**In-situ TEM Observation of ε→γ Transformation during Heating in an Fe–Mn–Si Shape Memory Alloy**

Bohong Jiang*, Tsugio Tadaki**, Hirotaro Mori*** and T. Y. Hsu (Xu Zuyao)*

*Department of Materials Science, Shanghai Jiao Tong University, Shanghai 200030, P. R. China
**Department of Structural Characterization and Design Division of Advanced Materials Science and Technology, ISIR, Osaka University, 8-1 Mihogaoka, Ibaraki, Osaka 567, Japan
***Research Center for Ultra-High Voltage Electron Microscopy, Osaka University, Yamada-oka, Suita, Osaka 565, Japan

In-situ TEM observation of the deformed specimen on heating from R.T. up to 873 K in an Fe-30.3 mass%Mn-6.1 mass%Mn shape memory alloy has been performed. It is found that the thicker ε-martensite plates will be disintegrated layer by layer through the reverse motion and annihilation of partial dislocations and the last induced ε-martensite will be the first to disappear. The in-situ observations on heating of the secondary deformed specimen after once thermo-mechanical cycling has also been conducted, from which the improvement mechanism of thermo-mechanical training can be well explained.

(Received April 14, 1997)

Keywords: iron-manganese-silicon, shape memory alloy, in-situ transmission electron microscopy, ε→γ transformation, partial dislocations

I. Introduction

Fe–Mn–Si based alloys are known to show a promising shape memory effect (SME)\(^1\)\(^-\)\(^3\). In our previous paper\(^4\) the in-situ TEM observation of γ→ε martensitic transformation process under extending in an Fe–30.3 mass%Mn–6.1 mass%Mn shape memory alloy has been performed. In order to know how the stress-induced ε-martensites reversely transformed into γ-phase on annealing the in-situ TEM observation under heating is conducted on the same alloy in the present study.

II. Experimental Procedure

The tested alloy (Fe–30.3Mn–6.1Si (mass%)), was as same as in the previous paper\(^4\). The thin foil specimens with a size of 7 mm × 3 mm × 0.05 mm were prepared by two jet electro-polishing in a solution of 100 mL perchloric acid and 900 mL acetic acid at 287 K. After in-situ deformation by using the tensile specimen holder in a Hitachi 700 type TEM, the thin foils were cut into disks of 3 mm diameter and then in-situ observed at the same TEM with a heating stage specimen holder.

III. Experimental Results

Figure 1(a) shows the over-lapped stacking faults and ε-martensite plates from the TEM image of the foil strained by 92 μm at room temperature before heating. On heating this thin foil within the TEM, those shorter thinner and finally strain-induced ε-martensite plates such as E\(_1\) and E\(_2\) etc. start disappearing at 371 K through the movement of partial dislocation and the annihilation of them at the place intersected with other thick ε-martensite as shown in Fig. 1(b). With increasing the temperature up to 508 K, the thicker ε-martensite plates such as E\(_3\) and E\(_4\) gradually decrease their degrees of overlapping also by the movement and elimination of the Shockley partial dislocations as shown in Fig. 1(c). Through the repeating of such layer-by-layer removal of individual stacking faults at last the thick ε-martensite plate such as E\(_3\) is completely eliminated at a temperature of 703 K as shown in Fig. 1(d). Figure 1(d) also illustrates that the Shockley partial dislocations are observed to move toward the grain boundaries of austenite in which they are finally annihilated, as indicated by arrows, resulting in reducing the width of stacking faults and the thickness of their overlapping (E\(_a\) and E\(_b\)). The ε-martensite plates initially formed constructing rectangular frames such as E\(_a\) and E\(_b\) are still remaining at 703 K, but disappeared on further annealing. On heating up to 843 K, most of the ε-martensite plates and stacking faults are eliminated, but in some visual fields it is observed that the stacking faults are contracting in the length direction by the wriggle movement of partial dislocation located on one side of fault and contracting in the edge direction too, as clearly shown in Fig. 1(e) and (f). Owing to the volatile contamination of the specimen in such a high temperature, the image of diffraction contrast becomes blurred.

