Shape Memory Effect Induced by Stress-induced α' Martensite in a Metastable Fe–Cr–Ni Austenitic Stainless Steel

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Abstract It is not clear whether a shape memory effect (SME) can be realized by stress-induced α' martensite in metastable austenitic stainless steels although the stress-induced ε martensite in these materials can result in the SME. To clarify this problem, the relationship between the shape recovery and the reverse transformation of the stress-induced ε and α' martensite in a 304 stainless steel was investigated. The results show that the stress-induced α' martensite can result in the SME when heating above 773 K. After deformation at 77 K and step heating or directly holding at 1073 K, two-stage shape recoveries below 440 K and above 773 K can be obtained due to the reverse transformation of the stress-induced ε martensite and α' martensite, respectively. After deformation at room temperature, the α' martensite produced can result in the SME only when directly holding at 1073 K. The intrusion of more dislocations before the formation of the α' martensite at room temperature than at 77 K is the reason that the α' martensite induced at room temperature cannot result in the SME in the case of slow heating. The recovered strains resulting from the stress-induced ε and α' martensite are proportional to the amounts of their reverse transformation, respectively.

KEY WORDS: Austenitic stainless steel; Shape memory effect; Alpha prime martensite; Epsilon martensite; Two-stage recoveries

1 Introduction

Type 304 austenitic stainless steels are extensively used in various industries, such as petroleum, chemistry, power generation, and nuclear industries, due to their good combination of mechanical properties, corrosion resistance, and weldability [1–4]. In addition, many work clearly showed that the austenitic phase (γ) in the 304 stainless steels is

metastable. Consequently, the metastable γ phase can be transformed to alpha prime martensite (α') and epsilon martensite (ε) when it is deformed below the M_d temperature, the highest temperature where stress-induced martensitic transformation can take place [5, 6]. Many studies showed that a good shape memory effect (SME) can be realized in high manganese Fe–Mn–Si–Cr–Ni-based austenitic stainless steels and in Fe–Ni–C and Fe–Ni–Co–Ti alloys by the stress-induced ε and α' martensite, respectively [6–16]. However, to our knowledge, so far only a very poor SME was reported in 304 stainless steels due to the stress-induced ε martensite, while the SME realized by the stress-induced α' martensite has not been reported [17–19].

Enami et al. [17] first obtained a 65% recovery rate in 304 stainless steel through bending about 1% strain at 77 K and then dipping in boiling water. No further shape recovery was detected when further heating the partly recovered specimen up to 474 K. After tensile deformation

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by 3.3% and 7.9% at 77 K, 26.7% and 13.8% recoveries were obtained in a 304 stainless steel when heated in boiling water, respectively [17]. To correlate the shape recovery with the martensitic transformation, later the same authors made a direct observation of the reverse transformation of martensite in the deformed 304 stainless steel by optical and electron microscopy, respectively [20]. The results showed that both the ε and α' martensite were produced after deformation at 77 K. The stress-induced ε plates gradually reverted to the γ phase when heating, and they completely reverted to the γ phase at 553 K. However, the α' martensite remained unchanged up to 553 K. Based on the above fact, they thought that the SME in the 304 stainless steel resulted from the stress-induced ε martensite and its reverse transformation, not from the stress-induced α' martensite. Furthermore, they speculated that even when heating to above the finish temperature A_f of the reverse α' martensite transformation, the shape recovery due to the α' martensite would occur to a very limited extent because the crystal lattice correspondence between the α' martensite and the γ phase would have been destroyed by the diffusion process of carbides formation below the A_f temperature [17]. However, the same authors did not heat the deformed specimens to above the A_f temperature to confirm their speculation. In addition, the result by Sato *et al.* [18] also showed that a 40% recovery was obtained in a Fe–18Cr–14Ni single crystal after it was tensile deformed at 220 K and then heated to room temperature (RT). Note that they also did not heat the deformed specimens to above the A_f temperature. Clearly, it is of interest to investigate whether or not the SME can be realized by the stress-induced α' martensite in the 304 stainless steels.

