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Negative temperature-dependence of stress-induced R→B19' transformation in nanocrystalline NiTi alloy

Taotao Wang^{a,b}, Xiangxiang Rao^a, Qiang Zhang^a, Daqiang Jiang^a, Yang Ren^c, Lishan Cui^a and Kaiyuan Yu^a

^aDepartment of Materials Science and Engineering, China University of Petroleum-Beijing, Beijing, People's Republic of China; ^bSchool of Materials Science and Engineering, Shaanxi University of Technology, Hanzhong, People's Republic of China; ^cDepartment of Physics, City University of Hong Kong, Kowloon, Hong Kong, People's Republic of China

ABSTRACT

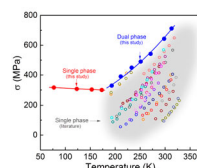
The temperature dependence of the critical stress of R→B19' transformation in NiTi shape memory alloys remains unclear due to the difficulties in precisely identifying the phase constitution and acquiring sole R phase. Here we investigate the thermally- and stress-induced martensitic transformation in a near-equiatomic nanocrystalline NiTi alloy. *In situ* synchrotron X-ray diffraction is used to identify the phase evolution such that the temperature window for sole R phase is firmly determined. We find that the temperature dependence of R→B19' is negative (−0.2 MPa/K) in our alloy, in sharp contrast to 3–5 MPa/K reported. Possible reasons for the discrepancies are discussed.

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KEYWORDS

R phase; NiTi; temperature dependence; martensitic transformation; synchrotron XRD



IMPACT STATEMENT

Negative temperature-dependence of stress-induced R→B19' transformation was discovered for the first time in NiTi alloys, being in sharp contrast to previous reports. This calls for a revisit of such transformation.

1. Introduction

The temperature dependence of critical stress ($d\sigma/dT$) is essential for the understanding of martensitic transformation physics, because it contains thermodynamic and crystallographic terms as described by the Clausius-Claperyon equation $d\sigma/dT = -\Delta S/\epsilon V_m$, where ΔS is entropy difference between the parent and martensite phase, ϵ is transformation strain and V_m is molar volume [1–3]. The parameters on the right are typically constant once the structures of the parent and martensite phase are known [1–3]. For NiTi SMAs that undergo R→B19' stress-induced martensitic transformation (SIMT), $d\sigma/dT$ (R→B19') is typically 3–5 MPa/K [4–8]. It appears that the values of $d\sigma/dT$ (R→B19') have been well-recognized (very excellent repeatability) and the underlying physics has been well-understood [4–17].

However, it was argued that the specimens used were probably consisted of both R and B2 phase [12,16]. In this regard, $d\sigma/dT$ measured was associated with both R→B19' and B2→B19' (6–8 MPa/K) SIMT, instead of R→B19' SIMT by itself [12,16]. If so, the true value of $d\sigma/dT$ (R→B19') remains unclear and the current understandings on the SIMT physics of NiTi may be incomplete.

To obtain the true value of $d\sigma/dT$ (R→B19'), the phase constitution of the specimen must be firmly identified in the first place. This requires a precise determination of the finishing temperature of B2→R transformation (i.e. R_f), below which single R phase is present. So far R_f has been mostly determined by calorimetric, electrical or lab source X-ray diffraction (XRD) measurements [12,16–29]. Nevertheless, the setbacks of these methods

CONTACT Kaiyuan Yu ✉ kyyu@cup.edu.cn Department of Materials Science and Engineering, China University of Petroleum-Beijing, Beijing 102249, People's Republic of China; Lishan Cui ✉ lsui@cup.edu.cn Department of Materials Science and Engineering, China University of Petroleum-Beijing, Beijing 102249, People's Republic of China

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have been suggested [12,16]. For instance, it has been shown that an ongoing R transformation occurs gradually below R_f determined by differential calorimetric scanning (DSC) [12,16–21,29]. Such an ongoing transformation is usually (and reasonably to some extent) ignored because the heat flow during this stage (i.e. the long ‘tail’ towards lower temperatures in the DSC curve) does not change significantly with temperature as it does within the DSC peak [12,16–21,29]. For similar reasons, the electrical method is limited by not being able to differentiate the resistance variation of R lattice distortion from that of B2→R transformation [22–24]. Lab source XRD is more reliable by comparison since it measures R_f by examining crystal structures, but the problem remains because the major XRD peaks of B2 and R phase (e.g. B2 (110) and R (300) or R (112)) are too close to distinguish when the latter is dominant in volume fraction [25–28]. For these reasons, the measurement of R_f and $d\sigma/dT(R\rightarrow B19')$ in NiTi remains an issue.