The following series of pictures are taken from the in-situ observation during heating the specimen which has

---

\(^1\) Present address: Division of Physics, Department of Natural Science, Osaka Women’s University, 2-1 Daisei-cho, Sakai, Osaka 590, Japan.
Fig. 1 Successive annihilation of stacking faults and decomposition of $\varepsilon$-martensite plates observed in a thin foil (prepared from a thin strip deformed in TEM) upon in-situ heating within TEM, photographed at indicated temperature. (a) before heating, 297 K, (b) heating up to 371 K, (c) heating up to 508 K, (d) heating up to 703 K and (e) heating up to 843 K.
Fig. 2 Successive decomposition processes of ε-martensite plates observed in a thin foil (prepared from the specimen undergone once thermo-mechanical cycling plus secondary deformation) on heating within TEM, photographed at indicated temperature. (a) 297 K, (b) 533 K, (c) 550 K, (d) 603 K and (e) 689 K.
undergone once thermo-mechanical cycling plus secondary deformation. Figure 2(a) shows multiple variants of ε-martensite plates and overlapped stacking faults after secondary deformation at room temperature. On heating within TEM up to 533 K single layer of stacking faults and short segments of ε-martensite formed latest disappear firstly. Some narrow and parallel stacking faults piled up contract into perfect dislocation lines as shown in Fig. 2(b). Increasing temperature up to 550 and 603 K, some long stacking fault ribbons composed of several stacking faults ‘block’, everyone with two separated partial dislocations, as shown in Fig. 2(c) indicated by E₁ and E₂ are broken early or late into several parts (in this picture only two parts can be visual) by virtue of the contracting of the stacking faults as shown in Fig. 2(c) and (d), respectively. On heating to higher temperature (e.g. 689 K), most of stacking faults disappear and remain perfect dislocations (such as D₁ and D₂) and very narrow stacking faults (such as E₂ in Fig. 2(e)). While cooling back to room temperature, some perfect dislocations again decompose into two partial dislocations with a very narrow stacking fault ribbon marked D₁ or narrower one, marked E₂ as shown in Fig. 3(a). In some other visual fields, we can see wider and very wide remained stacking faults after cooling as shown in Fig. 3(b) and (c), respectively. At some grain boundaries a lot of short stacking fault segments arranged in parallel can be seen (Fig. 3(c)). All these stacking faults can be the nuclei of ε-martensite both for thermal induced and stress induced.

IV. Discussion

There are lots of overlapped stacking faults and ε-martensite plates with different variants forming frame structures in specimen after deformation. On heating the ε-martensite reversibly transforms to γ-phase by the motion of partial dislocations and annihilation into their sinks such as interfaces of martensite plates and grain boundaries etc. Under the in-situ observation on heating, the motion of partial dislocation is clearly visual, however the rate of motion is too fast to be recorded by photograph.

The ε-martensite plates that first start disappearing are those shorter, thinner and finally strain-induced one in
the frame-structure. Afterwards the thicker and longer \(\varepsilon\)-martensite plates are disintegrated layer by layer and eliminated at last through the movement and annihilation of partial dislocations. This is in agreement with the results of Inagaki’s work\(^5\). Some of stacking faults that disappear or contract into perfect dislocations on heating are formed again on cooling to room temperature which will become the nuclei of \(\varepsilon\)-martensite on further deformation. Therefore after every thermo-mechanical cycle annealing more stacking faults remain which will assist the formation of strain-induced \(\varepsilon\)-martensite resulting in the improvement of the SME.

V. Conclusions

Through in-situ TEM observation on an Fe-30Mn-6Si shape memory alloy and under heating to different temperature, the following conclusions can be drawn:

On heating the deformed specimen, the \(\varepsilon\)-martensites plates reversibly transform to \(\gamma\)-phase by the movement of partial dislocations and annihilation into their sinks such as interfaces of martensite and grain boundaries etc. The last induced \(\varepsilon\)-martensite will be the first to disappear. The thicker \(\varepsilon\)-martensite plates will be disintegrated layer by layer through the reverse motion and annihilation of partial dislocations.

Acknowledgment

The authors would like to express their appreciation that this work is supported by the National Natural Science Foundation of China, and the TEM work is performed with the support of the JSPS Cooperation Programs in the Institute of Scientific and Industrial Research, Osaka University. The grateful acknowledge should be sent to the host scientist, Prof. Y. Hirotsu, and Dr. H. Yasuda as well as Mr. Y. Murai of Osaka University for their sincere and cordial help. The one of the authors (Prof. Jiang) also would like to thank Prof. T. Maki and Dr. K. Tsuzaki of Kyoto University for profitable discussion.

REFERENCES