Effect of Thermal Cycling on the Martensitic Transformation in an Fe-24Mn-6Si Shape Memory Alloy

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The effect of $\gamma \rightleftharpoons \epsilon$ cyclic transformation on the subsequent $\gamma \rightarrow \epsilon$ martensitic transformation has been studied in an Fe-24%Mn-6%Si shape memory alloy in order to obtain fundamental information on the role of Si in the shape memory effect. The $\gamma \rightarrow \epsilon$ martensitic transformation is markedly enhanced by the thermal cycling between 305 K (below M_s) and 573 K (above A_f), although the M_s temperature slightly decreases. The amount of ϵ martensite at 305 K is less than 30% after a solution treatment and increases to 40% after 10 thermal cycles. Microstructural observation demonstrates that the Fe-Mn-Si alloy exhibits a good reproducibility of ϵ martensite plate formation. Moreover, there are few dislocations in the austenite of a thermal-cycled specimen unlike the case of Fe-Mn binary alloys. These results suggest that the addition of Si plays an important role in the reversible movement of transformation dislocations (i.e., Shockley partial dislocations) during $\gamma \rightleftharpoons \epsilon$ cyclic transformation. It is concluded that the role of Si in the shape memory effect is to make the movement of partial dislocations reversible as well as to restrict the permanent slip in austenite during a shape change.

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I. Introduction

It is now well known that the addition of Si to Fe-high Mn alloys improves a shape memory effect associated with fcc (austenite; γ) $\rightarrow$ hcp (ϵ martensite) transformation⁽¹⁾⁻⁽⁵⁾. Sato *et al.*⁽²⁾ have first presented that a single crystal of an Fe-30%Mn-1%Si alloy[†] exhibits a complete shape memory effect when it is deformed in a specific direction. They⁽²⁾ have clearly shown that the origin of the shape memory effect is the reverse transformation of stress-induced ϵ martensite. Murakami *et al.*⁽⁵⁾ have developed polycrystalline Fe-Mn-Si shape memory alloys, and reported that Fe-Mn-Si alloys with 28~33%Mn and 4~6%Si exhibit a nearly complete shape memory effect. The reason for the improvement of shape memory effect by the addition of Si has been discussed from a viewpoint of a shape change accomplished by stress-induced ϵ martensite without permanent slip⁽⁴⁾⁽⁵⁾.

Recently, Ghosh *et al.*⁽⁶⁾ investigated the effect of thermal cycling on the $\gamma \rightarrow \epsilon$ transformation in Fe-(26~32)%Mn-(3~6)%Si alloys and reported that the amount of ϵ martensite at room temperature significantly increases by the thermal cycling between room temperature and 573 K. This effect of thermal cycling is contrary to that in

Fe-Mn and Fe-Mn-C alloys. It has been reported⁽⁷⁾⁻⁽¹²⁾ that austenite becomes stable against $\gamma \rightarrow \epsilon$ martensitic transformation by thermal cycling in the cases of Fe-Mn and Fe-Mn-C alloys. Tsuzaki *et al.*⁽¹²⁾ reported that M_s temperature^{††} of an Fe-24%Mn binary alloy is decreased by over 60 K and the structure becomes fully austenitic at room temperature after 7 thermal cycles between 305 K (below M_s) and 573 K (above A_f). This discrepancy between Fe-Mn-Si and Fe-Mn alloys strongly suggests that the addition of Si plays an important role in $\gamma \rightarrow \epsilon$ forward and $\epsilon \rightarrow \gamma$ reverse transformations per se in addition to the previously reported effects⁽⁴⁾⁽⁵⁾, i.e., the solution hardening of austenite and the decrease in the Néel point.

In the present study, it is thus intended to clarify the microstructural change by thermal cycling and the effect of upper peak temperature of thermal cycling tests on the martensitic transformation in an Fe-24%Mn-6%Si shape memory alloy in order to obtain fundamental information on the role of Si in the shape memory effect.