Here we investigate $d\sigma/dT(R\rightarrow B19')$ of a near-equiatomic nanocrystalline (NC) NiTi alloy. NC NiTi possesses R phase over a wide temperature range due to the constraining effect of grain boundaries [30–32]. It is thereby convenient to perform tensile experiments at various temperatures with reasonably large temperature increment so as to minimize errors of $d\sigma/dT(R\rightarrow B19')$ measurement. *In situ* synchrotron high energy XRD (HE-XRD) is used to examine the phase evolution during cooling while differential scanning calorimeter (DSC) is used for comparison. Tensile tests at various temperatures are employed to obtain stress–strain curves and the phase change during tension is probed by *in situ* HE-XRD. $d\sigma/dT(R\rightarrow B19')$ is measured and compared with counterparts from the literature. Possible reasons for the discrepancies are discussed and open questions are finally raised.

2. Materials and methods

A commercial Ni_{50.2}Ti_{49.8} alloy wire of 0.62 mm in diameter was annealed at 1023 K for 0.6 ks before subjected to cold drawing to a diameter of 0.30 mm at room temperature without intermediate annealing. The cross-sectional area reduction is 76%. The as-drawn wire, which was consisted of an amorphous phase [33–36], was later annealed at 623 K to fully crystallize to obtain the specimens used in this study. An FEI Tecnai F20 transmission electron microscope (TEM) was used to characterize the microstructure. TEM specimens were mechanically ground and polished before ion-milled by a Gatan 691 system. Tensile tests were performed at a strain rate of 0.001 s^{−1} using a WDT II-20 Instron-type tensile machine equipped with a clip-on strain extensometer.

The gauge length of tensile specimens was 50 mm. Tensile testing temperature ranges from 373 to 77 K. Iso-baric heating tests were carried out using the same tensile machine in load-control mode. The specimens were firstly held at 300, 350, 400 and 500 MPa of applied stresses at 340 K and subsequent cooled from 340 to 110 K.

Thermally induced transformation behavior was examined by *in situ* HE-XRD and a Q20 differential scanning calorimeter (DSC) at a cooling/heating rate of 5 K/min. SIMT behavior was investigated by *in situ* HE-XRD during tensile tests. HE-XRD experiments were conducted at Advanced Photon Source in Argonne National Laboratory, USA. X-ray beam had an energy of 105.7 KeV, a wavelength of 0.01173 nm and a size of 0.5 × 0.5 mm. HE-XRD spectrums were obtained by integrating over 360° azimuthal angle of the 2D patterns using the FIT2D package [35,37].

3. Results and discussion

Figure 1 presents the longitudinal microstructure and thermally-induced transformation behavior. The bright-field TEM image in Figure 1(a) shows equiaxed nano-sized grains. The average size is ~15 nm as shown in Figure 1(b). An SAD pattern and a dark-field TEM image are shown in Figure S1. The specimen is consisted of B2 and R phase at room temperature, as confirmed by the corresponding SAD pattern and two-dimensional HE-XRD pattern (Figure S1). DSC results in Figure 1(c) reveal the occurrence of B2→R and R→B2 transformation. The peak temperatures were 316 K (R_p) and 323 K (A_p), where p for peak and A for Austenite. The thermal hysteresis is about 7 K, which is typical for R transformation [35,38,39]. By using the intersecting tangent lines [35,38,39], the starting (R_s) and finishing (R_f) temperatures of B2→R transformation were determined to be 326 and 302 K, and those of R→B2 (A_s , A_f) were 309 and 333 K.

Figure 2(a) displays *in situ* HE-XRD spectrums from 420 K to 90 K. Inset figures are magnifications of the dashed boxes. B2 phase was the sole starting phase at 420 K as indicated by the presence of B2 peaks (inset a1-a2) and the absence of R peaks (inset a3-a4). With decreasing temperature, B2→R occurred as indicated by the emergence of R (300), R (112), R (210), R (10 $\bar{2}$) and R (10 $\bar{1}$) peaks. R_s was determined to be 320 K at which R (210), R (10 $\bar{2}$) and in particular R (10 $\bar{1}$) peaks emerged (green thick lines in inset a3-a4). For lab source XRD, these R peaks are usually too weak to resolve adequately. In practice, R_s was often considered as the temperature at which B2 (110) peak split into the R (300) and R (112) peaks [25–27]. Nevertheless, it has been suggested

