while the intensity of austenite peaks

increases. When the sample was unloaded to zero stress, the martensite peaks disappear and the austenite peaks return to their original state. Meanwhile, the macroscopic strain becomes zero after complete unloading. As seen from the evolution of lattice strain for $(110)_A$ and $(100)_A$ during loading and unloading (Figure 3e), the lattice strain of the Al2 alloy is fully reversible during the loading-unloading process. All these results show the full reversibility of SIMT in the Al2 alloy, which is consistent with the stress-strain curves in Figure 2(b).

Noteworthy, the austenite peak $(110)_A$ shows abnormal peak broadening in the linear elastic stage of deformation with strain less than 1.4%, as shown in the zoomed-in view in the right panel of Figure 3(b). When the strain is greater than 1.4%, a small shoulder appears on the left-hand side of $(110)_A$. All these peculiarities were also observed in a Ni-Fe-Ga-Co strain glass alloy [29]. Usually, the peak broadening during plastic deformation is considered to be caused by dislocations [29]. However, the peak broadening occurs in the linear elastic stage in our present case, which does not involve dislocation activities. It is believed that the peak broadening during elastic stage in the present case is due to the spatial heterogeneity in the modulus of different nanodomains in strain glass state [29]. Furthermore, the small shoulder emerging during further loading is believed to be related to the preferential growth of nanodomains under stress. Overall, the peak broadening in the linear elastic stage and the emergence of the small shoulder upon further loading imply the existence of strain glass state in the Al2 alloy [29,30].

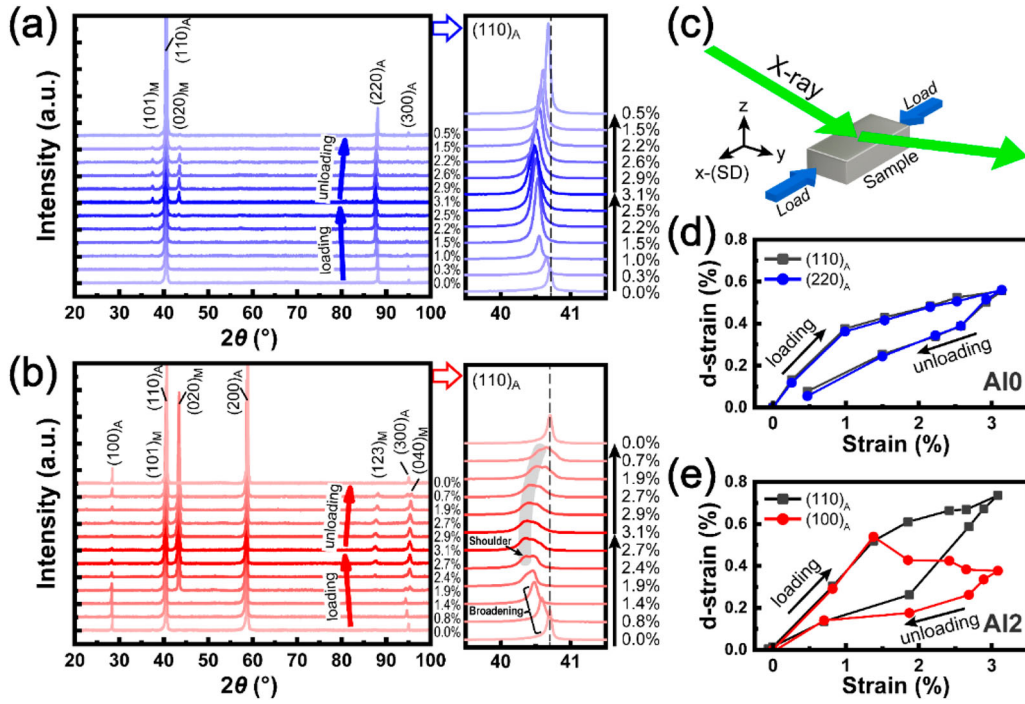


Figure 3. (a, b) XRD patterns of (a) Al0 and (b) Al2 alloys during loading and unloading at room temperature; zoomed-in views are shown in the right panel. (c) Schematic diagram for the *in situ* XRD experiment. (d, e) Lattice strains in the direction transverse to the loading direction associated with several B2 reflections of (d) Al0 and (e) Al2 alloys.

