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H. Ohtsuka, S. Kajiware, T. Kikuchi, T. Ishihara, K. Nagai. Growth Process and Microstructure of ϵ Martensite in an Fe-Mn-Si-Cr-Ni Shape Memory Alloy. Journal de Physique IV Proceedings, 1995, 05 (C8), pp.C8-451-C8-455. 10.1051/jp4:1995868 . jpa-00254117

HAL Id: jpa-00254117

<https://hal.science/jpa-00254117>

Submitted on 1 Jan 1995

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Growth Process and Microstructure of ϵ Martensite in an Fe-Mn-Si-Cr-Ni Shape Memory Alloy

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Abstract Growth process and microstructure of ϵ martensite formed by cooling in thermomechanically-treated Fe-Mn-Si-Cr-Ni shape memory alloys have been investigated in order to clarify whether or not very fine martensite plates such as observed in the case of stress-induced transformation are also formed in the case of simple cooling. Either such fine martensite plates or the lamella structure which consists of h.c.p. and f.c.c. phase is not observed in the case of the simple cooling, both in thermomechanically and non-thermomechanically-treated specimens, but there is a tendency that thinner martensite plates are formed in the former specimens. In the present study, additional diffraction spots, besides h.c.p. spots, are formed, which may have resulted from double hexagonal structure or some kinds of long period stacking structures. Stacking fault density in the h.c.p. martensite was examined by electron diffraction and it was found that the values of fault parameter α are almost the same in both the stresses-induced and thermally-induced martensites.

1. INTRODUCTION

Fe-based shape memory alloys have been developed as alternatives to Ti-Ni shape memory alloy because of its low cost, good workability and high strength, and are classified into Fe-Mn-Si[1-9], Fe-Mn-Si-Cr-Ni[10-16], Fe-Ni-Co-Ti[17-20], Fe-Ni-C[21,22] alloys. Among these alloys Fe-Mn-Si alloys which show perfect shape memory effect in some conditions have been most extensively studied in the past decade. Recently it was found that the addition of Cr and Ni to this alloy system greatly improves the corrosion resistance property. However, newly developed Fe-Mn-Si-Cr-Ni alloys do not show perfect shape memory effect in the as-solution treated state. To obtain a nearly perfect shape memory, some kinds of thermomechanical treatments, such as so-called "training" are necessary. As one of the fundamental researches to make clear the mechanism of the improvement of shape memory effect by the thermomechanical treatments, formation process and microstructures of the stress-induced martensite in Fe-Mn-Si-Cr-Ni shape memory alloys have been investigated in detail very recently by Kikuchi et al[23,24]. The essential feature of their results is that the $\gamma \rightarrow \epsilon$ transformation by deformation is basically accomplished, in both the optical and electron microscopic scales, by formation of groups of very thin martensite plates and subsequent coalescence of these plates. This transformation mode produces the deformation band consisting of nanometric lamella structures with a mixture of h.c.p. and f.c.c. phase, which were revealed by HREM study by Ogawa and Kajiwara[25]. Such characteristic transformation mode may be attributed to the existence of many stacking faults introduced by thermomechanical treatments of austenite, which can serve preferential nucleation sites for the f.c.c. to h.c.p. transformation. In the case of the transformation by simple cooling, it is then expected that very fine martensite plates are also formed in the similarly thermomechanical-treated specimens. The primary purpose of the present work is to clarify this point. The stacking fault density in the h.c.p. martensite itself is also examined to elucidate the difference in the transformation mechanism between the deformation-induced and thermally-induced martensites.

2. EXPERIMENTAL

The specimen used in this study is an Fe-14Mn-6Si-9Cr-5Ni(wt%), which is one of the most practically useful Fe-based shape memory alloys. One group of specimens were sealed in evacuated silica capsules and solution-treated at 1320 K for 1.8 ks and air-cooled (these specimens are denoted as "non-

thermomechanically-treated specimens" hereafter). Another group of specimens were solution-treated, cold-rolled by 10% and held at 970 K for 0.6 ks (these specimens are denoted as "thermomechanically-treated specimens" hereafter). The shape memory effect is improved by this thermomechanical heat treatment from 50% to 80%. These specimens were chemically polished by 0.1 mm to remove the surface layer whose chemical composition may be different from that inside the specimen, and cooled to 77 K by immersing in liquid nitrogen. The microstructure of ϵ martensite was observed by JEM-2000FX operated at 200 kV. All the bright field images and diffraction patterns were taken under the condition that the incident electron beam is parallel to the habit plane of ϵ martensite plate. The shift of h.c.p. spots was measured from the electron diffraction patterns and the density of stacking fault was calculated from this shift, using the same method as that employed by Kajiwara for Co and its alloys[26].