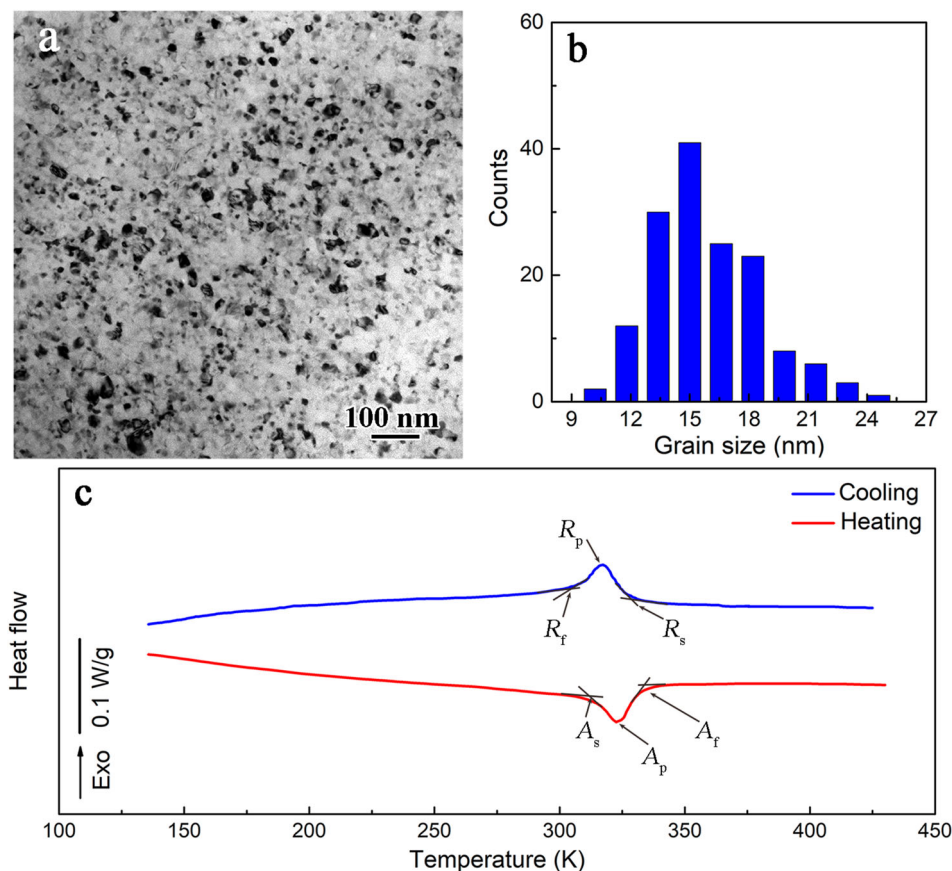


Figure 1. Microstructure and thermally-induced transformation behavior. (a) Bright-field TEM image showing nanosized equiaxed grains. (b) Grain size statistics showing the average grain size of ~ 15 nm. (c) DSC curves during cooling and heating showing B2 $\rightarrow$ R and R $\rightarrow$ B2 transformation. See Figure S1 in supplementary for more microstructural analyses.

that B2 $\rightarrow$ R transformation already occurs when B2 (110) peak broadens such that the splitting of B2 (110) does not necessarily indicate the right R_s [28,40,41]. This is confirmed by the present study. As shown by the inset a3, the B2 (110) peak split at roughly 190 K, which is way lower than R_s (green thick line).

We next show how R_f was measured. By calculating the peak area of R (10 $\bar{1}$) (inset a4), the volume fraction of R as a function of temperature was plotted in Figure 2(b) (red dots) and the balance was the volume fraction of B2 (blue dots). Above 320 K (R_s), the volume fraction of R remained zero and the specimen was consisted of sole B2. Below 320 K, the volume fraction of R started to increase continuously down to 190 K (black thick lines in insets). Afterwards, the volume fraction of R became nearly unchanged. Therefore, 190 K was determined to be R_f . So, Figure 2(b) can be divided into three regions. Region I (above 320 K) indicates single B2, Region II (190–320 K) indicates mixture of B2 and R, and Region III (below 190 K) indicates single R. Of special note is that B2 $\rightarrow$ R transformation occurred within a 130 K-wide (190–320 K) temperature window (Figure 2(b)), which is nearly five times of that (24 K-wide, 302–326 K)

measured by DSC in Figure 1(c). This again indicates the uncertainties of using DSC to exploit the R transformation and to identify the phase constitution [12,16].