With the lattice parameters of austenite and martensite, we calculated the value of λ_2 to examine the crystallographic compatibility between austenite and martensite. We found that the λ_2 of both alloys (Al0 and Al2) deviates far from 1 ($\lambda_2 = 0.9412$), suggesting inferior crystallographic compatibility between austenite and martensite. The inferior crystallographic compatibility accounts for the plasticity accompanying SIMT in the Al0 alloy. Although the Al2 alloy also exhibits inferior crystallographic compatibility between austenite and martensite, it displays fully reversible superelasticity. This is probably because there exists an intermediate structure like strain nanodomains between austenite and martensite which modifies the transformation path from austenite to martensite.

Dynamic mechanical testing is often used to verify the existence of strain glass. Figure 4(a and b) compare the dynamic mechanical responses (storage modulus and internal friction $\tan \delta$) of the Al0 and Al2 alloys. For the Al0 alloy (see Figure 4a), the storage modulus exhibits a dip around M_s , and a corresponding peak appears in the $\tan \delta$ curve at a slightly lower temperature. Both the dip temperature of storage modulus and the peak temperature of internal friction are frequency independent, which is characteristic of martensitic transformation [31]. The Al2 alloy shows a different dynamic mechanical response. As shown in Figure 4(b), the storage modulus shows a dip and the $\tan \delta$ curve exhibits a

peak. Unlike the Al0 alloy, the dip temperature of storage modulus and the peak temperature of $\tan \delta$ for the Al2 alloy display prominent frequency dependence, which is a typical feature of strain glass transition [18]. The dip temperature of storage modulus (T_g) as a function of frequency (ω) obeys the Vogel–Fulcher relation [18],

$$\omega = \omega_0 \exp[-E_a/k_B(T_g - T_0)] \quad (1)$$

where ω is the frequency, ω_0 is the frequency prefactor, E_a is the activation energy, k_B is the Boltzmann constant, T_g is the strain glass transition temperature, and T_0 is the ideal freezing temperature (i.e. T_g at 0 Hz). The fitting yields a T_0 of ~ 173 K (inset of Figure 4b). Upon further cooling, a frequency-independent peak appears on the $\tan \delta$ curves at ~ 193 K, which suggests a spontaneous transition from strain glass to martensite [31,32]. We further conducted TEM observations at room temperature. As shown in Figure 4(c), the selected-area diffraction pattern (SADP) for the Al0 alloy shows sharp reflections of B2 phase, with no diffuse reflections. In contrast, the SADP for the Al2 alloy shows faint diffuse reflections, as marked by the red circle in Figure 4(d). The corresponding dark-field image (Figure 4e) reveals the presence of nanodomains in Al2. The above features confirm the presence of strain glass in the Al2 alloy.

Based on above results and analyses, it is reasonable to conclude that the introduction of strain glass