II. Experimental Procedure

An Fe-24%Mn-6%Si alloy used in the present study was prepared by induction melting in an argon gas atmosphere. Chemical composition of the alloy is 0.0027C, 5.89Si, 24.29Mn, 0.0008P, 0.0081S, 0.0010N, and the balance Fe (mass%). After homogenization at 1523 K for 86.4 ks, the alloy was hot-rolled into plates with a

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[†]In the present paper, chemical compositions of alloys are expressed in mass%.

^{††}The starting temperature of $\gamma \rightarrow \epsilon$ forward transformation is referred to as M_s , and the starting and finishing temperatures of $\epsilon \rightarrow \gamma$ reverse transformation are referred to as A_s and A_f , respectively.

thickness of 15 mm. After removing surface layer by about 2 mm from both sides, plates were solution-treated at 1273 K for 3.6 ks and then cut or machined to specimens for dilatometric measurement and microstructural observation. Some plates were further rolled around 720 K (above A_f) into sheets with a thickness of 1.2 mm, and then cut and ground into tensile specimens with a 1 mm by 4 mm gage cross section and a 30 mm gage length. Tensile specimens were taken along the rolling direction of sheets.

The changes in M_s , A_s and A_f temperatures by thermal cycling were measured by means of a dilatometer. The amount of ϵ martensite was also determined by dilatometry^{†††}. A schematic diagram of the heat treatment of thermal cycling is shown in Fig. 1. Specimens with 3 mm in diameter and 10 mm in length were first solution-treated at 1273 K for 0.6 ks and cooled to 305 K (just above room temperature). Thermal cycling was then performed between 305 K (below M_s) and 573 K (above A_f). The heating and cooling rates were set at 2.5 K/s. The specimens were not held at either 305 or 573 K. Thermal cycling tests with an upper peak temperature of 873 or 1173 K were also performed. All heat treatments were carried out in an argon gas atmosphere.

For specimens for microstructural observation and tensile test, thermal cycling was conducted after solution treatment at 1273 K for 0.6 ks, using iced water (273 K) and a salt bath set at 573 or 873 K. In these thermal cycling tests, the specimens were kept at the upper temperature for 60 s. Specimens for optical metallography were etched with a 10 g $\text{Na}_2\text{S}_2\text{O}_5$ + 95 ml water solution after electrolytic polishing. Foils for transmission electron microscopy were thinned in a 0.5 g CrO_3 + 5 ml HClO_4 + 95 ml CH_3COOH solution using a twin-jet polisher. The foils were examined with a JEM 200CX operated at 200 kV.

The change in the strength of austenite by thermal cycling was examined by tensile tests at 573 K (above A_f). The tensile tests were performed on an Instron type machine at an initial strain rate of $1.1 \times 10^{-3} \text{ s}^{-1}$. Tensile specimens were heated in an argon gas atmosphere with an infrared ray furnace.

Throughout this paper the treatment, which is com-

Fig. 1 Schematic diagram of the heat treatment of thermal cycling.

^{†††} From the lattice constants of ϵ and γ measured by X-ray analysis, it was obtained that the $\gamma \rightarrow \epsilon$ transformation accompanies 0.43% contraction in specimen length.

posed of the cooling to 305 or 273 K (below M_s) after the solution treatment at 1273 K and the subsequent heating up above A_f , will be referred to as the first cycle of thermal cycling (Fig. 1).

III. Results

1. Change in $\gamma \rightarrow \epsilon$ transformation behavior by thermal cycling

Figure 2 shows the changes in M_s , A_s and A_f temperatures and the amount of ϵ martensite at 305 K by the thermal cycling between 305 and 573 K. Although M_s temperature slightly decreases with increasing number of thermal cycles, the amount of ϵ martensite at 305 K markedly increases by the thermal cycling. The amount of ϵ is less than 30% after the solution treatment and increases up to 40% after 10 thermal cycles. On the other hand, A_s temperature is almost constant, while A_f temperature significantly increases by the thermal cycling. The effect of thermal cycling between 305 and 573 K on the $\gamma \rightarrow \epsilon$ transformation curve during cooling is shown in Fig. 3. The slope of transformation curve just below M_s becomes large by the thermal cycling, while in the first forward transformation the amount of ϵ martensite gradually increases with decreasing temperature. These results clearly indicate that the $\gamma \rightarrow \epsilon$ transformation of the present Fe-Mn-Si alloy is enhanced by the thermal cycling, as Ghosh *et al.*⁽⁶⁾ reported in Fe-(26~32)%Mn-(3~6)%Si alloys.

