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# Strain glass in ${\rm Ti}_{50\mbox{-}x\mbox{-}y}{\rm Ni}_{50\mbox{+}y}{\rm Nb}_y$ alloys exhibiting a boson peak glassy anomaly

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ABSTRACT

understand the glass nature of strain glass. Shape memory alloys (SMAs) exhibit distinctive shape memory effect and superelastic behavior, and thus have been widely used in industrial and medical fields [1,2]. All these properties are tightly related to the martensitic transformation in SMAs. The martensitic/ferroelastic transformation is a diffusionless structural phase transformation in which the parent phase undergoes lattice shear strain long-range ordering and transforms into a martensitic phase [3]. As a result, a conventional martensitic transformation (or a ferroelastic transformation) is associated with the formation of long-range ordered martensitic domains [4]. However, when the martensitic transformation is suppressed by some type of defects such as point defects [5], dislocations [6] and nanoprecipitates [7], the long-range ordering of the lattice strain fails to take place, while a new state with lattice strain short-range ordering is frozen at low temperature, denoted as strain glass [7,8].

Strain glass is characterized by some typical glassy features similar to the relaxor in ferroelectric systems and the spin glass in ferromagnetic systems [9–12], including no calorimetric change, unchanged average structure as well as the slowing-down of dynamics during the glass transition. Moreover, strain glass also exhibits many novel properties,

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such as high damping [13], Invar and Elinvar effects [14,15], slim superelasticity [16] and low-field-triggered large magnetostriction [17]. Even though some new methods to generate strain glass have been developed [4–7], doping sufficient point defects is still the main way to obtain strain glass. Through this way, strain glass has been found in various shape memory alloy systems such as Ti-Ni-based, Ti-Pd-based, Ti-Au/Pt-based and Ni-Mn-Ga-based SMAs [18–24].

Strain glass, which is a type of ferroic glass with lattice strain short-range ordering, has been found in many shape

memory alloy systems, while its unique properties still await to be investigated. Here we report a strain glass

found in Ti<sub>50-x-v</sub>Ni<sub>50+v</sub>Nb<sub>x</sub> due to the synergistic effect of Ni and Nb atoms and its boson peak glassy anomaly. It

is found that Nb atoms can only slightly suppress the martensitic transformation, but with the help of 1 at%

excess Ni atoms (y=1), 2% Nb atoms (x=2) are enough to lead the system to the strain glass state. A three-

dimensional phase diagram is established accordingly. Moreover, a boson-peak-like glassy feature is also found in the strain glass state, which resembles the boson peak in metallic glass. This work provides more clues to

Despite that strain glass has been found in many SMAs, the nature of strain glass is still full of mystery. In particular it is still hard to understand the glass nature of strain glass, as compared with the structural glasses such as metallic glass, because metallic glass corresponds to a disorder of atomic packing while strain glass possesses a crystalline structure. Intriguingly, a recent work has reported that strain glass possesses glasslike phonon damping above the transition [25]. More importantly, this work links the formation of strain glass to the electronic structure near the Fermi surface, thus providing the most fundamental perspective to understand the origin of strain glass [25]. On the other hand, a boson-peak-like glassy anomaly has been found in strain glass that resembles the boson peak in metallic glass, a well-known fingerprint universally found in structural glasses [26]. This work

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**Fig. 1.** (a-c) X-ray diffraction patterns of  $Ti_{44}Ni_{50}Nb_6$ ,  $Ti_{43}Ni_{51}Nb_6$  and  $Ti_{42}Ni_{52}Nb_6$  respectively. (d-e) SEM images of  $Ti_{44}Ni_{50}Nb_6$ ,  $Ti_{43}Ni_{51}Nb_6$  and  $Ti_{42}Ni_{52}Nb_6$  after solution-treated for 10h (labeled as ST-10h), respectively. (g) SEM and EDS images of  $Ti_{44}Ni_{50}Nb_6$  with no solution treatment (no-ST). (h) SEM and EDS image of  $Ti_{44}Ni_{50}Nb_6$  with solution treatment.

confirms the glasslike phonon damping in strain glass by molecular dynamics simulations and also sheds new light on the nature of strain glass.

Among the Ti-Ni-based ternary SMAs, the Ti-Ni-Nb SMA has received a lot of attention in recent years and are widely used in aerospace, mechanical and automatic control systems because of its excellent properties, such as shape memory effect, superelasticity, high damping properties and high yield strength [19,27,28]. Large elastic strain, low modulus and high strength are achieved by engineering Nb nanowires into Ti-Ni SMA [29]. Meanwhile, the Ti-Ni-Nb alloy has a wide phase transformation temperature hysteresis, which is important for coupling and sealing [30,31]. On the other hand, strain glass has been found in this system [19], but a systematic investigation on the strain glass in this system is still lacking.

