In-situ TEM Observation of $\gamma \rightarrow \varepsilon$ Martensitic Transformation during Tensile Straining in an Fe–Mn–Si Shape Memory Alloy

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Through in-situ TEM observation on an Fe–30.3 mass%Mn–6.1 mass%Mn–Si shape memory alloy under extending at room temperature, it is found that on a lower applied stress level, the $\varepsilon$-martensite plates are preferentially formed at interfaces of pre-existing overlapped stacking faults or $\varepsilon$-martensites with $\gamma$-phase especially at those near by the stress-concentrated locations e.g. inclusions. It is also observed that the $\varepsilon$-martensite is growing by the motion of partial dislocations resulting in the widening and thickening of stacking faults until it is stopped by another martensite plate or grain boundary. On higher stress level, groups of highly oriented $\varepsilon$-martensite plates are formed by an autocalytic activating mechanism suggested by the present authors.

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I. Introduction

Recently, Fe–Mn–Si based shape memory alloys have attracted increasing attentions owing to their promising shape memory effect (SME) and cheap cost(1–3). The SME of Fe–Mn–Si based alloys is concerned in the $\gamma$(fcc)$\rightarrow\varepsilon$(hcp) martensitic transformation induced by strain and its reverse transformation. The mechanism of $\gamma \rightarrow \varepsilon$ transformation and related SME is quite different from that of NiTi and Cu-based SMAS(4). As is well known that the $\frac{a}{2}[110] \pm \frac{a}{2}[011] \pm \frac{1}{3}[1\bar{1}1] \pm \frac{a}{6}[\bar{1}2],$

The overlapping of stacking faults on every second close packed plane in fcc phase forms $\varepsilon$-martensite.

Therefore the stacking fault of parent phase plays an important role for the fcc$\rightarrow$hcp transformation. On the nucleation mechanism of $\varepsilon$-martensite, however, a consistent conclusion has not yet been obtained. There mainly are two mechanisms on the nucleation of $\varepsilon$-phase in previous studies in stainless steels, high manganese steels and cobalt based alloys etc., i.e. the screw-pole mechanism proposed by Seeger(5) and the mechanism of spontaneous nucleation of stacking fault(6–8). Fujita and Ueda(9) suggested that the formation of $\varepsilon$-martensite is from the irregular overlapping process of stacking faults to the regular one through cross-slip of partial dislocations leaving stair-rod dislocation at the intersections of the two shear planes.

In Fe–Mn–Si based alloys usually with a very low stacking fault energy, the stacking faults also play a decisive role on the formation of $\varepsilon$-martensite and SME. Hoshino et al.(10,11) discovered in an Fe–Mn–Si alloy by in-situ observation that the $\varepsilon$-martensite formed by a pole mechanism and a pole-dislocation was found at a small angle boundary possibly generated by a reaction,

II. Experimental Procedure

The tested material was a 1.2 mm thick hot-rolled plate of an Fe–30.3Mn–6.1Si (mass%) alloy, from which strips with a size of 65 mm $\times$ 3 mm $\times$ 1 mm were cut by the spark machine, and sealed into a quartz tube filled with

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argon. The sealed specimens were solution-treated at 1273 K for 30 min followed by quenching into ice-water. After thinning to 0.05 mm by mechanical grinding and chemical polishing, thin foils with a size of 7 mm × 3 mm × 0.05 mm for tensile holder of TEM were prepared by two jet electro-polishing in a solution of 100 mL perchloric acid and 900 mL acetic acid at 287 K. In-situ deformation was carried out at 200 K with a Hitachi 700 type TEM equipped with a single tilt tensile specimen holder.

In order to observe the thermo-mechanical training effect, some specimens were in-situ deformed secondary after annealing the first tensioned foil in vacuum furnace at 873 K for 10 min. During the observation under deformation, thin foils were tilted synchronously, in order to obtain the best contrast always.

