

Research Article

Effect of Annealing on Strain-Temperature Response under Constant Tensile Stress in Cold-Worked NiTi Thin Wire

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The present paper aims to understand the influence of annealing on the strain-temperature response of a cold-worked NiTi wire under constant tensile stress. It was found that transformation behavior, stress-strain relationship, and strain-temperature response of the cold-worked NiTi wire are strongly affected by the annealing temperature. Large martensitic strains can be reached even though the applied stress is below the plateau stress of the martensite phase. At all stress levels transformation strain increases with increasing annealing temperature in the range of 350°–450°C and decreases with increasing annealing temperature in the range of 450°–650°C. The martensitic strain at lower stress levels exhibits the same tendency. At higher stress levels the martensitic strain increases with increasing annealing temperature.

1. Introduction

Among many shape memory alloys (SMAs), NiTi has been widely used in many technological and engineering applications due to its excellent shape memory effect, superelasticity, high damping capacity, and others [1, 2]. Its remarkable properties result from a reversible martensitic phase transformation between austenite (B2) and martensite (B19') phases, which can be either stress induced or temperature driven. The transformation is sensitive to factors such as material composition, deformation processing, and heat treatments. Therefore, a mix of cold work followed by a specific annealing process has been comprehensively considered to optimize the physical and mechanical properties of a NiTi product and achieve shape memory and/or superelasticity. Significant efforts have been made to address the effects of heat treatment on transformation behavior [3–5], microstructure [6–8], recovery stress [9, 10], damping [11, 12], plateau stress and strain [13, 14], as well as lifetime of NiTi SMAs [15, 16].

To open new applications of NiTi SMAs, including smart structures, intelligent controllers, and memory devices, the understanding of strain and phase transformation behavior under constant applied stress is essential.

In the present paper the influence of annealing on martensitic transformation and residual strains of cold-worked NiTi wire developed during cooling/heating under constant tensile stress has been investigated.

2. Experimental Procedure

The experiments were performed on a commercial NiTi wire provided by SAES Getters (Italy) with a diameter of 0.076 mm and a normal composition of 50.2 at % Ni. As-received wires (35% cold-worked) with a length of 100 mm were annealed in an argon atmosphere for 10 min at 350°, 450°, 550°, and 650°C, respectively.

The transformation temperatures of the annealed specimens were determined by differential scanning calorimetry (DSC TA-Q2000). The heating and cooling rates were set at 10°C/min. The phase transformation temperatures were characterized by a tangent line intersection method, whereas the transformation enthalpy is determined by the area under the curve.

All thermomechanical experiments were performed on a dynamic mechanical analyzer (DMA) instrument

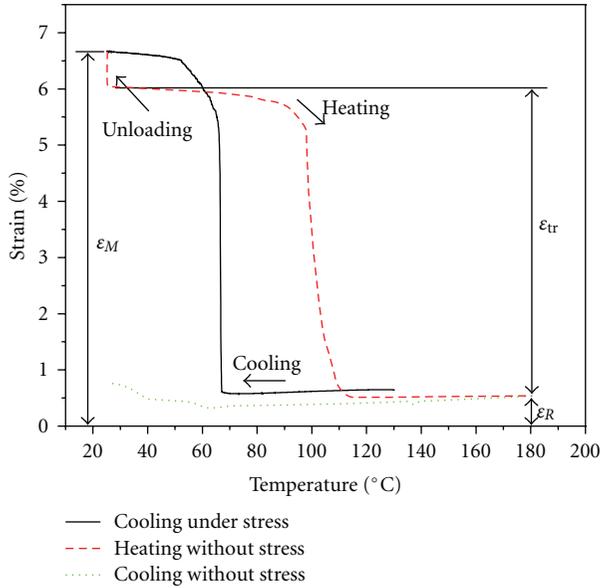


FIGURE 1: Schematic illustration of the experimental settings.

(TA Q800). Uniaxial tensile tests to failure were conducted at a strain rate of 0.5%/min at a constant temperature $T = 25^\circ\text{C}$.

The strain-temperature curves of annealed specimens were investigated as a function of applied stress. The experimental settings are as follows:

- (1) loading the specimen at 130°C in the austenitic phase with a constant tensile stress,
- (2) cooling the specimen from 130°C to 25°C under this constant tensile stress,
- (3) unloading the sample to zero stress,
- (4) heating the sample to 180°C followed by
- (5) cooling down to 25°C .