Figure 3(a) shows the stress–strain curves from 373 to 77 K. The three regions labeled are the same with those in Figure 2(b). The stress plateaus in Region I correspond to B2 $\rightarrow$ B19' SIMT [34]. In Region II and III, two stress plateaus were observed, i.e. a lower plateau (point A to B) and a higher plateau (point C to E). A magnified view is shown in Figure 3(b). To exploit the deformation mechanisms of the two plateaus, *in situ* tensile tests were performed under HE-XRD at 153 K. We found that elastic deformation of R phase occurred from point O to A as evidenced by continuous rightwards shift of R (300) and R (112) peaks (Figure 3(c)). From point A to B, the two peaks gradually merged into one (blue thick line). The peak intensity evolution is shown in Figure S2. This indicates the reorientation (detwinning) of the self-accommodated (twinned) R phase, being consistent with the literature [12,25,27,35,42]. Lüders-like R $\rightarrow$ B19' SIMT occurred between C and E, as indicated by the abrupt presence of B19' peaks (red thick line) [34,37,43]. Combining these analyses, the deformation mechanisms

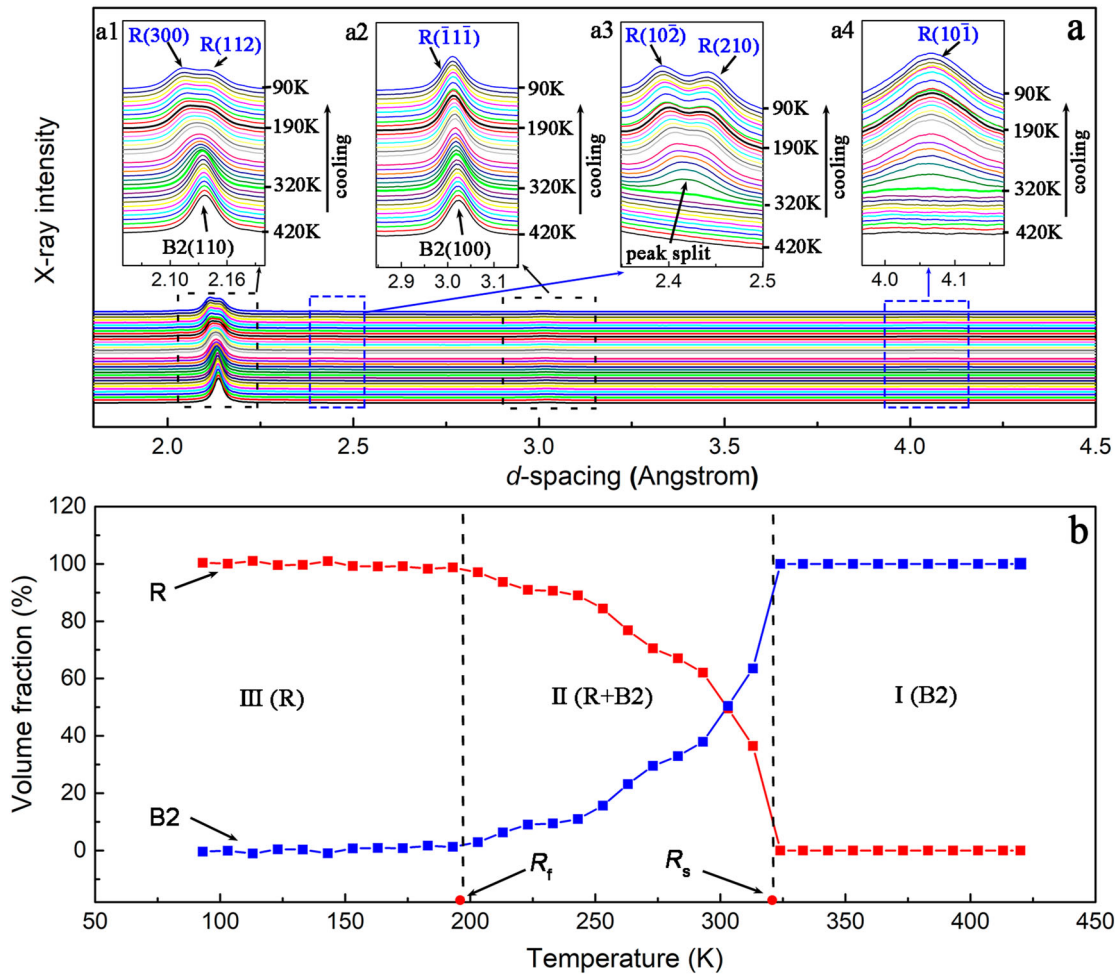


Figure 2. Microstructural evolution during cooling by *in situ* HE-XRD. (a) XRD spectra from 420 to 90 K. Insets show magnified dashed boxes. The green and black thick lines refer to R_s and R_t , respectively. (b) Variation in volume fraction of B2 and R during cooling. Roman numbers (I–III) indicate the temperature regimes of different phase constitution.