III. Experimental Results

1. TEM observation of as quenched specimen

TEM observation of the specimen quenched from 1273 K reveals that some ε-martensite plates are already present as shown in Fig. 1, which is consistent with the result of $M_t$ temperature determined by dilatometer as ∼329 K. It is visible that one end of stacking faults (S.F.) starts to appear at the interface of these ε-martensite plates. Figure 2 shows that in the other field of the same specimen many long stacking faults are aligned in parallel to each other and some of them are overlapped. They are intersected by other group of stacking faults situated in edge-on position shown as a very thin and straight line. Selected area electron diffraction patterns taken from this area indicate the fcc structure. Trace analysis reveals that these stacking faults are aligned in parallel to the trace of the {111} planes. Several dislocation lines also can be seen in Fig. 2.

2. In-situ deformation

Figure 3(a) shows the bright field image of the pre-existing overlapping S.F. and ε-martensite before deformation, in which only darker and denser contrasts can be seen by virtue of the thicker area of the foil. While driving the tensile holder to let the specimen extend 28 µm, the new ε-martensite plates are preferentially induced from the site of the pre-existing martensite especially near by the inclusion particles A, B and C as pointed in Fig. 3(b). Also ε-martensites nucleate from some small stacking faults dissociated from perfect dislocation and grow by the movement of Shockley partial dislocation as marked of D toward C shown in Fig. 3(b). After extending 40 µm on the one side, some ε-martensite plates continuously increase their length until stop at grain boundary or intersect with other ε-martensite e.g. D→D' and G→G' in Fig. 3(c); On the other hand new ε-martensite plates further nucleate at the pre-existing stacking faults and ε-martensites as pointed as H→H', I→G' and J→B shown in Fig. 3(c). While further increasing the displacement to 48 µm, much more nucleating sites of ε-martensite are activated, and the autocatalytic phenomenon which just likes the Dominoes is observed that ε-martensite plates grow quickly one by one in parallel to {111}, plane, as marked the area F shown in Fig. 3(d). For further straining (displacement of 52 µm), ε-martensite plates almost occupy whole area F and overlap each other as shown in Fig. 3(e) (arrows indicate the extending direction of ε martensite plates). In the other area the ε-
Fig. 3 Successive stages of formation processes of ε-martensite upon extending. (a) as quenching state (solution treated at 1273 K, 30 min, followed by quenching into ice water). (b) (c) (d) and (e) are photographed during extending by a displacement of 28 µm, 40 µm, 48 µm and 52 µm, respectively.
3. In-situ observation of the secondary deformation

After the first extending in TEM some specimens are put into a vacuum furnace for annealing at 873 K for 10 min followed by the secondary in-situ observation on extending. Figure 4(a) is as annealing state before the secondary deformation, where a lot of stacking faults aligning in parallel each other along different orientations can be seen. There also exist many dislocations in the stacking fault plane as marked A, and near by the inclusion, I, as shown in Fig. 4(a). While extending the specimen by displacement of 45 µm, the new stacking fault is formed adjacent to the pre-existing S.F., such as A by exciting the movement of partial dislocations as shown in Fig. 4(b), and the two stacking faults are overlapped partly. When further increasing the displacement to 49 µm, the more parallel stacking faults are formed adjacent to A; and also to B (see Fig. 4(c)), still further to C, as shown in Fig. 4(d). At the same time a new parallel stacking fault D is excited and formed between the pre-existing stacking

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**Fig. 4** Successive stage of the formation of a group of stacking faults upon extending in the specimen gone through once thermo-mechanical cycling. (a) as cycling annealing state (873 K, 10 min, followed by air cooling). (b) (c) and (d) are photographed during extending by displacement of 45 µm, 49 µm and 54 µm, respectively.
faults A and C, (see Fig. 4(d)). Another stacking faults situated on the unpreferential orientation with applying stress such as E and F keep no change during extension. Figure 5(a) is a magnified image of Fig. 4(a), where dislocations lying on stacking fault plane A are clearly visualized. When the specimen is extended by 54 μm some dislocations such as 1, 2, 3, 4, 5 and 6 decompose into two partial dislocations trailing a stacking fault ribbon as shown in Fig. 5(b). However, because of the larger internal stress field set by the pile up of these dislocations in that area, the stacking fault ribbons cannot be extended wider. When further increasing the displacement to 58 μm some dislocations such as 7 and 8, decompose into a peach-shaped stacking fault bound with a part of perfect dislocation retained like a "tail" as shown in Fig. 5(c), where some other parallel short ε-martensite plates (9 and 10) are nucleated from dislocations along the direction vertical to the original stacking faults under such a stronger applied stress.