The heating/cooling rate is $3^\circ\text{C}/\text{min}$. A schematic illustration of the experimental settings is shown in Figure 1. Here, ϵ_M , ϵ_{tr} , and ϵ_R indicate martensitic strain, transformation strain, and residual strain, respectively.

3. Results

3.1. Thermal Transformation Behavior. The transformation temperatures and heats of transformation of the specimens for different annealing conditions were measured, and the results are shown in Table 1

It is seen that specimens annealed at 350° and 450°C exhibit a two-stage $B2 \rightarrow R \rightarrow B19'$ transformation on cooling and a single-stage $B19' \rightarrow B2$ transformation on heating. With increasing annealing temperature, the transformation temperatures increase except for R_s . Specimens annealed at 550° and 650°C exhibit identical transformation behavior of single-stage $B19' \leftrightarrow B2$ transformation. With increasing annealing temperature, the transformation temperatures increase slightly. It is also seen that the values of

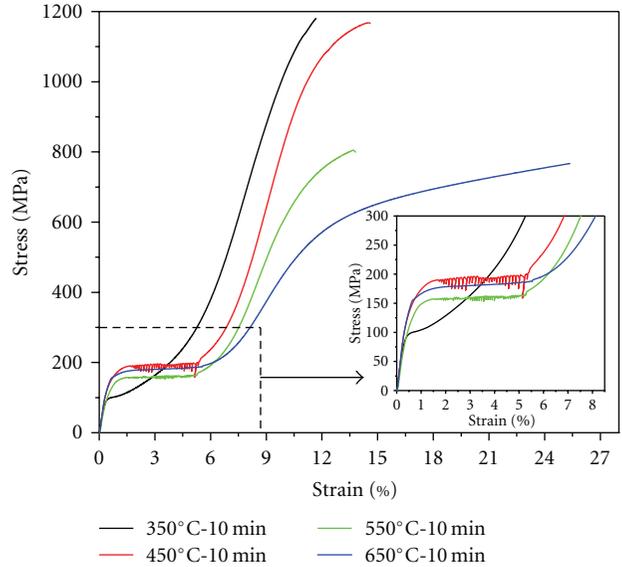


FIGURE 2: Stress-strain curves of annealed specimens measured at 25°C .

heat of transformation increase with increasing annealing temperature except for ΔH_M for the 650°C annealed specimen.

3.2. Tensile Tests at 25°C . Figure 2 shows the stress-strain curves of the annealed specimens measured at 25°C in strain control. The inset in Figure 2 shows details of the stress-strain curves up to 300 MPa. It is seen that the values of strength for the specimens annealed at lower temperatures (350° and 450°C) are much higher than those annealed at higher temperatures (550° and 650°C). The specimen annealed at 350°C shows no obvious stress plateau and a sharp increase in stress with increasing strain above 3%. Others show clear Lüders-type deformation over a stress plateau with plateau strain more than 5%. It is interesting to note that the 450°C annealed specimen displays a higher and more unstable plateau stress than others. A significant amount of stress fluctuation about 40 MPa is seen. The 550°C annealed specimen has a relative low plateau stress with small fluctuation about 10 MPa. The 650°C annealed specimen exhibits a medium and near constant plateau stress with no fluctuation.

3.3. Transformation Strain upon Cooling at Various Constant Stresses. Figures 3(a)–3(d) shows the strain-temperature curves of the annealed specimens under constant loads during cooling. All specimens were first heated to 130°C to austenite and then loaded prior to the actual cooling process. Specimens annealed at 350° and 450°C exhibit two-step transformation upon cooling at loads of 55 and 110 MPa. The first step is related to $B2 \rightarrow R$ transformation. The second step, occurring at lower temperatures, is related to $R \rightarrow B19'$ and $B2 \rightarrow B19'$ transformation. However, R-phase transformation appears to be almost completely inhibited upon cooling at loads of 200 and 300 MPa.

TABLE 1: The transformation temperature ($^{\circ}\text{C}$) and heats of transformation (J/g) obtained from the DCS curves.