In this work, we investigate the evolution of the transformation behavior by the influence of both Ni and Nb atoms in  $Ti_{50-x-y}Ni_{50+y}Nb_x$ and establish the three-dimensional phase diagram of this strain glass system. It is found that Nb atoms can only slightly decrease the martensitic transformation temperature ( $M_s$ ), but 2% Nb atoms (i.e., x=2) are enough to lead the system to the strain glass state with the help of 1 at% excess Ni atoms (i.e., y=1). At last, the low temperature specific heat ( $C_p$ ) of this system is studied, and a boson-peak-like anomaly is found. This work provides some more clues to understand the nature of strain glass.

Alloy buttons of  $Ti_{50-x-y}$ - $Ni_{50+y}$ - $Nb_x$  (x=2-6, y=0-2) were prepared from the pure metals (> 99.95 at%) by arc-melting under an Ar atmosphere on a water-chilled copper hearth. The alloy buttons were flipped over and remelted 8 times to ensure good homogeneity and cast into a copper mould with dimensions of 20 mm \* 1.5 mm \* 80 mm. The sheets were solution-treated at 1273K for 10 hours in a vacuum quartz tube, followed by water quenching. Subsequently, the specimens were mechanically polished and cut into shapes suitable for different measurements.

X-ray diffraction (XRD, RIGAKU miniflex600) with Cu Ka radiation was used to detect crystal structure at room temperature. Possible structural change during cooling was detected by in-situ XRD experiments in an XRD (Shimadzu XRD 7000) with a cooling holder. Scanning electron microscope (SEM, Fei quanta FEG 450) instrument equipped with energy disperse spectroscopy (EDS) was used to observe the phase and element distribution. The differential scanning calorimeter (DSC, TA-Q200) experiments were carried at a cooling/heating rate of 10 K/ min. Electrical resistivity (ER) measurements were performed by the four-probe method with a constant current of 100 mA and a cooling/ heating rate of 3 K/min. Potential strain glass transitions were tested in a single cantilever mode on a Dynamic Mechanical Analysis (DMA, TA-Q800) with an amplitude of 5 µm, a cooling rate of 2 K/min and a frequency range from 0.2 to 20 Hz. The specific heat of the samples was measured in a physical property measurement system (PPMS dynaCool) from Quantum Design through a thermal relaxation method from 150 K to 2 K.

Figs. 1a-c show the XRD curves of  $Ti_{44-y}Ni_{50+y}Nb_6$  (y = 0, 1, 2) alloys at room temperature. It can be seen that they all have the same diffraction peaks, which is consistent with a standard B2 structure. No other obvious peaks can be found in Figs. 1a-c, indicating all the alloys possess a pure B2 phase in average structure. In addition, the microstructure of the alloy was observed by SEM. As shown in Fig. 1d-f, some



**Fig. 2.** (a-c) DSC curves of  $Ti_{50-x}Ni_{50}Nb_x$ ,  $Ti_{49-x}Ni_{51}Nb_x$  and  $Ti_{48-x}Ni_{52}Nb_x$  (x=2, 4, 6) upon cooling and heating. (d) Electrical resistivity Curves during cooling and heating. (e-f) In-situ XRD profiles of  $Ti_{48}Ni_{50}Nb_2$  and  $Ti_{47}Ni_{51}Nb_2$  at various temperatures.

precipitates can be found in the matrix of the  $Ti_{44.y}Ni_{50+y}Nb_6$  (y = 0, 1, 2) alloys with the solution treatment at 1273 K for 10 hours. EDS images of the  $Ti_{44}Ni_{50}Nb_6$  alloy was observed, as shown in Fig. 1g and h. Before the solution treatment, large size of precipitates that are rich in Nb elements, can be clearly observed in the matrix. In comparison, after the solution treatment, no obvious precipitates can be found in the matrix and the elements are uniformly distributed. As a result, the alloy can be regarded as a homogeneous material after the solution treatment. In order to eliminate the impacts of precipitates, the samples after the solution treatment are used to perform subsequent tests.