Figure 4 shows the changes in M_s , A_s and A_f temperatures and the amount of ϵ martensite at 305 K by the thermal cycling between 305 and 873 K. Unlike the result shown in Fig. 2, the enhancement of ϵ martensite forma-

Fig. 2 Changes in M_s , A_s and A_f temperatures and the amount of ϵ martensite at 305 K by the thermal cycling between 305 and 573 K.

Fig. 3 Change in the $\gamma \rightarrow \epsilon$ martensitic transformation curve by the thermal cycling between 305 and 573 K.

Fig. 4 Changes in M_s , A_s and A_f temperatures and the amount of ϵ martensite at 305 K by the thermal cycling between 305 and 873 K.

tion by the thermal cycling is not observed. The amount of ϵ martensite at 305 K, A_s and A_f temperatures are almost constant, whereas M_s temperature somewhat decreases with increasing the number of thermal cycles. When the thermal cycling was performed with a higher upper peak temperature of 1173 K, the result was essentially the same as that for the thermal cycling between 305 and 873 K (Fig. 4). These results indicate that the

effect of thermal cycling depends on the upper peak temperature of thermal cycling, and the enhancement of $\gamma \rightarrow \epsilon$ transformation occurs when the upper peak temperature is low.

2. Microstructural change by thermal cycling

Figure 5 shows the change in optical microstructure at room temperature by the thermal cycling between 273 and 573 K. Micrographs were taken from the same area of a specimen. It is clearly seen that the number of ϵ martensite plates increases with an increase in the number of thermal cycles. It must be emphasized that the Fe-Mn-Si alloy exhibits a good microstructure memory, i.e., a good reproducibility of ϵ martensite plate formation. Martensite plates form in the same position where the martensite plates formed in the last forward transformation. For instance, the ϵ plates indicated by the arrows in Fig. 5(d) are seen in all of the micrographs.

Figure 6 shows the change in optical microstructure at room temperature by the thermal cycling between 273 and 873 K. Each one of the micrographs taken from the same area of a specimen shows somewhat different structure. It is thus concluded that the microstructure memory in addition to the enhancement of the $\gamma \rightarrow \epsilon$ transformation disappears when the thermal cycling is performed with a higher upper peak temperature of 873 K.

Figure 7 shows transmission electron micrographs demonstrating the change in microstructure at room temperature by thermal cycling. The microstructure hardly changes when the thermal cycling is conducted between 273 and 573 K. Dislocations are scarcely observed in the austenite after the first $\gamma \rightarrow \epsilon$ transformation (Fig. 7(a)) and even after 9 thermal cycles between 273 and 573 K (Fig. 7(b)). However, a different substructure is observed in a specimen after 9 thermal cycling between 273 and 873 K (Fig. 7(c), (d)); there are many stacking faults on four $\{111\}_\gamma$ planes in the γ phase after 9 thermal cycles.

3. Change in the strength of austenite by thermal cycling

Figure 8 shows the change in the 0.2% proof stress of austenite at 573 K (above A_f) by the thermal cyclings between 273 and 573 K and between 273 and 873 K. In the case of thermal cycling between 273 and 873 K, specimens were cooled to 573 K from 873 K, and then tensile tests were performed. Zero for the number of cycles in the figure means that a specimen was kept at 573 K (above M_s) after the solution treatment at 1273 K. In both of the thermal cycling tests, the strength of austenite significantly increases with an increase in the number of thermal cycles. In the thermal cycling test between 273 and 573 K, the increase in the 0.2% proof stress by 10 thermal cycles is approximately 50 MPa. The strengthening of austenite is lower in the thermal cycling tests between 273 and 873 K.