in Region II can be inferred, that is, detwinning of R occurred at lower plateau and $B2 + R \rightarrow B19'$ occurred at higher one. The evidence for the coexistence of B2 and R and their concurrent transformation to $B19'$ is shown in Figure S3.

Figure 3(d) shows the temperature dependence of critical SIMT stresses. The critical stresses were obtained by averaging the stress plateaus (errors of ± 10 MPa shown in the inset). $d\sigma/dT(B2 \rightarrow B19')$ was 4.7 MPa/K (Region I), which has been previously reported [34]. These measured stress and temperature values were cross-validated by the isobaric heating tests, as shown in Figures S4–5. Combining these results, two interesting trends were discovered. The first is that $d\sigma/dT(B2 + R \rightarrow B19')$ was non-linear, ranging from 4.1–1.8 MPa/K (Region II). Note that the limit values were obtained by performing the first derivation on the non-linear fit (dash line). The second is that $d\sigma/dT(R \rightarrow B19')$ was surprisingly -0.2 MPa/K (Region III). Figure 4 compares our results with those from the literature (Table S1) [4–17]. Note

that all data were based on claimed $R \rightarrow B19'$ transformation. We found that $d\sigma/dT(R \rightarrow B19')$ from literature are 3–5 MPa/K, showing very impressive consistency. It is however surprising to find that $d\sigma/dT(B2 + R \rightarrow B19')$ of the present study resides within this range. Furthermore, the $d\sigma/dT(R \rightarrow B19')$ value of the present study (-0.2 MPa/K) differs significantly from those of the previously claimed $R \rightarrow B19'$ transformation (Table S1) and other transformation paths (Tables S2 and S3). Especially, the negative sign has not been reported before.

In what follows, we attempt to explore the possible mechanisms accounted for the abnormal $d\sigma/dT(R \rightarrow B19')$ of NC NiTi. We first revisit the Clausius-Claperyon relation for $R \rightarrow B19'$ SIMT, i.e. $d\sigma/dT(R \rightarrow B19') = -\Delta S_{R \rightarrow B19'} / \epsilon_{R \rightarrow B19'} V_m$. It has been suggested that the reduction of $d\sigma/dT(R \rightarrow B19')$ with decreasing temperature is related to the decrease of $\Delta S_{R \rightarrow B19'}$ due to lattice distortion of R phase [16]. We thereby next examine the R lattice distortion in our specimens. As shown