In this work, we investigated the relationship between the shape recovery and the reverse transformation of the stress-induced ε and α' martensite in a 304 stainless steel after deformation at 77 K or at RT and then step heating up to 1073 K or directly holding at 1073 K, respectively. The results clearly showed that after deformation at 77 K, two-stage shape recoveries below 440 K and above 773 K occurred due to the reverse transformation of the ε and α' martensite, respectively. However, the α' martensite induced by deformation at RT can result in the shape recovery only when directly holding at 1073 K. In addition, a positive relationship existed between the recovered strains and the amounts of the reverse transformation of stress-induced ε and α' martensite, respectively.

2 Experimental

The experimental material was a commercial 304 stainless steel rod of 20 mm in diameter. Its chemical compositions (in wt%) were: 18.12Cr, 8.16Ni, 1.51Mn, 0.53Si, 0.11V,

0.04 Cu, 0.02P, 0.04C, balance Fe. The rod was solution-treated at 1353 K for 30 min, followed by air cooling. The SME was measured by a bending test, and the specimens with a size of 70.0 mm $\times$ 1.5 mm $\times$ 2.0 mm were obtained by cutting the solution-treated rod using a Mo filament cutter. They were bent around moulds with different radii to 180° at 77 K or at a RT of 293 K. The pre-strain ε was taken as the maximum tensile strain at the outer edge of the specimens and was determined by the equation: $\varepsilon = t/(2R + t)$, where R is the bend radius and t the sample thickness [9]. The bent specimens were step-heated at different temperatures for 3 min up to 1073 K or directly held at 1073 K for 3 min. The residual strains ε_r were measured after heating at different temperatures according to the above equation. The recovered strains were given by the difference between the values of ε and ε_r .

The reverse transformation processes of the stress-induced ε and α' martensite during the process of recovery heating were characterized by means of dilation measurements and in situ measurements of X-ray diffraction (XRD). Because it was difficult to make these measurements using the bent samples due to too small area, the tensile-deformed samples were used to simulate the bent samples. First, the specimens cut from the solution-treated rod were tensile deformed at 77 K or RT, and then the specimens for the dilation and XRD measurements were cut from the tensile-deformed specimens. The dilation measurements were made using farmastar-F II phase transformation equipment, and the in situ measurements of XRD were made using Bruker D8 Discover XRD apparatus equipped with a heating accessory system, in which a protective cover of Al₂O₃ was used. The heating/cooling rate was 20 K/min in both measurements.

The amounts of α' martensite and their evolution were accurately characterized by the magnetization changes. Because only the α' martensite is ferromagnetic in the 304 stainless steel, the magnetization is directly proportional to the amount of ferromagnetic α' martensite. The magnetization curves at 300 K of the tensile-deformed specimens after heating at different temperatures for 3 min were measured by a Quantum Design's superconducting quantum interference device (SQUID). The optical observations were made using an OLYMPUS GX51 optical microscope after the polished specimens were etched in a solution of 1.2% K₂S₂O₅ + 0.5% NH₄HF₂ in water. The substructures of the specimens were studied using a Tecnai F20 transmission electron microscope (TEM). The TEM specimens were first mechanically ground and then polished in a solution of 40 mL methanol and 10 mL sulphuric acid at 293 K with a twin-jet polisher.

3 Results

3.1 Effect of Recovery Heating Temperature on Shape Recovery

Figure 1a shows the recovered strains of the specimens bent by 3.8% and 10.0% at 77 K as functions of the step-heating temperature, respectively. Two-stage recoveries below around 440 K and above around 773 K were very evident in both the bent specimens. When heating to RT from 77 K, about 0.1% strain was restored for both the specimens. After further heating to about 423 K, the recovered strain increased to about 0.31% in the 3.8% specimen, while it was 0.24% in the 10% specimen. Between 423 and 773 K, it remained almost constant in the 3.8% specimen, but it increased to 0.33% in the 10% specimen. Interestingly, after heating at 873 K, it rose rapidly to 0.68% in the 10% specimen, while it only increased to 0.45% in the 3.8% specimen. After heating at 973 K, it further increased to 0.74% in the 10% specimen; above 973 K, it remained almost constant. However, it changed little in the 3.8% specimen as the temperature was increased up to 1073 K from 873 K. The shape recoveries measured after bending and then directly heating at 1073 K, also given in Fig. 1a (solid symbols), were almost equal to those measured after step heating to 1073 K.