Fig. 5 Optical micrographs showing the change in ϵ martensite structure at room temperature by the thermal cycling between 273 and 573 K. Micrographs were taken from the same area of a specimen after (a) 1st, (b) 2nd, (c) 4th and (d) 6th forward transformation.

Fig. 6 Optical micrographs showing the change in ϵ martensite structure at room temperature by the thermal cycling between 273 and 873 K. Micrographs were taken from the same area of a specimen after (a) 1st, (b) 2nd, (c) 4th and (d) 6th forward transformation.

Fig. 7 TEM micrograph showing the change in microstructure at room temperature by thermal cycling. (a) before thermal cycling, (b) after 9 thermal cycles between 273 and 573 K, (c) and (d) after 9 thermal cycles between 273 and 873 K.

Fig. 8 Change in the 0.2% proof stress of austenite at 573 K by thermal cycling. Zero for the number of cycles means that a specimen was kept at 573 K after the solution treatment at 1273 K.

IV. Discussion

1. Effect of thermal cycling on the $\gamma \rightarrow \epsilon$ transformation in Fe-Mn and Fe-Mn-Si alloys

Tsuzaki *et al.*⁽¹²⁾ have investigated the effect of thermal cycling on the $\gamma \rightarrow \epsilon$ transformation in an Fe-24.4%Mn binary alloy, and reported that thermal cycling between 305 K (below M_s) and 573 K (above A_f) suppresses the transformation. Changes in M_s , A_s , A_f temperatures and the amount of ϵ martensite at 305 K by the thermal cy-

cling between 305 and 573 K in the Fe-24.4%Mn alloy reported by Tsuzaki *et al.*⁽¹²⁾ is shown in Fig. 9. M_s temperature is 366 K after the solution treatment at 1273 K, while it decreases by over 60 K and the structure becomes fully austenitic at room temperature after 7 thermal cycles. Furthermore, they⁽¹²⁾ observed a large number of dislocations in the austenite after 9 thermal cycles between 273 and 573 K (Fig. 10⁽¹²⁾). Moreover, the Fe-24.4%Mn alloy does not exhibit the microstructure memory. Nakagawara *et al.*⁽¹³⁾ have examined the change in the strength of austenite in an Fe-24.1%Mn binary alloy by the thermal cycling between 273 and 573 K. They reported that the 0.2% proof stress of the austenite at 573 K (above A_f) is 40 MPa after a solution treatment at 1273 K and increases to 250 MPa by 10 thermal cycles. These results in the Fe-Mn binary alloys⁽¹²⁾⁽¹³⁾ are much different from the results in the present Fe-24%Mn-6%Si alloy. The effect of thermal cycling in the Fe-24%Mn-6%Si alloy can be characterized by the enhancement of $\gamma \rightarrow \epsilon$ transformation and the appearance of microstructure memory when the thermal cycling is conducted with an upper peak temperature of 573 K. As was shown in Fig. 8, the increase in the 0.2% proof stress of austenite by thermal cycling is significant (50 MPa) but small compared with the case in the Fe-24.1%Mn binary alloy (210 MPa). Dislocations are scarcely observed in the austenite even after 9 thermal cycles between 273 and 573 K (Fig. 7(b)), unlike the case of the Fe-24.4%Mn alloy (Fig. 10).

It has been well established that $\gamma \rightarrow \epsilon$ forward and $\epsilon \rightarrow \gamma$

Fig. 9 Change in M_s , A_s and A_f temperatures and the amount of ϵ martensite at 305 K by the thermal cycling between 305 and 573 K in an Fe-24.4 mass%Mn binary alloy⁽¹²⁾.