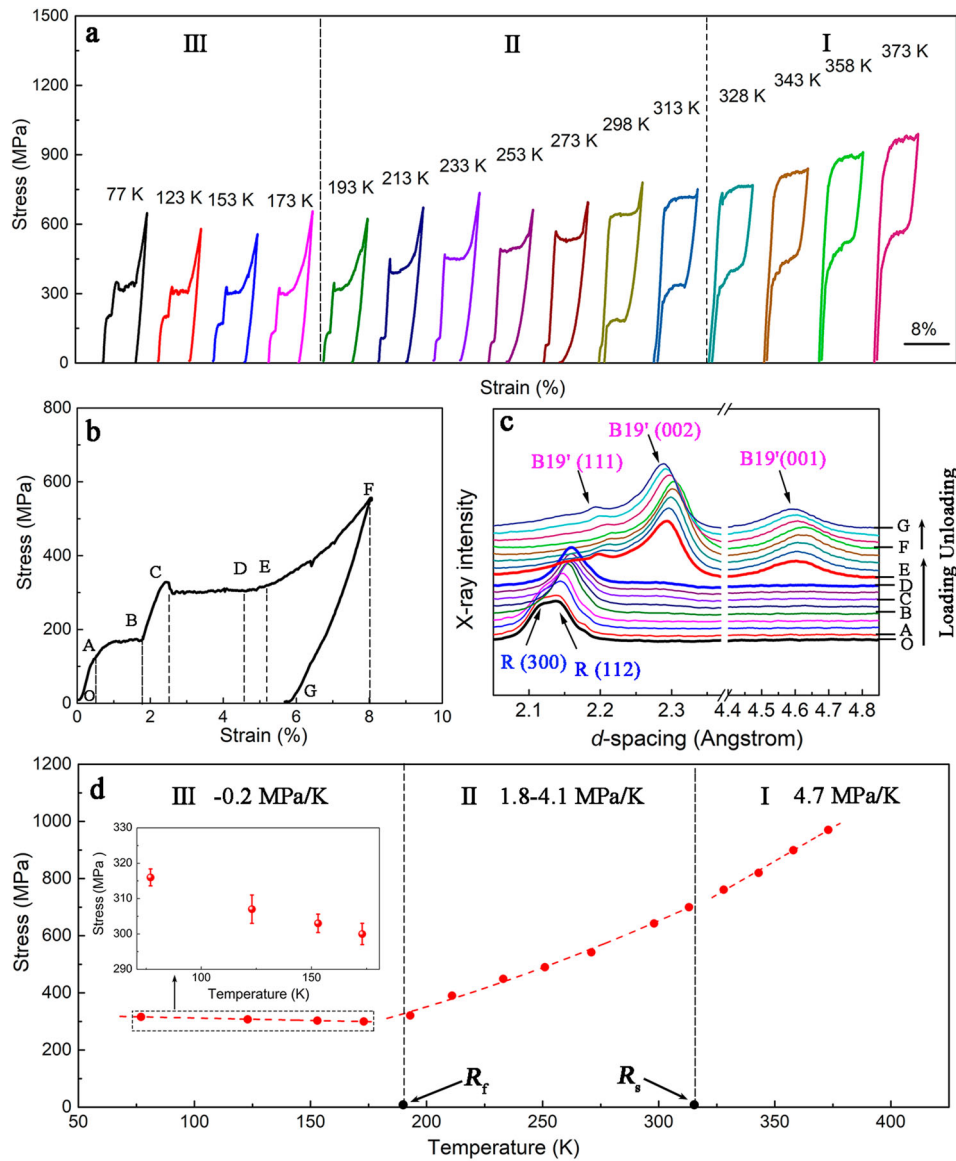


Figure 3. SIMT behavior. (a) Stress–strain curves from 373 to 77 K. (b) Stress–strain curve at 153 K. (c) *In situ* HE-XRD spectrums during tensile test at 153 K. Blue thick line indicates the end of R detwinning and red thick line indicates the start of B2→B19'. (d) Critical SIMT stresses as a function of temperature. Inset is magnification of dashed box.

in Figure 5(a) and Figure S6, the rhombohedral distortion angle of R lattice decreases with decreasing temperature. This leads to an increase of R detwinning strain with decreasing temperature (Figure S7), being consistent with previous reports [27,35,44]. By comparing with the literature [25,44–47], we find that the rhombohedral angle of R lattice of our specimen is significantly smaller. This implies that the magnitude of R lattice distortion of our specimen is more intensive towards B19' lattice, leading to possibly reduced $\Delta S_{R \rightarrow B19'}$ [25,44–47]. Such a mechanism, although is likely accountable, has two shortcomings. First, it does not necessarily result in lowered $d\sigma/dT(R \rightarrow B19')$. While $\Delta S_{R \rightarrow B19'}$ is reduced, $\epsilon_{R \rightarrow B19'}$ in the Clausius-Claperyon relation could also

be reduced due to lattice distortion. In this regard, the changing trend of $d\sigma/dT(R \rightarrow B19')$ cannot be justified without knowing whether $\Delta S_{R \rightarrow B19'}$ or $\epsilon_{R \rightarrow B19'}$ changes more dramatically. Atomistic simulations may be needed to clarify this. Second, even if the reduction of $\Delta S_{R \rightarrow B19'}$ outweighs that of $\epsilon_{R \rightarrow B19'}$, it is unknown if the distortion will eventually lead to negative $\Delta S_{R \rightarrow B19'}$. It thereby cannot explain the negative sign of $d\sigma/dT(R \rightarrow B19')$.

