

## Effect of heat treatment on martensitic transformation in Fe–12.5%Mn–5.5%Si–9%Cr–3.5%Ni alloy

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**Abstract.** In this study, thermally-induced martensitic transformation ( $g(fcc) \rightarrow e(hcp)$ ) in Fe–12.5%Mn–5.5%Si–9%Cr–3.5%Ni (weight) alloy was studied by scanning electron microscopy (SEM) and transmission electron microscopy (TEM). The effect of cooling rate was investigated. It was observed that fast cooled sample exhibited regular overlapping of stacking faults and  $e$  martensite plates were formed parallel to each other. TEM investigations showed that the orientation relationship between  $g$ – $e$  phases corresponds to Shoji–Nishiyama type orientation relationship.

**Keywords.** Martensitic transformation; Fe–Mn–Si–Cr–Ni; grain size;  $e$  martensite.

### 1. Introduction

It is well known that the martensitic transformation, which occurs in many Fe, Cu and Ti based alloys and ceramics, is one of the most typical first-order structural diffusionless phase transition. It was widely studied to determine its characteristics from physical, metallographical and crystallographical viewpoints (Nishiyama 1978). Some aspects of martensitic transformations, such as transformation temperature, crystallography and the amount of the product martensite and its morphology, are strongly influenced by external fields such as temperature and uniaxial stress (Nishiyama 1978; Kakeshita *et al* 1999). Among the iron based alloys, the most studied ones are Fe–Mn and Fe–Mn–Si alloys which present non-thermoelastic martensitic transformation (Baruj *et al* 2000; Lee *et al* 2003). The microstructure of  $e$  martensite formed by cooling in thermomechanically-treated Fe–Mn–Si–Cr–Ni shape memory alloys has also been investigated (Inagaki 1992; Yang and Wayman 1992; Li *et al* 1999; Baruj *et al* 2004). The effect of Mn and Ni on the shape memory effect in Fe–Mn–Si–Cr–Ni alloys was quantitatively investigated (Inagaki 1995; Jiang *et al* 1995). It is known that the  $g \rightarrow e$  transformation occurs by the motion of stacking faults on alternate close packed planes of the  $fcc$  structure. All  $\{111\}$  planes of the  $fcc$  structure are possible shear planes. It is well known that the crystallographic relationships between  $fcc$  and  $hcp$  structures are:  $\{111\}_g // \{0001\}_e$  and  $\langle 1\bar{1}0 \rangle_g // \langle 11\bar{2}0 \rangle_e$  (Bergeon *et al* 1998).

In the present work, the effect of cooling rate on the thermally-induced martensitic transformation in Fe–12.5%Mn–5.5%Si–9%Cr–3.5%Ni alloy is investigated by SEM and TEM.

### 2. Experimental

Fe–12.53%Mn–5.35%Si–8.95%Cr–3.38%Ni alloy was prepared by induction melting under an argon atmosphere, using high purity iron, manganese, silicon, chromium and nickel. The chemical composition of the alloy was determined by EDS. The arc-melted ingots were cut by a diamond cutter at room temperature. Samples obtained from the buttons were sealed in quartz capsules. The sealed specimens were homogenized at 1050°C for 1.5 h and after homogenization one of the samples was furnace cooled and another one was quenched in water at room temperature. The SEM specimens were prepared by conventional mechanical polishing followed by etching with acetic glyceric acid (1 ml HCl + 10 ml acetic acid + 5 ml HNO<sub>3</sub> + 2 drops of glycerin) (Andrade *et al* 1999). SEM observations were performed in a JEOL 5600 scanning electron microscope.