Fig. 10 TEM micrograph demonstrating the introduction of many dislocations into the austenite by the thermal cycling between 273 and 573 K in an Fe-24.4 mass%Mn binary alloy⁽¹²⁾. The micrograph was taken from a specimen after 9 thermal cycles.

reverse transformations occur by motion of $a/6\langle 112 \rangle$ Shockley partial dislocations⁽¹⁾⁽²⁾. If the partial dislocations move reversibly in the forward and reverse transformations, the structure of austenite does not change by thermal cycling and the reproducibility of ϵ martensite plate formation can be expected. Thus, the microstructure memory and the dislocation-free austenite observed in the present Fe-24%Mn-6%Si alloy strongly suggest that most of transformation dislocations (i.e., Shockley partial dislocations) can move reversibly unlike the case in the Fe-Mn binary alloys. The difference in the effect of thermal cycling between the Fe-Mn-Si and Fe-Mn alloys indicates that the addition of Si plays an important role

in the crystallographic reversibility during the $\gamma \rightleftharpoons \epsilon$ cyclic transformation.

When an upper peak temperature of thermal cycling is higher (i.e., 873 K), the microstructure memory becomes poor (Fig. 6). Comparing the values of 0.2% proof stress of austenite after 1 thermal cycle in Fig. 8, it is clear that the strength of austenite at 573 K is decreased by heating a specimen up to 873 K. This decrease of the strength of austenite is apparently caused by recovery of austenite, and suggests that dislocation climbing and dislocation rearrangement frequently occur at 873 K. This means that dislocations no longer remain on the $\{111\}_\gamma$ planes where the dislocation moved in the reverse transformation. In this case, the microstructure memory can not be expected. Many stacking faults observed in the austenite after 9 thermal cycles between 273 and 873 K (Fig. 7(c)) may have been caused by dislocation rearrangement in the recovery process of austenite, although a detail mechanism for the formation of stacking faults is not known at present. It is concluded that the appearance of microstructure memory is closely associated with the degree of recovery of austenite during heating after the $\epsilon \rightarrow \gamma$ reverse transformation.

Recently, Shiming *et al.*⁽¹⁴⁾ reported that an Fe-29.4%Mn-6.2%Si alloy shows a small but an apparent two-way shape memory effect. Since a two-way shape memory effect is apparently associated with a reversible movement of partial dislocations, their result⁽¹⁴⁾ is consistent with the present result that the Fe-24%Mn-6%Si alloy exhibits the crystallographic reversibility (i.e., the microstructure memory) during the thermal cycling. It should be, however, noted that the heating temperature after a shape change is one of the important points to obtain a good two-way shape memory effect, because the dislocation rearrangement in austenite occurs more quickly at higher temperatures.

2. Role of Si in the shape memory effect

It is now well known that the addition of Si to Fe-high Mn alloys improves the shape memory effect associated with $\gamma \rightarrow \epsilon$ martensitic transformation⁽¹⁾⁻⁽⁵⁾. It has been established that the origin of the shape memory effect in Fe-Mn-Si alloys is the reverse transformation of stress-induced ϵ martensite⁽¹⁾⁽²⁾. Thus, a shape change must be accomplished by stress-induced $\gamma \rightarrow \epsilon$ martensitic transformation without permanent slip in order to obtain a good shape memory effect. From this viewpoint, the effects of Si have been discussed in connection with the solution hardening of austenite and the decrease in the Néel point⁽⁴⁾⁽⁵⁾. The decrease in Néel point of austenite by the addition of Si increases the driving force for the $\gamma \rightarrow \epsilon$ transformation, and makes the critical stress for the formation of ϵ martensite lower. Moreover, the strength of austenite becomes higher by the addition of Si due to solution hardening. These effects of Si inhibit the intrusion of permanent slip in austenite, so that a good shape memory effect can be expected.

However, Tomota *et al.*⁽¹⁵⁾ have recently reported that Fe-Mn binary alloys (Fe-15.7%Mn and Fe-24.4%Mn)

do not exhibit a good shape memory effect, even when a shape change is accomplished by the stress-induced $\gamma \rightarrow \epsilon$ martensitic transformation without permanent slip in austenite. They⁽¹⁵⁾ applied a tensile stress just above the 0.2% proof stress of austenite at 523 K to a specimen at 523 K (above A_f) and then cooled the specimen to room temperature (below M_s) under the constant stress. During the cooling the specimen exhibited a large extension (approximately 3% in the specimen length). This extension is attributed to the stress-induced $\gamma \rightarrow \epsilon$ martensitic transformation, and the intrusion of permanent slip is negligible because of the low stress level. Nevertheless, the Fe-Mn alloys did not exhibit any significant shape recovery during the subsequent heating up to above A_f . In contrast to the behavior of Fe-Mn alloys, they⁽¹⁵⁾ observed a large amount of shape recovery in the Fe-23.9%Mn-5.88%Si alloy. These results⁽¹⁵⁾ strongly suggest that the reason for the improvement of shape memory effect by the addition of Si should be discussed in a different way from the previous discussions⁽⁴⁾⁽⁵⁾.