It could be argued that ideally the stress term in the Clausius-Claperyon relation should be the equilibrium stress (σ_0) instead of the upper or lower plateau stress. Indeed, σ_0 can be calculated by averaging the upper and lower plateau stress, which is a prevailing convention. However, such a strategy could be tricky. In many cases,

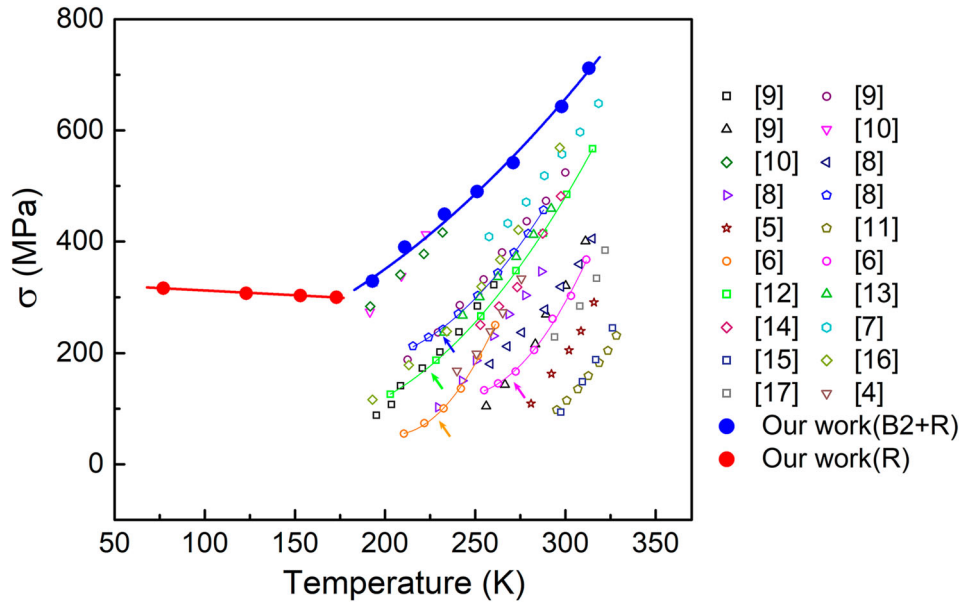


Figure 4. Comparison of $d\sigma/dT$. Solid dots refer to our results and hollow dots refer to claimed $R \rightarrow B19'$ SIMT in the literature [4–17]. Arrows indicate $d\sigma/dT$ with apparent non-linearity.

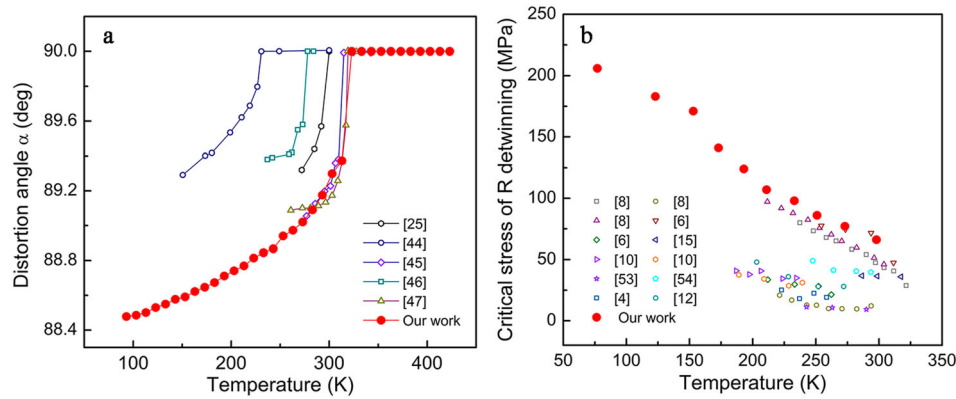


Figure 5. (a) Comparison of the distortion angle of the R phase during cooling between our NC NiTi and other NiTi-based SMAs [25,44–47]. (b) Critical stress of R detwinning as a function of temperature. Red dots were obtained from the lower plateaus in the stress–strain curves in Figure 4(a). All other data points were from the literature [4,6,8,10,12,15,53,54].

there is no spontaneous backward transformation such that a lower plateau does not exist [5,8–12,14,15]. This is the case in the present study as shown in Region III. Even for the cases with both of the stress plateaus being present, upper plateau stresses are widely used for calculating $d\sigma/dT$ and we did not find an exclusion for $d\sigma/dT(R \rightarrow B19')$ in literature to date [4,7]. Furthermore, recent studies have shown that the equilibrium stress may not reside right in between of the upper or lower plateau [48]. So, the Clausius-Claperyon relation may not be suitable for analysis in the present study.