IV. Discussion

From the results of the present in-situ TEM observation, it may be suggested that at a lower applied stress level the ε-martensite plates can preferentially be formed by the nucleation at the stress concentrated sites such as the location near by inclusion particles (A, B and C as shown in Fig. 3(b)) and by continuously growing with the aid of the motion of partial dislocation at the interfaces of pre-existing overlapped stacking faults with γ-phase

Fig. 5 Decomposition of perfect dislocations upon extending. (a) before extending, which is the magnified image of Fig. 4(a); (b) extending by 54 μm, (magnified image of Fig. 4(d)), dislocations lie in the stacking fault A; (c) extending by 58 μm, dislocations are near by stress concentrated part e.g. inclusion I.
as shown at B, E and I in Fig. 3(b) and (c).

At a higher applied stress level, on one hand the individual ε-martensite plates are formed at the interfaces of pre-formed ε-martensite plates of which various variants constitute frames of martensite, such as M and N in Fig. 3(d), and on the other hand the parallel ε-martensite plates group can be formed by an "autocatalytic activating mechanism", i.e. a growing ε-martensite plate impinges upon another pre-existing ε-martensite plate, or inclusion and grain boundary, the local stress concentration generated at these junctions will assist to induce a series new stacking faults in their neighboring slip planes in parallel to the growing one; by repeating this process, a group of highly oriented ε-martensite plates are formed, as shown at "F" in Fig. 3(d). This phenomenon is consistent with the result reported by Inagaki[12] in the Fe-14%Mn-6%Si-9%Cr-6%Ni alloy by a normal TEM observation. In the present study it is directly observed on extending.

After the first thermo-mechanical cycle annealing, there exist a lot of parallel stacking faults, which is consistent with the experimental result of the stacking fault probability measurement by X-ray profile analysis[13], so that the "autocatalytic activating mechanism" will be easy to be activated at a lower applied stress level on the second deformation resulting in much more highly oriented ε-martensite plates to be formed. This may be the reason of the improvement of shape recovery rate by the thermo-mechanical training[14].

In the in-situ observation under extending we have tried to find if any nucleation of stacking fault or ε-martensite occurred at large angle grain boundaries, however no pole-dislocation could be found at grain boundaries as reported by Hoshino et al.[10] and also it has not been directly observed that stacking faults were successively emitted from grain boundary[12]. Contrarily it is observed that ε-martensite plates are growing toward grain boundary until they are stopped by it. As a general rule grain boundaries are the preferential nucleation sites for a new phase, but for the Fe-Mn-Si based alloys with low stacking fault energy upon an applied stress the stacking faults may be more preferentially nucleated at interfaces of γ→ε phase, twin boundary and sub-grain boundary where the atoms of γ-phase only need move a slight displacement to transform into γ-lattice.

V. Conclusions

Through in-situ TEM observation on an Fe-30%Mn-6Si shape memory alloy under extending at room temperature, the following conclusions can be drawn:

1) On a lower applied stress ε-martensite plates are preferentially formed at interfaces of pre-existing ε-martensite especially of that near by the stress-concentrated locations e.g. inclusions. The ε-martensite plates also preferentially grow at the stacking faults decomposed by a perfect dislocation by the movement of partial dislocation resulting in the widening of the stacking fault.

2) On a higher applied stress ε-martensite plate groups are formed by an "autocatalytic activating mechanism", i.e. a series of partial dislocations in their neighboring slip planes are successively activated resulting in that a group of parallel stacking faults are formed and overlapped each other so that a group of highly oriented ε-martensite are obtained.

3) On the secondary deformation after once thermo-mechanical cycling, the "autocatalytic activating mechanism" is easier to occur resulting in more highly oriented ε-martensite group, which may be the reason of improving SME by the thermo-mechanical training in Fe-Mn-Si based alloys.

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