Figure 1b shows the dilatation curves for the specimens after tensile deformation at 77 K by 3.8% and 10.0%, respectively. Two-stage contractions may also be easily identified. In addition, the first contraction below 440 K in the 3.8% specimen was slightly larger than that in the 10% specimen, but its secondary contraction above 773 K was very much lower. Consequently, after heating and cooling again to RT, a much larger contraction occurred in the 10% specimen. These results are in agreement with the recovered strains measured in the bent specimens.

3.2 Stress-Induced ε and α' Martensite and Their Reverse Transformation

Figure 2 shows the optical micrographs at RT of the specimen after deep cooling at 77 K for 15 min and those of specimens after tensile deformation by 3.8% and 10% at 77 K, respectively. After deep cooling at 77 K, only a single phase existed, that is, no martensite was thermally produced. After deformation by 3.8%, many α' martensite plates (black) and ε martensite plates (white) were produced. After 10% deformation, more α' martensite plates, but less ε martensite plates were produced as compared to the 3.8% specimen.

The XRD patterns at RT also confirmed that the amount of α' martensite in the 10% specimen was much higher than that in the 3.8% specimen, while the amount of ε martensite was lower (Fig. 3). Two weak diffraction peaks of the ε martensite, (10.0) and (10.1), could be clearly identified in the 3.8% specimen, but they were hardly identified in the 10% specimen.

The peaks (10.0) and (10.1) of the ε martensite in the 3.8% specimen gradually became weaker with increasing temperature, and they were hardly observed after heating at 473 K. In contrast, the three peaks (110), (200), and (211) of the α' martensite remained almost unchanged up to 773 K, but they became clearly weaker when the temperature was increased above 773 K. Note that the peak (110) of the α' martensite was still higher after heating at 973 K, that is, some α' martensite did not revert to the parent γ phase. The α' martensite in the 10% specimen showed the same transformation process as that observed in the 3.8% specimen.

Figure 4a and b shows the magnetization curves at 300 K of the specimens after tensile deformation by 3.8% and 10% at 77 K and then heating at different temperatures for 3 min, respectively. Based on these curves, the magnetization at 15,000 Oe is shown in Fig. 4c as a function of the temperature. The magnetization at 15,000 Oe in the

Fig. 1 Recovered strain in 304 stainless steel after bending by 3.8% and 10% at 77 K **a** and height change after tensile deformation by 3.8% and 10% at 77 K **b** as functions of temperature, respectively

Fig. 2 Optical micrographs at room temperature of 304 stainless steel after cooling at 77 K **a** and after tensile deformation by 3.8% **b** and 10% **c** at 77 K

Fig. 3 In situ XRD patterns of 304 stainless steel after tensile deformation by 3.8% **a**, 10% **b** at 77 K and then step heating to different temperatures from room temperature. (The peaks of Al_2O_3 were from the protective cover of Al_2O_3)

Fig. 4 Effect of the heating temperature on the amount of α' martensite in 304 stainless steel after tensile deformation by 3.8% and 10% at 77 K and then heated at different temperatures for 3 min. **a**, **b** magnetization curves; **c** magnetization at 15,000 Oe as a function of the heating temperature

10% specimen before heating was 53.3 emu/g, 28 emu/g larger than that in the 3.8% specimen. After heating at 553 K, the magnetization in the 3.8% specimen increased by 5.4 emu/g, but it increased by only 1.1 emu/g in the 10% specimen. Interestingly, after heating at 773 K, the magnetization values dropped again close to the values measured before heating. After heating at 873 K, the magnetizations in the 3.8% specimen dropped remarkably to 16.3 emu/g from 23.5 emu/g; in contrast, it significantly

dropped to 10.7 emu/g from 54.7 emu/g in the 10% specimen. However, these values changed slightly after heating at 973 K. These results revealed that much more α' martensite was produced after deformation by 10% than by 3.8%, while some new α' martensite formed after heating at 553 K. The α' martensite in both specimens began to revert to the γ phase only when the temperature was above 773 K. Especially, the reverse transformation of the α' martensite in the 10% specimen was explosive.