Samples	R_s	R_f	ΔH_R	M_s	M_f	ΔH_M	A_s	A_f	ΔH_A
350 $^{\circ}\text{C}$ -10 minutes	67.12	50.24	5.788	32.24	8.54	12.06	59.60	79.59	25.26
450 $^{\circ}\text{C}$ -10 minutes	62.19	58.69	7.593	46.18	37.14	15.79	78.61	88.85	25.93
550 $^{\circ}\text{C}$ -10 minutes	—	—	—	67.60	60.95	31.68	92.36	105.34	26.43
650 $^{\circ}\text{C}$ -10 minutes	—	—	—	71.19	62.99	29.94	95.33	113.28	28.70

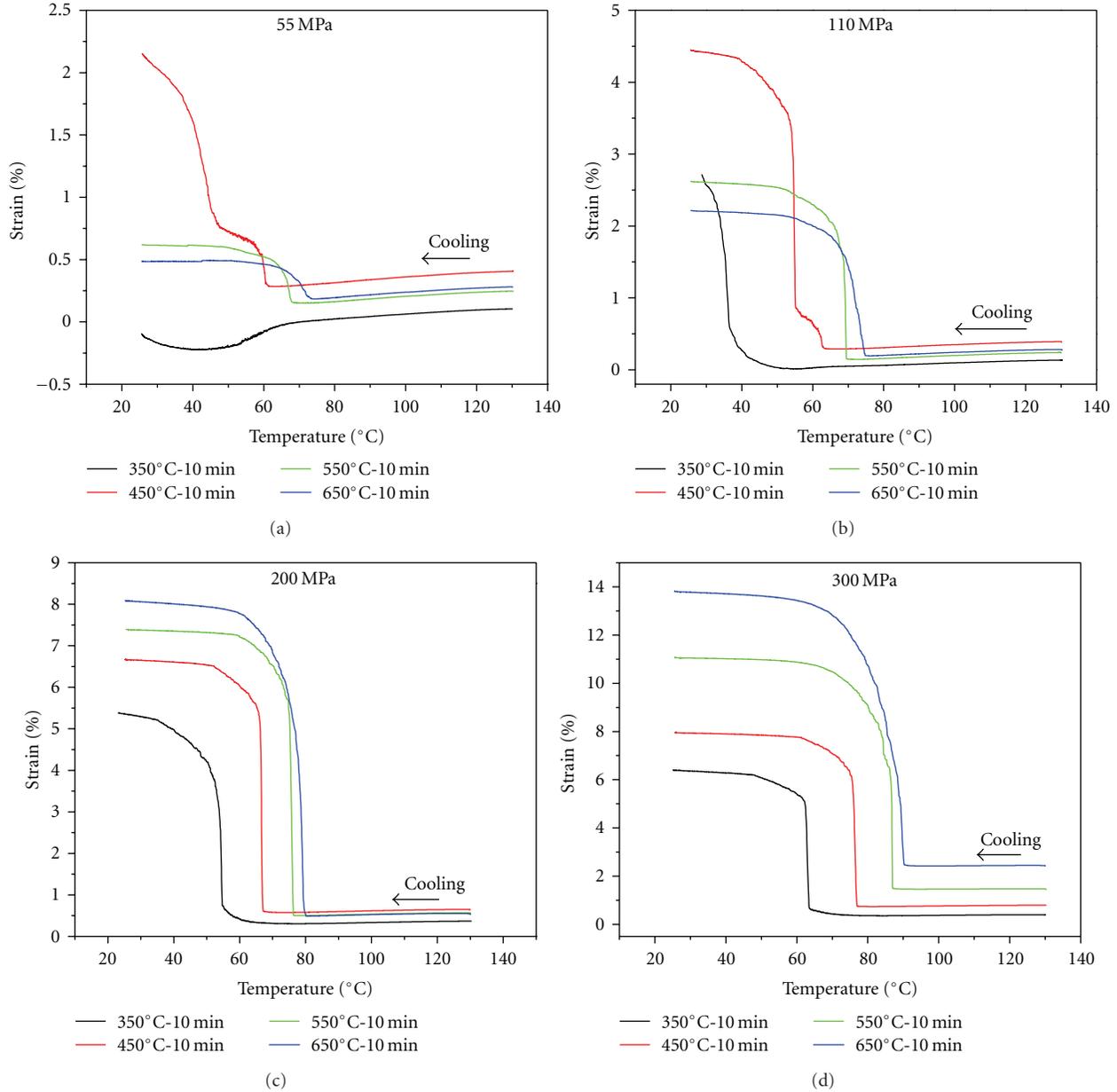


FIGURE 3: Strain-temperature curves upon cooling under various constant applied stresses.