The evolution of the phase transformation behavior and the associated structure change in  $Ti_{50-x-y}Ni_{50+y}Nb_x$  alloys are summarized in Fig. 2. Fig. 2a shows the variation of DSC curves for  $Ti_{50-x}Ni_{50}Nb_x$  (x = 2, 4, 6) alloys. With the increase of Nb content, the  $M_s$  moves downward drastically. In addition, the exothermic peak becomes weaker and broader, revealing that the change of transformation enthalpy decreases with Nb doping. This result indicates that the substitution of Nb atoms for Ti sites suppress the martensitic transformation of Ti-Ni-Nb alloys. However, the suppressing effect of the substitution of Nb atoms for Ti sites is weaker than that of the substitution of other elements (such as Mn, Cr and Co) for Ni sites [21]. Interestingly, when Ni atoms are introduced into the system, Nb atoms are able to completely suppress the martensitic transformation. It has been reported that the  $Ti_{49}Ni_{51}$ alloy undergoes a B2-B19' martensitic transformation at 234 K [23,32]. In comparison, as shown in Fig. 2b, no heat flow peaks can be observed in the  $Ti_{49-x}Ni_{51}Nb_x$  alloys where x = 2-6, which means that a low content of Nb atoms are enough to completely prohibit the formation of the martensite with the help of the excess of 1 at% Ni atoms. When the content of Ni atoms further increases, no heat flow peaks are observed with the increase of Nb atoms either in Fig. 2c. The evolution of heat flow peaks in the DSC curves indicates that Nb and Ni elements have a synergistic effect on the inhibition of phase transformation, which is consistent with the results of previous studies [23,33].

The variation of the ER curves with  $Ti_{50-x-y}Ni_{50+y}Nb_x$  ( $x = 2\sim 6$ ,  $y = 0\sim 2$ ) alloys is shown in Fig. 2d. In  $Ti_{50-x}Ni_{50}Nb_x$  (x = 2, 4, 6) alloys, there exists a hysteresis loop during heating and cooling, indicating the occurrence of the martensitic transformation. With the increase of the content of Nb, the martensitic transformation still occurs but the  $M_s$  lowers, consistent with the DSC results in Fig. 2a. In  $Ti_{49-x}Ni_{51}Nb_x$  and  $Ti_{48-x}Ni_{52}Nb_x$  (x=2, 4, 6) alloys, no hysteresis loop is observed in the whole temperature range down to 10 K, suggesting that the martensitic transformation is completely suppressed. Meanwhile, the ER curves of



**Fig. 3.** DMA images of storage modulus (E) and internal friction  $(\tan \delta)$  of  $Ti_{50-x-y}Ni_{50+y}Nb_x$  alloys at different x and y, including (a)  $Ti_{48}Ni_{50}Nb_2$ , (b)  $Ti_{46}Ni_{50}Nb_4$ , (c)  $Ti_{44}Ni_{50}Nb_6$ , (d)  $Ti_{47}Ni_{51}Nb_2$ , (e)  $Ti_{45}Ni_{51}Nb_4$  and (f)  $Ti_{43}Ni_{51}Nb_6$ . The inset figures in (d-f) exhibit the V-F law fitting of strain glass transition.

these two systems exhibit an abnormal increase with cooling. This feature is consistent with the characteristic of the strain glass transition in Ti-Ni-based alloys [18].

The in-situ XRD curves in Figs. 2e-f show the structural change of  $Ti_{48}Ni_{50}Nb_2$  and  $Ti_{47}Ni_{51}Nb_2$  alloys with varying temperature. At room temperature, the structure of  $Ti_{48}Ni_{50}Nb_2$  alloy only exhibits a single  $(110)_{B2}$  peak (Fig. 2e1), while at low temperature, the  $(110)_{B2}$  peak splits into multiple small peaks (Figs. 2e2-e3), which are identified as the peaks of the B19' martensitic phase [23], and this result reveals that a B2-B19' martensitic transformation occurs in the  $Ti_{48}Ni_{50}Nb_2$  alloy. In contrast, the  $(110)_{B2}$  peak of  $Ti_{47}Ni_{51}Nb_2$  remains unchanged throughout the considered temperature range from 298K down to 123K, indicating that no phase transformation occurs, as presented in Fig. 2f. Thus, Fig. 2 exhibits that Nb and Ni atoms have a synergistic effect to suppress the martensitic transformation, and the phase transformation completely disappears with 2 at% Nb doping under the help of 1 at% excess Ni atoms.