3.3 Relationship Between the Shape Recovery and the Reverse Transformation of ϵ and α' Martensite

Figure 5 replots the recovered strains (Fig. 1) and the magnetizations at 15,000 Oe (Fig. 4) as functions of the heating temperature. Very clearly, the shape recoveries above 773 K in the 3.8% and 10% specimens were closely related to the corresponding reverse transformation of the α' martensite. In addition, a positive relationship existed between the recovered strain and the amount of the α' martensite.

To further clarify whether the stress-induced α' martensite can result in a shape recovery in the 304 stainless steel, we measured the shape recoveries in the specimens after they were bent by 3.8% and 10% at RT, respectively (Fig. 6a). For the 3.8% specimen, no shape recovery occurred when step heating up to 1073 K or directly heating at 1073 K. In contrast, 0.12% strain was recovered in the 10% specimen when heated at 373 K, while it remained unchanged when further step-heated up to 523 K. Above 523 K, it fluctuated around 0.12%. Interestingly, when the specimen was directly held at 1073 K, 0.43% strain was recovered. The XRD patterns revealed that both the peaks of α' martensite and those of ϵ martensite were not clearly identified after tensile deformation by 3.8% at RT (Fig. 6b), while the peaks of α' martensite, (110) and (211), appeared after tensile deformation by 10% (Fig. 6c). In other words, small amounts of α' and ϵ martensite were produced after tensile deformation by 3.8% at RT, but some α' martensite was produced after tensile deformation by 10%. The TEM observation also revealed that some α' martensite was induced in addition to many dislocations after tensile deformation by 10% at RT (Fig. 7a), while the amount of α' martensite was clearly lower as compared to the specimen after tensile deformation by 10% at 77 K (Fig. 7b).

Fig. 5 Relationship between recovered strains and magnetization at 15,000 Oe at room temperature in 304 stainless steel after deformation by 3.8% and 10% at 77 K and then heated at different temperatures, respectively

4 Discussion

4.1 Reasons for Poor SME Induced by Stress-Induced ϵ in Metastable Fe–Cr–Ni Austenitic Stainless Steels

Many researches showed that when the metastable Fe–Cr–Ni austenitic stainless steels are deformed at subzero temperatures, both the stress-induced ϵ and α' martensite can be produced [5, 17, 19, 21, 22]. In addition, the amount of α' martensite rises with increasing strain, while the amount of ϵ martensite first increases with a strain up to 5.0% and then decreases with a further rise in the strain [5]. Our results also confirmed this. The amount of α' martensite rose as the strain increased, while more ϵ martensite was introduced in the specimen deformed by 3.8% than by 10% (Figs. 2–4). Note that a strain of 0.12% was recovered after deformation by 10% at RT and heating at 373 K. Because 373 K is below the A_s temperature of the α' martensite (Fig. 4), a small amount of ϵ martensite was induced after deformation by 10% at RT, even though the peaks of ϵ martensite cannot be clearly identified (Fig. 6b).