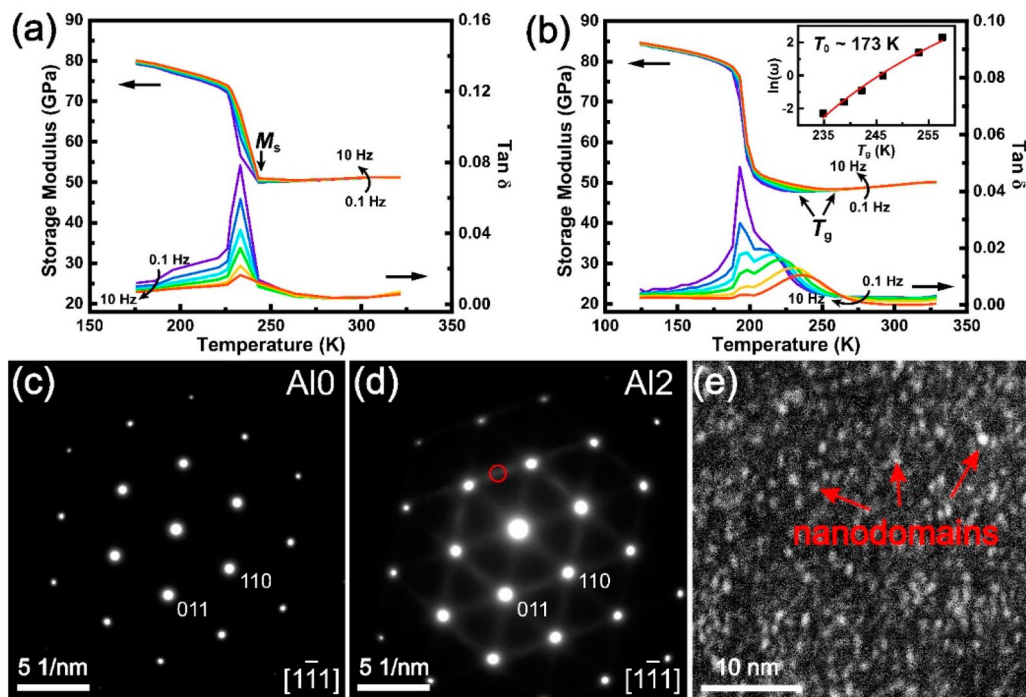


Figure 4. (a, b) Temperature dependence of storage modulus and internal friction ($\tan \delta$) measured at different frequencies for (a) Al0 and (b) Al2 alloys. Inset of (b) shows the T_g vs $\ln(\omega)$ curve fitted by the Vogel-Fulcher relation. (c, d) Electron diffraction patterns along $[1\bar{1}1]_{B2}$ zone axis taken at room temperature for (c) Al0 and (d) Al2 alloys. (e) Dark-field TEM image obtained by using the diffuse spot marked by a circle in (d) for Al2 alloy.

nanodomains altered the reversibility of phase transformation. Figure 5 illustrates the schematic diagrams of microstructural evolution during the loading and unloading process for the Al0 and Al2 alloys. For the Al0 alloy, elastic deformation of austenite occurs at a low stress level. As stress increases, martensite nucleation and growth take place, and meanwhile dislocations form at the austenite-martensite interface owing to the inferior compatibility between austenite and martensite. The accumulation of dislocations creates resistance to the phase transformation and results in stabilization of martensite. Upon unloading, the reverse transformation is inhibited and there exists substantial residual strain. This leads to the plasticity accompanying SIMT. For the Al2 alloy, doping results in the formation of randomly distributed strain nanodomains which manifest the local strain order. At a low stress level, the preferentially aligned strain nanodomains grow in size at the expense of the others. As stress further increases, some of the preferentially aligned nanodomains evolve into long-range strain-ordered martensite while others continue to grow. The strain nanodomains serve as an intermediate structure to mitigate the incompatibility between austenite and martensite. In this way, the plastic deformation during stress-induced transformation is suppressed and the phase transformation becomes reversible. Upon unloading, the martensite and strain nanodomains revert

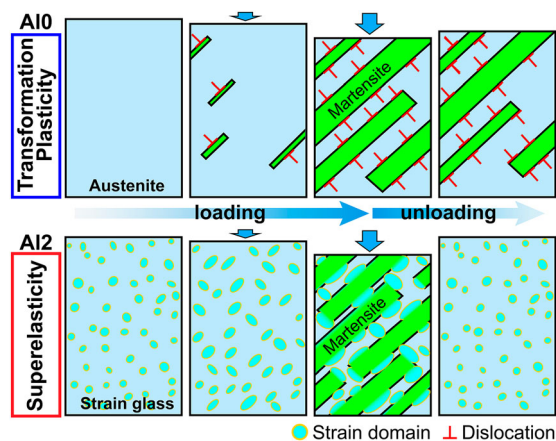


Figure 5. Schematic diagram of microstructure evolution in different alloys during loading and unloading.

back to the initial state of strain glass with randomly distributed nanodomains. This deformation behavior during loading and unloading leads to ideal reversible superelasticity (SE) in the Al2 alloy.

4. Conclusions

The strain glass state was introduced to enhance the reversibility of phase transformation in Ti-Zr-Ni-Cu

alloys. The undoped alloy shows significant plastic deformation during stress-induced martensitic transformation due to the inferior compatibility between austenite and martensite. Doping with Al leads to the formation of strain nanodomains which serve as an intermediate structure to mitigate the incompatibility between austenite and martensite. As a result, the plastic deformation during stress-induced transformation is suppressed and the stress-induced transformation becomes reversible, leading to the ideal superelasticity. In this way, conversion from transformation plasticity to reversible superelasticity is realized by employing the strain glass concept. It is believed that this strategy is readily applicable to a wide range of phase-transforming materials to enhance the reversibility of phase transformation. The present work opens a new avenue for developing advanced phase-transforming materials with reversible functional properties.

Disclosure statement

No potential conflict of interest was reported by the author(s).

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ORCID

Shaohui Li  <http://orcid.org/0000-0003-3078-0338>

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