It is seen that the strain slowly increases at lower stress levels but drastically increases at higher stress levels. The martensitic strain of the specimen annealed at 350 $^{\circ}\text{C}$ is zero at 25 $^{\circ}\text{C}$ when cooling under a constant stress of 55 MPa.

Figure 4 shows the variation of transformation temperature M_s as a function of applied stress for the annealed specimens. The transformation temperatures were determined by a tangent line intersection method from curves in Figure 3. It is seen that the M_s temperatures increase with

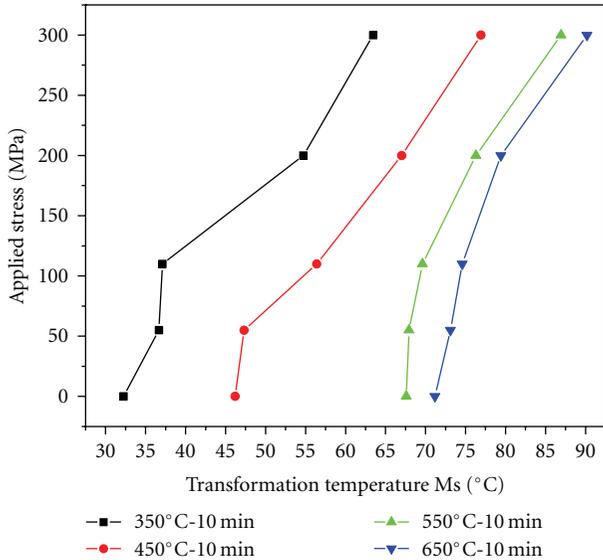


FIGURE 4: Effect of applied stress on the transformation temperature M_s .

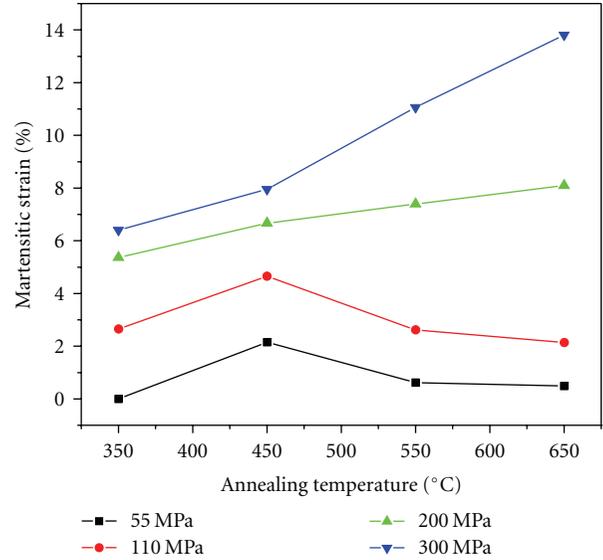


FIGURE 6: Effect of annealing temperature on the martensitic strain ϵ_M .

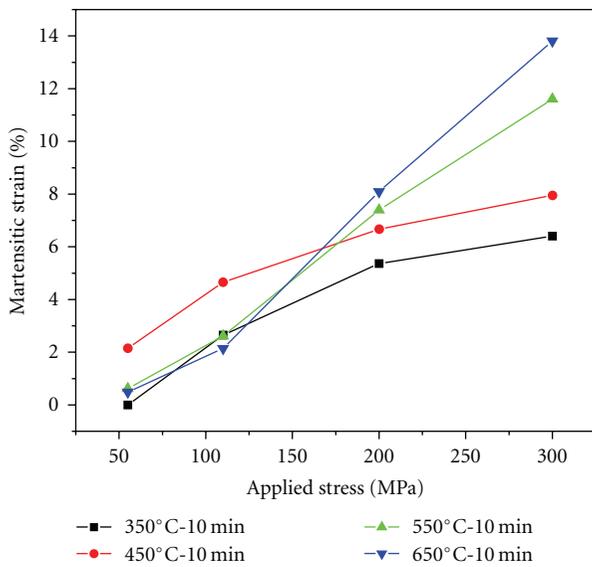


FIGURE 5: Effect of applied stress on the martensitic strain ϵ_M .