Many investigations showed that a recovery strain of 2%–3% can be obtained in the solution-treated Fe–Mn–Si–Cr–Ni-based austenitic stainless steels through the stress-induced ϵ martensite and its reverse transformation [7]. However, in the present metastable austenitic stainless steel the recovery strain was only about 0.3% due to the stress-induced ϵ martensite (Figs. 1a and 3). The recovery strain in the work by Enami *et al.* was also below 1% [6, 21]. This can be ascribed to intrusions of slip due to dislocations motion. The yield strength of the parent phase γ is rather low in type 304 stainless steels. Consequently, the dislocations motion takes place first when deformed [23]. Fujita and Ueda thought that after dislocations first piled up at certain obstacles on the primary slip plane when deformed at 77 K, stacking faults overlapped there regularly as a whole every second layer and produced the ϵ martensite with an increase in the strain [23, 24]. However, the slip deformation is not thermally reversible, and thus, this part of deformation cannot be recovered. On the other hand, the slip deformation damages the reversibility of the stress-induced ϵ martensite [8–12]. Therefore, the recovery strain must be low in type 304 stainless steels. Recently, our results also confirmed that the recovery strain in a 304 stainless steel was remarkably improved due to strengthening of the γ parent phase by a thermo-mechanical training, which comprised 13% tensile deformation at 178 K and subsequent annealing at 973 K for 15 min. After the training, a significant amount of α' martensite was introduced. As a result, the parent phase was strengthened [19].

Fig. 6 Effects of deformation at room temperature on recovered strains and stress-induced martensitic transformation in 304 stainless steel. **a** Recovered strain after bending by 3.8% and 10% followed by step heating; XRD patterns after tensile deformation by 3.8% **b**, 10% **c**

Fig. 7 Bright-field images of transmission electron microscopy of 304 stainless steel after tensile deformation by 10% at room temperature **a**, 77 K **b**, respectively

4.2 Relationship Between the SME and the Stress-Induced α' and its Reverse Transformation Mechanism

The studies by Guy *et al.* [25] revealed that the α' martensite, including that thermal-induced and that stress-induced by cooling or deformation at 77 K, reverts to the γ phase between the A_s and A_f temperatures first by an athermal shear mechanism, followed by an isothermal diffusional mechanism. Furthermore, the α' to γ transformation usually proceeds by the movement of the α'/γ interfaces. Consequently, the reversed austenite has the same orientation as the retained austenite. In addition, the γ phase also nucleates within α' martensite, producing the reversed austenite with a different orientation from the retained phase [25]. The reverse transformation of α' martensite by the shear mechanism can lead to a shape recovery, but a shape recovery cannot be realized by diffusional mechanism. Our present results also clearly confirmed this. After deformation by 10% at 77 K, a strain of 0.5% was recovered due to the reverse transformation of the stress-induced α' martensite whose A_s temperature was 773 K (Fig. 1b). The strain of 0.5% is the difference between the total shape recovery (0.74%) and the shape

recovery due to the stress-induced ε martensite (0.24%). After deformation by 10% at RT and then directly heating at 1073 K, 0.31% strain was recovered due to the reverse transformation of the stress-induced α' martensite. These results clearly revealed that a small part of the α' martensite produced at 77 K or RT can result in a partial shape recovery due to their reverse transformation by a shear mechanism.

Note that in case of step heating, no shape recovery occurred in the specimen deformed by 10% at RT. This result reveals that almost all the α' martensite induced at RT cannot transform into the parent phase by the shear mechanism in case of step heating (Fig. 6). The production of more dislocations at RT than at 77 K may explain this result (Fig. 7). In addition, after deformation by 3.8% at 77 K, both α' martensite and ε martensite were produced. In contrast, small amounts of martensite were produced at RT (Figs. 3 and 6b). This distinctive difference also indicates that the dislocations motion mainly consumes the deformation of 3.8% at RT. This is also the reason why no shape recovery occurred in the specimen bent by 3.8% at RT. However, as the strain increases further, the α' or ε martensite can nucleate at the introduced dislocations and grow. This explains why some α' martensite was produced

after tensile deformation by 10% (Fig. 6c). As a result of the intrusion of many dislocations, the carbides or the γ phase easily nucleate and grow at these defects when slow heating, and thus the crystal lattice correspondence between the γ phase and α' martensite is destroyed below the A_s temperature [3]. Accordingly, no further shape recovery can take place in the specimen after deformation by 10% at RT and slow step heating up to 1073 K. However, when directly heated at 1073 K, the time is insufficient for the formation of carbides or the nucleation of γ phase. Therefore, some α' martensite can revert to the γ phase by the shear mechanism, and thus, a partial shape recovery can be obtained (Fig. 6a).