To further investigate the changes occurring in the nontransformation region, the dynamic mechanical properties were measured by DMA experiments, which are shown in Fig. 3. For the Ti<sub>48</sub>Ni<sub>50</sub>Nb<sub>2</sub> alloy (shown in Fig. 3a), the storage modulus first decreases with cooling and exhibits a dip around 270 K, which is consistent with the  $M_s$  obtained in the DSC and ER curves, corresponding to the B2-B19' martensitic transformation. It can be found both the storage modulus dip temperature and the internal friction peak temperature are frequency independent (i.e., the peak position does not shift when the frequency changes). The other two compositions with the martensitic transformation exhibit two dips in the storage modulus curves in Figs. 3b-c. The dip at lower temperature in each curve locates around the  $M_s$ observed in DSC and ER curves in Fig. 2, which means that this dip corresponds to the martensitic transformation in each alloy. On the other hand, no other signal has been found in DSC and ER curves at the



**Fig. 4.** 3D phase diagram of  $\text{Ti}_{50-x-y}\text{Ni}_{50-y}\text{Nb}_x$  ( $x = 0 \sim 6$ ,  $y = 0 \sim 1$ ) with (a)  $\text{Ti}_{50-x}\text{Ni}_{50}\text{Nb}_x$  and (b)  $\text{Ti}_{49-x}\text{Ni}_{51}\text{Nb}_x$ . The data with x=0 comes from Ref. [23]. The red round points  $T_g/T_0$  are from the data of the DMA results, the black square points  $M_s$  are the data of the DSC experiments. M represents the martensitic phase. STG represents the strain glass state.

temperature range where the dip at higher temperature takes place, which is similar to the results in Ti-Ni-Cu alloys [20], suggesting this modulus dip corresponds to a strain glass transition.

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**Fig. 5.** Specific heat ( $C_p$ ) and thermal parameters in Ti-Ni-Nb alloys. (a) The  $C_p$  curve for each x in Ti<sub>50-x</sub>Ni<sub>50</sub>Nb<sub>x</sub> and Ti<sub>49-x</sub>Ni<sub>51</sub>Nb<sub>x</sub> (x = 2, 4, 6). (b)  $C_p/T$  vs.  $T^2$  relation for each x with the  $C_p$  data below 7 K. The linear fitting follows the formula of  $C_p/T = \gamma + \beta T^2$ . (c) BP-like anomaly of strain glass in the reduced specific heat ( $C_p \gamma T$ )/ $T^3$  as a function of *T*. The hump of strain glass around 10 K is similar to the boson peak of metallic glass. (d) Peak temperatures ( $T_p$ ) and peak values for the six components.

For  $\text{Ti}_{49-x}\text{Ni}_{51}\text{Nb}_x$  (x = 2, 4, 6) alloys which locate in the nontransforming composition region, the DMA results in Figs. 3d-f exhibit different features from those of the martensitic compositions. The storage modulus and internal friction of each non-transforming component have a clear frequency dependence behavior, and the peak temperature of the internal friction (i.e.,  $T_g$ ) as a function of frequency can be fitted by the Vogel-Fulcher relation as shown in the insets of Figs. 3d-f:

$$\omega = \omega_0 \exp\left[-E_a / k_B (T_g - T_0)\right],\tag{1}$$

where  $\omega$  is the frequency,  $\omega_0$  is the frequency pre-factor,  $E_a$  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, which is yielded to be ~152K, 155K and 160K for x = 2, 4, 6 respectively. The frequency dependence behavior is a key piece of evidence of the strain glass transition, and similar behavior can also be found in ferroelectric relaxor transitions and spin glass transitions [9–12]. The above results all boil down to one important fact: the change from a normal martensite transformation to an anomalous strain glass transition in Ti-Ni-Nb alloys when doped with sufficient point defects (excess Ni or Nb atoms in this work).

Combining all the above results, we plotted the three-dimensional phase diagram with the variation of Ni and Nb contents, as shown in Fig. 4. The phase diagram of  $Ti_{50-x}Ni_{50}Nb_x$  (x = 2, 4, 6) alloys is shown in Fig. 4a. It can be clearly seen in the phase diagram that the  $M_s$  decreases with increasing Nb content. This trend suggests that the doping of Nb reduces the thermodynamic stability of the B19' martensite. In contrast, for the  $Ti_{49-x}Ni_{51}Nb_x$  (x = 2, 4, 6) alloys, a strain glass transition occurs, as labelled by the red line of  $T_0$  in the phase diagram in Fig. 4b. With the increase of Nb content, the  $T_0$  shows a slight increase.