As described before, the origin of the shape memory effect in Fe-Mn-Si alloys is the reverse transformation of stress-induced ϵ martensite. Thus, in order to obtain a complete shape recovery, the reverse transformation must be accomplished by the backward movement of partial dislocations operated in the forward transformation. This is apparent, but less attention has been given to this point. The results of thermal cycling tests in the present Fe-24%Mn-6%Si alloy clearly indicate that the movement of partial dislocations is essentially reversible during the forward and reverse transformations. On the contrary, the reversibility of the dislocation movement is poor in the Fe-Mn binary alloys, as suggested by the fact⁽¹²⁾ that a large number of dislocations are introduced into austenite after the thermal cycling in the Fe-24.4%Mn alloy (Fig. 10). Consequently, it is concluded that the role of Si in the shape memory effect is to make the movement of partial dislocations reversible as well as to restrict the permanent slip in austenite during a shape change.

A backward movement of the partial dislocations operated in forward transformation can be assisted by the back stress formed by martensitic transformation shear. If a plastic accommodation takes place in the austenite of a tip of the ϵ martensite plate, the back stress decreases. The solution hardening of austenite by Si may restrict the plastic accommodation. In this sense, the solution hardening by Si is important for the reversible movement of the partial dislocations. Another possible explanation is the existence of short range ordered structure or small coherent ordered particles in austenite, as was implied by Sade *et al.*⁽¹⁶⁾ If austenite has an ordered structure, the free energy of austenite becomes higher by an irreversible movement of the partial dislocations and thus a reversible movement becomes preferential. However, ordered structures or ordered particles in austenite have not been reported in the Fe-Mn-Si alloys. We need further investigation in order to clarify the mechanism for the reversible movement of partial disloca-

tions by the addition of Si.

V. Conclusions

The effect of thermal cycling on the martensitic transformation in an Fe-24%Mn-6%Si shape memory alloy has been studied in order to obtain fundamental information on the role of Si in the shape memory effect. Main results obtained are as follows.

(1) The $\gamma \rightarrow \epsilon$ martensitic transformation is markedly enhanced by the thermal cycling between 305 K (below M_s) and 573 K (above A_f). Microstructural observation demonstrates that the alloy exhibits a good microstructure memory, i.e., a good reproducibility of ϵ martensite plate formation. Moreover, there are few dislocations in the austenite even after 9 thermal cycles between 273 and 573 K. These results are in contrast to the results reported in Fe-Mn binary alloys.

(2) When the thermal cycling tests are conducted with a higher upper peak temperature (873 or 1173 K), the enhancement of ϵ martensite formation is not observed and the microstructure memory becomes poor. This may be associated with faster recovery of austenite (i.e., dislocation rearrangement) after reverse transformation at higher temperatures.

(3) The strength of austenite is significantly increased by the thermal cycling between 273 and 573 K. The 0.2% proof stress of austenite at 573 K is 100 MPa after the solution treatment and increases to 150 MPa by 10 thermal cycles. The increase in the 0.2% proof stress (50 MPa) is however small compared with that reported in an Fe-24.1%Mn binary alloy (210 MPa).

(4) The microstructure memory and the dislocation free-austenite observed in the present Fe-Mn-Si alloy strongly suggest that the movement of partial dislocations is essentially reversible during the forward and reverse transformations unlike the case of Fe-Mn alloys. It is concluded that the role of Si in the shape memory effect is to make the movement of partial dislocations reversible as well as to restrict the permanent slip in austenite during a shape change.

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