The increase in the amount of α' martensite after heating at below the A_s temperature has been well documented in metastable austenitic stainless steels which were deeply cooled or deformed at subzero temperatures [5, 25]. At present, there is no clear explanation of this phenomenon. Mangonon *et al.* [5] thought that the increased α' martensite formed martensitically by thermal nucleation, but Guy *et al.* [25] argued that a likely explanation could be the growth of pre-existing α' martensite as the defect structure surrounding it immediately recovered, permitting the relaxation of the α'/γ interfaces.

4.3 Work to be Done in Future

Three different deformation modes occur successively when the metastable Fe–Cr–Ni austenitic stainless steels are deformed at subzero temperatures. The dislocations move first, and then the stress-induced ε martensite is produced, followed by the growth of the stress-induced α' martensite. Note that the α' martensite can be induced at the intersections between two ε martensite plates with different orientations. In addition, it can grow further into the adjacent ε martensite plates as the strain increases, so that finally these ε martensite plates almost disappear [26]. On the other hand, the α' martensite can directly form from the γ parent phase at the regions where the stress is highly concentrated [25]. This is the reason why the amount of α' martensite rises as the strain increases, while the amount of ε martensite first increases and then drops. At present, we cannot distinguish the propensity of reverse transformation by the shear mechanism between the α' martensite from the ε martensite and that from the γ phase. In addition, the growth of the α' martensite from the ε martensite is accomplished by two steps, i.e. $\gamma \rightarrow \varepsilon \rightarrow \alpha'$, while its reverse transformation involves only one step. This arises the question of whether the reverse transformation of the α' martensite from the ε martensite can result in a shape recovery or not. Clearly, it is of both fundamental and practical importance to address the above problems

in the future. This work will provide us a direction for further enhancing the shape recovery in metastable Fe–Cr–Ni austenitic stainless steels.

On the other hand, the morphology of the stress-induced α' martensite may be another reason why the SME associated with the stress-induced α' martensite is quite limited in the present austenitic stainless steel. Normally, the typical morphology of the stress-induced α' martensite is lath-like in the 304 stainless steel when deformation at 77 K [5]. The studies clearly showed that when the morphology of the stress-induced α' martensite is lath-like or lenticular, its resulting SME is also poor in Fe–Ni–C and Fe–Ni–Co–Ti alloys. Only when the morphology of the α' martensite becomes thin plate, a good SME can be obtained in the Fe–Ni–C and Fe–Ni–Co–Ti alloys [27, 28]. The reason is that the thin-plate α' martensite shows good reversibility, while the reversibility is poor for other morphologies of α' martensite. Some studies revealed that the precipitation of a coherent fine second phase and an increase in order degree of austenite can significantly promote the formation of the thin-plate α' martensite [1, 6–13, 17, 20, 21, 29, 30]. Therefore, it is of significance to investigate the precipitation of a coherent fine second phase on the SME in metastable Fe–Cr–Ni austenitic stainless steels by alloying and ageing.

5 Conclusions

To know whether the growth of stress-induced α' martensite and its reverse transformation can result in the shape memory effect in metastable austenitic stainless steels or not, the relationship between the shape recovery and the reverse transformation of the stress-induced martensite was investigated in a 304 stainless steel after deformation at 77 K and at room temperature. The following conclusions have been obtained.

- (1) After deformation at 77 K and then step heating up to 1073 K or after directly heating at 1073 K, two-stage shape recoveries below 440 K and above 773 K because of the stress-induced ε and α' martensite can be obtained in the 304 stainless steel, respectively.
- (2) The α' martensite in the 304 stainless steel induced at room temperature can result in shape memory effect only when directly held at 1073 K. The production of more dislocations at RT than at 77 K may be the reason that the α' martensite induced at RT cannot lead to shape recovery in case of slow step-heating.
- (3) The recovered strains resulting from the stress-induced ε and α' martensite are proportional to the extent of their reverse transformation.

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