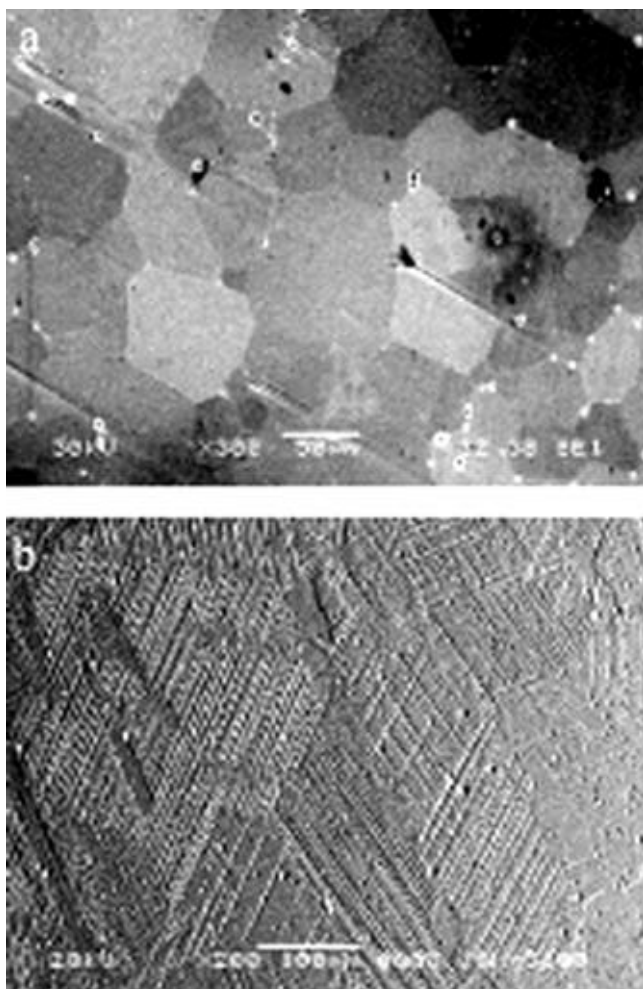
From the quenched sample, several TEM specimens were prepared. Discs of 0.4 mm thickness were spark cut and thinned to 0.2 mm with a 800 grit emery paper. Finally, the discs were electrolytically thinned by the double-jet technique using a solution of 92% acetic acid and 8% perchloric acid at 10°C. The thinning voltage was set at 20 V and the current was 64 mA. The TEM observations were performed by means of a JEOL 3010 electron microscope with an operating voltage of 300 kV with a double tilt specimen.

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### 3. Results and discussion

#### 3.1 Effect of cooling rate on behaviour of martensitic transformation

The effect of cooling rate on the behaviour of martensitic transformation was investigated by comparing the microstructure of the specimens cooled in furnace or quenched in water. Figure 1a shows scanning electron microscopic image of Fe–12.5%Mn–5.5%Si–9%Cr–3.5%Ni alloy which was furnace cooled. As seen in figure 1a, the *g* austenite appears as large domains and the transformation has not begun. The austenite grain size was from 20 to 100  $\mu\text{m}$  in the furnace cooled sample. Figure 1b shows large domains of martensite appearing as a very tight juxtaposition of thin plates in the water quenched specimen. The microstructure of this specimen at room temperature consists of several thermally-induced *e* martensite bands



**Figure 1.** SEM micrographs of specimens: **a.** secondary electron image (SEI) of austenite phase in furnace cooled Fe–12.5%Mn–5.5%Si–9%Cr–3.5%Ni and **b.** back scattering electron image (BEI) of *e* martensite phase which formed in the austenite grains in the water quenched alloy.

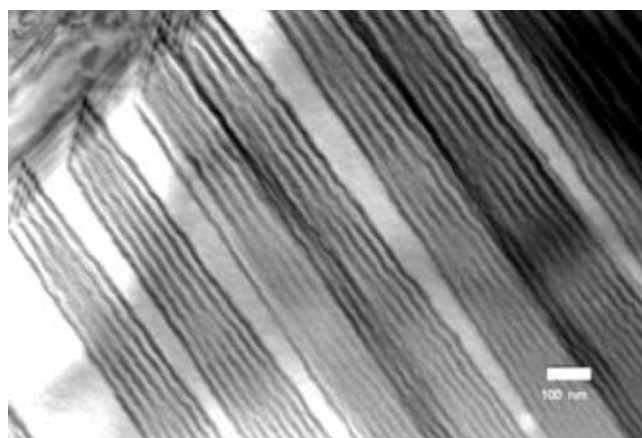
parallel to each other and untransformed *g* phase. Most of the bands of *e* martensite pass through the whole grain and for smaller grains, usually different directions of *e* martensite bands are formed in a grain (Jiang *et al* 1995).