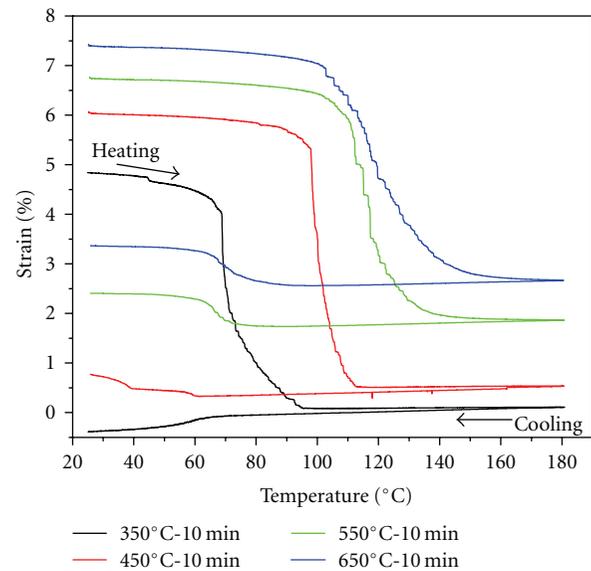


FIGURE 7: Representative strain-temperature curves measured during free shape recovery after cooling under a constant stress of 200 MPa.

increasing applied stress for all specimens. This is closely in-line with the Clausius-Clapeyron relationship that as the applied external stress increases so do the transformation temperatures [17].

Figure 5 shows the effect of applied stress on the martensitic strain ϵ_M . For all samples, the martensitic strain increases with increasing applied stress. The increase is noticeably more pronounced in samples annealed at higher temperature.

Figure 6 shows the effect of annealing temperature on the martensitic strain ϵ_M , a different manner of presenting the results in Figure 5. It is worth noting that the martensitic strains at loads of 55 and 110 MPa increase with increasing annealing temperature up to 450°C and decrease when

annealed above 450°C. The martensitic strains at loads of 200 and 300 MPa monotonically increase with increasing annealing temperature, and the increase is noticeably more pronounced for higher loads.

3.4. Free Shape Recovery. Figure 7 shows representative strain-temperature curves measured during free shape recovery after cooling at constant stress of 200 MPa. For 550° and 650°C annealed specimens the austenite was not fully recovered at the highest temperature, possibly due to the development of microplasticity generated during the first cooling from the austenite under load.

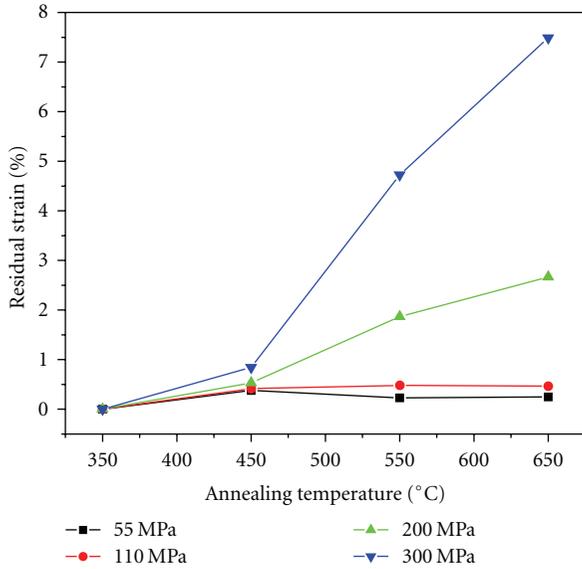


FIGURE 8: Effect of annealing temperature on the residual strain ϵ_R .

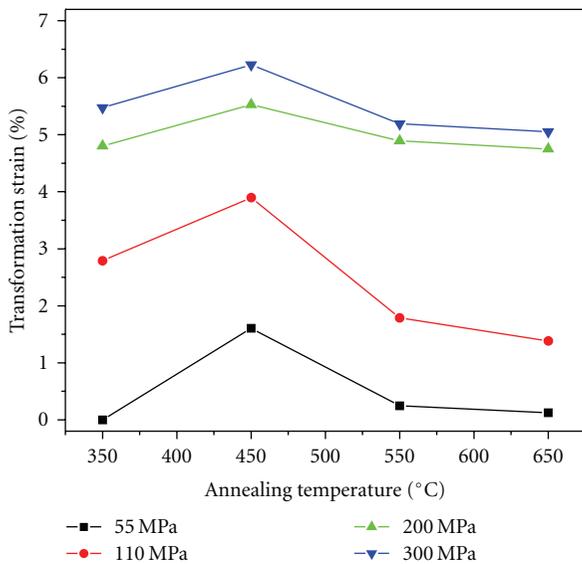


FIGURE 9: Effect of annealing temperature on the transformation strain ϵ_{tr} .