Despite that strain glass has been found in many systems, the glass nature is still elusive. As a result, it still needs to further unravel the nature of strain glass. Recently, a work has successfully established a new link between the strain glass and structural glasses by discovering that the strain glass exhibits a boson-peak-like anomaly in  $C_p$  similar to the well-known boson peak in metallic glass [26]. In this work, we also investigated the low-temperature  $C_p$  of this Ti-Ni-Nb system. Fig. 5a exhibits the specific heat curves for different x and y. It can be seen that the strain glass compositions of Ti<sub>49,x</sub>Ni<sub>51</sub>Nb<sub>x</sub> exhibit a higher  $C_p$  than that of martensitic compositions of Ti<sub>50-x</sub>Ni<sub>50</sub>Nb<sub>x</sub>. For non-magnetic metals, the low-temperature specific heat mainly composes of the contribution of electrons and phonons, which can be represented by the relation,

$$C_p = \gamma T + \beta T^3, \tag{2}$$

where the intercept  $\gamma$  is the electronic specific heat coefficient, and the slope  $\beta$  is the calorimetric cubic coefficient. Fig. 5b exhibits a plot of  $C_{\rm p}$ / T vs.  $T^2$  below 7 K, which can be well fitted by the formula (2). After removing the part of electronic specific heat, we then can obtain the behavior of phonon specific heat in a plot  $(C_p - \gamma T)/T^3$  vs. *T*, as shown in Fig. 5c. For the three compositions with the martensitic transformation  $(Ti_{50,r}Ni_{50}Nb_r)$ , there is a peak around 20 K at x=2, which is attributed to the van Hove singularity [26,34,35]. The peak temperature decreases and the peak value increases as the martensitic transformation becomes weak with the increase of x. In comparison, the three strain glass compositions exhibit similar broad humps around 10 K, and thus a large gap between the martensite and strain glass can be clearly seen below 20 K. Fig. 5d summarizes the peak temperature and peak value of these six compositions. Strain glass exhibits a lower peak temperature and a larger peak value, which is the same as the results in the previous work [26]. Therefore, a boson-peak-like glassy feature is also found in this strain glass alloy system. This result further confirms that the boson peak is a universal anomaly in strain glass alloys.

It is of interest to learn the site occupancy of excess Ni and Nb atoms. It is found that the crucial factor determining the site occupancy of alloying elements in Ti-Ni alloys is electron configuration of the alloying element and other important factors include relative chemical affinity, atomic size effect and the design of alloying formulas [36–38]. Since Nb is close to Ti in the periodic table, the chemical affinity of Nb is strong to Ti, rather than to Ni, and the atomic size of Nb is also similar to that of Ti. Thus, Nb has strong preference for entering into Ti-site in principle. However, in Nb-doped nearly equiatomic Ti-Ni ternary alloys, the site occupancy of Nb is also strongly affected by the compositional design [39]. In the case of  $Ti_{50-x/2}Ni_{50-x/2}Nb_x$  and  $Ti_{50}Ni_{50-x}Nb_x$ , Nb tends to equally enter into both Ti-site and Ni-site to form a pseudo-binary "TiNi-Nb" system. Only in the case of  $Ti_{50-x}Ni_{50}Nb_x$ , Nb prefers to mostly enter into Ti-site. Since the compositional design ( $Ti_{50-x-y}Ni_{50+y}Nb_x$ ) in this work is similar to the case of  $Ti_{50-x}Ni_{50}Nb_x$ , Nb is believed to mainly enter into Ti-site, and so does excess Ni due to the alloying formula.

After determining the site occupancy of excess Ni and Nb atoms to Tisite, we try to provide a simple interpretation to the origin of strain glass in this work from the perspective of electronic structure. Previous studies have found that Fermi surface nesting is responsible for the instability of the B2 structure in Ti-Ni based SMAs, and thus related to phonon softening of the transverse acoustic (TA<sub>2</sub>) branch along [110] above the transformation [40–42]. On the other hand, it is noted that the recent work has reported that the disruption of Fermi surface nesting, rather than transformation strain pinning, is responsible for strain glass formation [25]. As a result, we suggest the formation of strain glass in this work is also related to the disruption of the nesting effect of Fermi surface by excess Ni and Nb atoms, which serve as point defects to create localized electronic states to break up the nesting feature and cause the breakup of the well-defined transition wavevector into a multi-scale distribution.

In summary, we established the three-dimensional strain glass phase diagram with Ni and Nb content and found the boson peak anomaly in the Ti-Ni-Nb system. By varying the Ni and Nb atomic contents, we reveal the synergistic effect of Ni and Nb elements on the suppression of the martensitic transformation and the appearance of the strain glass transition. Accordingly, a three-dimensional phase diagram is established. Moreover, a boson peak like anomaly is also found in the strain glass of this alloy system, which suggests strain glass may share more glassy features similar to the structural glasses. This work may provide some more clues to the in-depth understanding of the glass nature of strain glass.

### **Declaration of Competing Interest**

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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