3. RESULTS AND DISCUSSION

Figure 1(a) shows the martensite plates formed by simple cooling in the thermomechanically-treated specimen. The arrows show the width of martensite plate, and the maximum width is about 1.5 μm and that of other plates are less than 0.25 μm . Many white striations seen in the martensite plates are considered to be the contrasts due to stacking faults. Figure 1(b) shows the diffraction pattern taken from the largest martensite plate shown in the right side of Fig. 1(a), but the diffraction condition in (b) is slightly different from that in (a). Only the h.c.p. spots and streaks are observed, and no f.c.c. spots are observed. This fact indicates that there exist no such lamella structures having a mixture of h.c.p. and f.c.c. phase as observed in the deformation-induced transformation[25]. Figure 2 shows the martensite plate formed by simple cooling in the non-thermomechanically-treated specimen. The width of martensite plate is more than 5 μm and is larger than that of thermomechanically-treated specimen. Shockley partial dislocations and lots of striations due to the stacking fault are observed in the plate. The diffraction pattern taken from this plate is basically the same as in Fig. 1(b).

There is a tendency that the martensite plate with a smaller width is formed in the thermomechanically-treated specimens than in the non-thermomechanically-treated specimens. This is because there are many stacking faults which act as nucleation sites for ϵ martensite in the former case. The width of martensite plate formed by simple cooling, however, is not so small as those observed in the case of deformation-induced transformation.

The density of stacking faults was estimated by analysis of diffraction patterns such as shown in Fig. 1(b). Figure 3 shows the fault parameter α in thermomechanically-treated and non-thermomechanically-treated specimens. The data for the specimens in which the $\gamma \leftrightarrow \epsilon$ cyclic transformation was repeated twenty times in the thermomechanically or non-thermomechanically-treated specimens are also shown in the figure. There is a wide range of scatter in the value of α for each specimen, but these observed α values in martensite formed by simple cooling is in almost the same range as those of deformation-induced martensite reported by Kikuchi et al [24]. Therefore it is considered that the $\gamma \rightarrow \epsilon$ transformation mechanism itself is the same in both the specimens. The stacking fault parameter α decreases with increasing number of $\gamma \leftrightarrow \epsilon$ cyclic transformation both in the thermomechanically and non-thermomechanically-treated specimens, but the reason for such decrease is not clear at this moment.

As indicated from the diffraction pattern of Fig. 1(b), lamella structures with a mixture of h.c.p. and f.c.c. phase do not exist in the case of simple cooling, which is quite different from that of deformation-induced transformation. However, other type of peculiar structures are observed in the case of simple cooling. Figure 4(a) shows the a part of a martensite plate with about 1 μm width formed in the thermomechanically-treated specimen and (b) is the diffraction pattern from (a). A pair of two extra spots are observed as shown by arrows in (b). These extra spots are considered to have resulted from thin plates indicated by arrows in (a). These spots indicate the existence of some kinds of long period stacking structures. This type of structure was observed only in the thermomechanically-treated specimens. Another type of structure, which is considered to be double hexagonal, was observed in some of non-thermomechanically-treated specimens.

In conclusion, very fine martensite plates such as observed in the case of stress-induced transformation are not present in the case of thermally-induced transformation. The reason for this may be as follows. In the case of the deformation-induced transformation, stress will supply a larger driving force for the $\gamma \rightarrow \epsilon$ nucleation process than in the case of simple cooling, and many ϵ martensites are easily stress-induced at the stacking faults in the very early stage of transformation. On the other hand, much smaller number of

Fig. 1 (a) Electron micrograph of martensite plates formed by simple cooling in thermomechanically-treated specimen, (b) diffraction pattern taken from the martensite plate on the right of (a).

Fig. 2 Electron micrograph of martensite plates formed by simple cooling in non-thermomechanically-treated specimen.

Fig. 3 Fault parameter of martensite plates formed in thermomechanically and non-thermomechanically-treated specimens. The data for the specimens in which the $\gamma \leftrightarrow \epsilon$ cyclic transformation was repeated twenty times in these specimens are also shown.