Figure 8 shows the effect of annealing temperature on the residual strain ϵ_R . The tendency is similar to the effect of annealing temperature on the martensitic strain ϵ_M , as shown in Figure 6. It is interesting to note that, even at high applied stress, the martensitic strain of the 350°C annealed specimen can completely recover upon heating above the austenite finish temperature. Thus, the residual strain is non-existent in the strain-temperature curves.

Figure 9 shows the effect of annealing temperature on the transformation strain ϵ_{tr} . It is seen that, for all samples, the transformation strain increases with increasing applied stress. It is worth noting that, regardless of the applied stress level, the transformation strain increases with increasing

annealing temperature up to 450°C and decreases when annealed above 450°C. The decrease is noticeably more pronounced at lower applied stresses.

4. Discussion

In this study the materials were not deformed in the martensite condition. We start cooling the specimens from austenite under a constant applied stress. The results show that the martensitic strain ϵ_M , transformation strain ϵ_{tr} , and residual strain ϵ_R developed during cooling and heating are strongly affected by annealing and/or applied stress. This phenomenon could be related to the number and distribution of dislocations and textures of the materials.

The previous studies have shown that the texture orientation significantly influences the stress-strain curve and shape memory strain of NiTi SMAs [18–20]. Polycrystalline NiTi with a high density of grains oriented with a $\langle 111 \rangle$ axis parallel to the loading direction will generally demonstrate large recoverable transformation strains in tension. It is known that a cold-worked NiTi alloy usually has a strong $\langle 111 \rangle$ texture. When the annealing temperature is below the recrystallization temperature, annealing causes recovery to occur without changing the crystalline orientation distribution appreciably. So that the deformation texture remains and affects the shape memory characteristics of the cold-drawn wire [19]. The degree of texturing was much less in the higher temperature annealing condition. From this point of view, the recoverable transformation strain could decrease with increasing annealing temperature.

It is also well known that cold working introduces high density of dislocations in NiTi alloys, which increases the strength but decreases the ductility. Also the dislocations act to inhibit the martensitic transformation [21]. Annealing induces annihilation of the dislocations and recrystallization of the cold-worked specimen. The higher the annealing temperature, the more the dislocations are rearranged and annihilated. Therefore, with increasing annealing temperature, the dislocation density decreases, which could cause an increase in the recoverable transformation strain.

In this study, specimens annealed at 350° and 450°C exhibited two-stage $B2 \rightarrow R \rightarrow B19'$ transformations on cooling, implying that the samples were partially annealed without recrystallization. For the specimen annealed at 350°C, the dislocation density and the strength were high, which inhibits the free propagation of martensitic variants across neighboring grains, so the martensitic strain ϵ_M , transformation strain ϵ_{tr} , and residual strain ϵ_R are small. Annealing at 450°C causes a decrease in dislocation density, which leads to less resistance to martensitic transformation. On the other hand, the degree of the texturing is still high, so the largest transformation strain ϵ_{tr} was obtained at all stress levels, as shown in Figure 9. At the same time, the residual strain ϵ_R was relatively small, as shown in Figure 8. When annealing above the recrystallization temperature, the dislocation density and the strength decrease largely. In this case, the microplastic deformation was easily induced during the martensitic transformation under loads, which leads to the decrease in the transformation strain ϵ_{tr} and

the increase in the residual strain ϵ_R , as shown in Figures 8 and 9. Interestingly, a slightly smaller residual strain ϵ_R in 550° and 650°C annealed specimens was observed under the stress levels of 55 and 110 MPa. One possible reason for this is that only the grains with the most favorable orientation transform into stress-induced martensite under the stress levels of 55 and 110 MPa, and the number of these grains decreases with increasing annealing temperature. This phenomenon needs further investigation.

5. Conclusions

In this paper we examined the influence of annealing on strain-temperature behavior of cold-worked NiTi wires under constant tensile stresses. The results show that a large martensitic strain can be reached, even though the applied stress is below the plateau stress of the martensite phase. The martensitic strain is strongly affected by annealing temperature and applied stress. The strain increases with increasing applied stress for all annealing conditions. At higher applied stress the martensitic strain increases with increasing annealing temperature; at lower applied stress the value increases when annealing until 450°C and then decreases when annealing above 450°C. The same tendency is also seen in the variation of residual strain with annealing temperature. Regardless of the level of applied stress, transformation strain increases first and then decreases with increasing annealing temperature.

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