It has been recently suggested that the SIMT of doped NiTi alloys is a thermally activated process [49–51]. For instance, negative $d\sigma/dT(B2 \rightarrow B19')$ and rate-dependent critical stresses have been convincingly shown in superelastic Ni-rich NiTi and are attributed to

kinetic arresting of SIMT [49–51]. While the equilibrium $d\sigma/dT$ was claimed to remain following the Clausius-Claperyon relation, the upper and lower stresses are strongly affected by the reduced thermal activation at low temperatures [49–51]. Such an effect comes into play possibly via affecting the lattice friction of phase interface migration [52]. We are not able to directly characterize the lattice friction of $R \rightarrow B19'$ SIMT, but we noticed in Figure 5(b) that the detwinning stress of our specimens increases more drastically with decreasing temperature in comparison with that of other NiTi-based SMAs [4,6,8,10,12,15,53,54]. Given that the previous reports have successfully used a dislocation-based model to describe the thermally activated SIMT process [49–51,55], the similar dependence on temperature between detwinning (a dislocation facilitated process)

and SIMT can be possibly established. Therefore, the temperature-dependence of R detwinning stress implies that the thermal activation for $R \rightarrow B19'$ SIMT is likely reduced in the same manner. As a possible result, the lattice friction of phase interface migration is particularly strong at cryogenic temperatures, leading to the negative $d\sigma/dT(R \rightarrow B19')$.

From another perspective, it has been suggested that the critical conditions of SIMT are associated with not only the thermodynamic criterion of phase stability but also the detwinning process [13,56]. Even if $R \rightarrow B19'$ SIMT is thermodynamically ready, the actual initiation of transformation calls for the change of internal microstructure to a favorable state with respect to the stress field (i.e. detwinning). As such, SIMT will occur only after this second criterion is met. In this regard, the negative $d\sigma/dT$ in Region III is probably related to the detwinning of $B19'$ martensite, the stress of which has been well-known to increase with decreasing temperature [9,57]. We thereby suggest that the continuous increase in the detwinning stress with decreasing temperature may contribute to the negative $d\sigma/dT(R \rightarrow B19')$ in Region III.

One last issue to be noted is the surprising consistency between $d\sigma/dT(B2 + R \rightarrow B19')$ of our specimen and $d\sigma/dT(R \rightarrow B19')$ of other NiTi alloys (Figure 4). We suggest that the consistency is not a coincidence. There is a possibility that the 3–5 MPa/K of $d\sigma/dT(R \rightarrow B19')$ in literature is a combined value of $d\sigma/dT(B2 \rightarrow B19')$ and $d\sigma/dT(R \rightarrow B19')$ (Figure 3(d)). Provided that the phase identification (R_f) in literature was mostly carried out by DSC which is not quite reliable, the tested specimens were likely consisted of both B2 and R phase. Another possible argument is that many of the reported σ - T relationships are non-linear [6,8,12], as indicated by the arrows in Figure 4. These trends are similar to our results (blue line in Figure 4), all owing to the increasing volume of R during cooling. But it must be noted that the grain size of our specimens is ~ 15 nm, being way smaller than the micron-level grain sizes reported [4–17]. So an ideal comparison should be made by using coarse-grained NiTi-based alloys with single R phase. In particular, the alloys better remain sole R phase over a wide temperature range to allow reasonable temperature increment for tensile tests. This requires a strong suppression of thermally-induced $R \rightarrow B19'$ and/or $B2 \rightarrow B19'$. Otherwise, $B19'$ forms before thermally-induced $B2 \rightarrow R$ completely finishes, which is probably why most previous studies cannot obtain useful data below 200 K (Figure 4). Furthermore, the suppression should not be too strong, otherwise $B2 \rightarrow R$ may be completely suppressed and R-like (average structure remains B2) instead

of R forms during cooling [24,50,58–62]. The achievement of such coarse-grained alloys may require new alloy design strategy because the current methods such as alloying [10,63,64], aging [65–67] and pre-deformation [68–70] can hardly meet the requirements.

4. Conclusions

We have exploited the thermally- and stress-induced transformation behavior of a near-equiatomic NC NiTi alloy, particularly focusing on $d\sigma/dT(R \rightarrow B19')$. Thermally-induced $B2 \rightarrow R$ transformation occurred within a 130 K-wide temperature window (190–320 K), as revealed by HE-XRD. Such a window is five times wider than that (302–326 K) measured by DSC. $d\sigma/dT(R \rightarrow B19')$ of our NC NiTi was -0.2 MPa/K, being in sharp contrast to the 3–5 MPa/K reported before. The discrepancy is likely attributed to the reduced thermal activation or enhanced detwinning stresses at cryogenic temperatures.

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Disclosure statement

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

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