Fig. 4 (a) Electron micrograph of a martensite plate formed by simple cooling in thermomechanically-treated specimen showing thin plates with other structures within one martensite plate, (b) diffraction pattern taken from area in (a).

martensite plates are formed by simple cooling in the very early stage of transformation and the subsequent growth is easier than the nucleation of another martensite plate, and consequently, martensite plates grow into much thicker plates during cooling. As a result, the nanometric lamella structures with the mixture of f.c.c. and h.c.p. phase are not formed in the case of thermally-induced transformation.

REFERENCES

- [1] Sato A, Chishima E, Soma K. and Mori T., *Acta Metall.*, **30** (1982) 1177.
- [2] Sato A, Chishima E, Yamaji Y. and Mori T., *Acta Metall.*, **32** (1984) 539.
- [3] Sato A, Yamaji Y. and Mori T., *Acta Metall.*, **34** (1986) 287.
- [4] Murakami M, Otsuka H, Suzuki H. G. and Matsuda S., Proc. Int. Conf. Martensitic Transformation, Nara, Japan, 1986 (The Japan Institute of Metals, 1987) p. 985.
- [5] Sato A. and Mori T., *Mater. Sci. Eng.*, **A 146** (1991) 197.
- [6] Hoshino Y, Nakamura S, Ishikawa N, Yamaji Y, Matsumoto S, Tanaka Y. and Sato A., *Mater. Trans. JIM*, **33** (1992), 253.
- [7] Tsuzaki K, Ikegami M, Tomota Y, Kurokawa Y, Nakagawara W. and Maki T., *Mater. Trans. JIM*, **33** (1992) 263.
- [8] Tomota Y, Nakagawara W, Tsuzaki K. and Maki T., *Scripta Metall.*, **27** (1992).
- [9] Otsuka H, Murakami M. and Matsuda S., Proc. MRS Int. Meeting on Advanced Materials, Tokyo, 1988 (Materials Research Society, 1989, vol. 9) p. 451.
- [10] Otsuka H, Yamada H, Tanahashi H. and Maruyama T., Proc. Int. Conf. Martensitic Transformation, Sydney, 1989 (Materials Science Forum vol. 56-58 (1990), Pt. II), p. 655.
- [11] Otsuka H, Yamada H, Maruyama T, Tanahashi H, Matsuda S. and Murakami M., *ISIJ International*, **30** (1990) 674.
- [12] Moriya Y, Kimura H, Ishizaki S, Hashizume S, Suzuki S, Suzuki H. and Sampei T., *J. Physique IV* (1991), C4-433.
- [13] Ohtsuka H, Kajiwarra S. and Ishihara T., Proc. Int. Conf. Martensitic Transformations, Monterey, California, 1992, (Monterey Institute for Advanced Studies, 1993), p. 1169.
- [14] Fedrzon L, Gex D, Gu Q, Guenin G, Labrosse D, Mantel M. and Van Humbeeck J., *ibid.*, p. 1175.
- [15] Yang J. H. and Wayman C. M., *ibid.*, p. 1193.
- [16] Inagaki H., *Z. Metallkd.*, **83** (1992) 90, 97, 304.
- [17] Maki T, Kobayashi K., and Tamura I., Proc. Int. Conf. on Martensitic Transformations, Leuven, Belgium, 1982, *J. Physique C4*, 43 (1982), p. 541.
- [18] Maki T, Kobayashi K, Minato M. and Tamura I., *Scripta Metall.*, **18** (1984) 1105.
- [19] Maki T, Furutani S. and Tamura I., *ISIJ International* **29** (1989) 438.
- [20] Kikuchi T. and Kajiwarra S., *Mater. Trans. JIM*, **34** (1993) 907.
- [21] Kajiwarra S., *Trans. JIM*, **26** (1985) 595.
- [22] Kajiwarra S. and Kikuchi T., *Acta Metall.*, **38** (1990) 847.
- [23] Kikuchi T, Kajiwarra S. and Tomota Y., *Mater. Trans. JIM*, **36**(1995)719.
- [24] Kikuchi T, Kajiwarra S. and Tomota Y., "Formation process of lamella structures by deformation in an Fe-Mn-Si-Cr-Ni shape memory alloy", in this issue.
- [25] Ogawa K. and Kajiwarra S., *Mater. Trans. JIM.*, **34**(1993)1169.
- [26] Kajiwarra S., *Japan. J. Appl. Phys.*, **9**(1970)385.