#### 3.2 Transmission electron microscopy observations

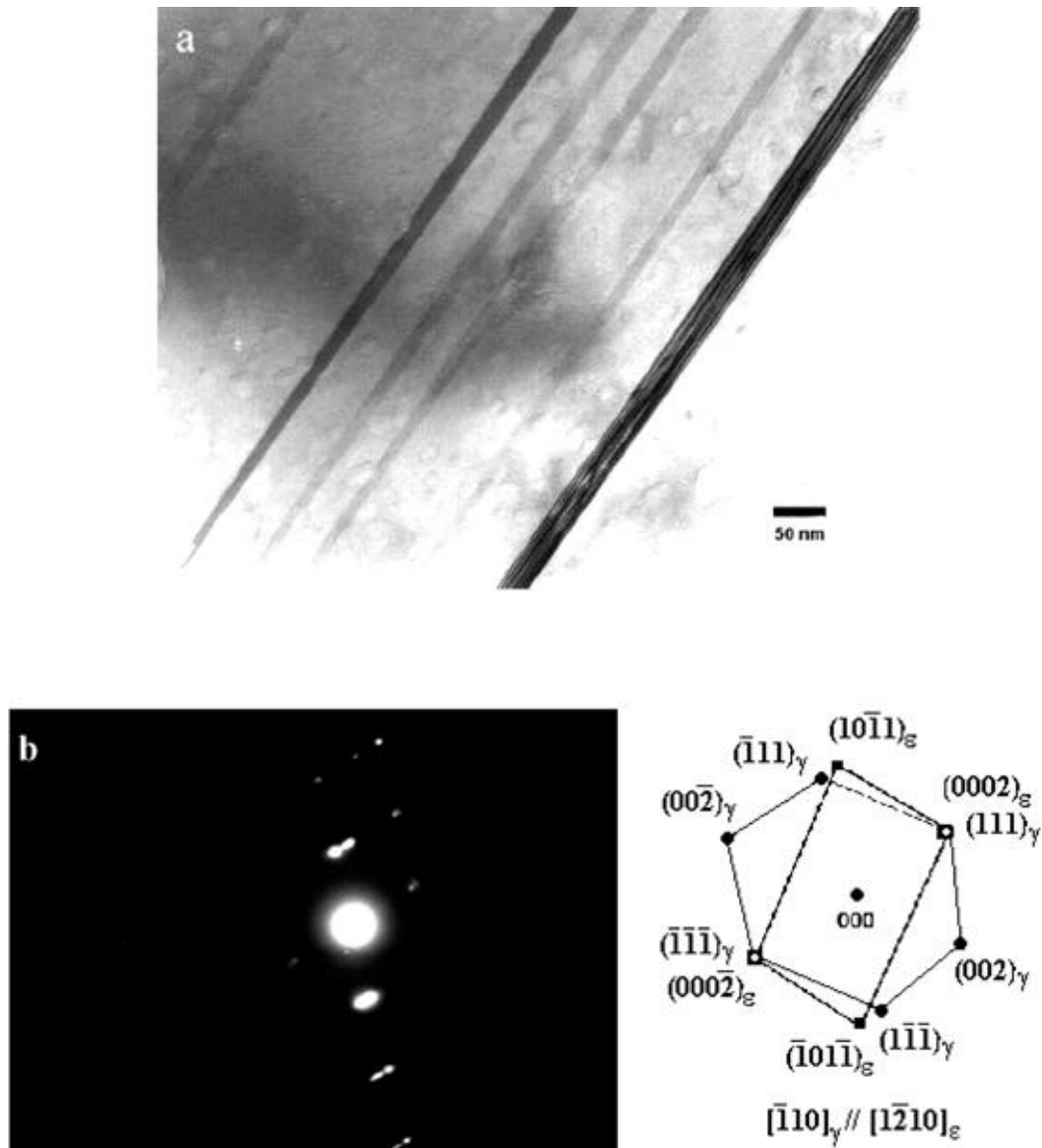
The TEM observations of water quenched sample revealed that the martensite plates generally formed in whole austenite grain. Within each *g* grain, a large number of widely extended overlapped stacking faults and *e* martensite bands intersecting frequently with each other were observed (Inagaki 1995).

Figure 2 shows the presence of a large number of stacking faults, which mainly run in the stacking direction. The stacking faults were formed in the  $\{111\}_g$  planes, that are usually the known fault planes for this system (Maki and Tsuzaki 1992; Yang and Wayman 1992). The stacking faults end in other defects, such as grain boundaries and dislocations. Based on the analysis of electron diffraction pattern and the morphology of stacking faults, it was found that the stacking faults were oriented in one of the 12  $\{111\} \langle 111 \rangle_g$  systems (Li *et al* 1999). The stacking fault energy of Fe–Mn–Si based shape memory alloys is very low, and perfect dislocations can easily split into two  $1/6 \langle 110 \rangle$  partial edge dislocations, which form stacking faults on the  $(111)_g$  planes along the  $\langle 110 \rangle_g$  directions (Yang and Wayman 1992).

Figure 3a shows the *e* martensite plates in the water quenched sample. These martensite bands are on the  $\{111\}_g$  planes in austenite phase where stacking faults have a  $\{0001\}_e$  fault plane. Figure 3b gives the diffraction pattern of the *e* plates and key diagram of this pattern. The zone axes for austenite and martensite phases were determined as  $[\bar{1}10]_g$  and  $[1\bar{2}10]_e$ , respectively and the



**Figure 2.** Typical bright field electron micrograph of Fe–12.5%Mn–5.5%Si–9%Cr–3.5%Ni alloy showing widely extended stacking faults.



**Figure 3.** a. TEM micrograph showing the formation of thermally induced *e* bands and b. diffraction pattern and indices diagram.

orientation relationship was determined as  $(111)_g // (0001)_e$ ,  $[\bar{1}10]_g // [\bar{1}2\bar{1}0]_e$  which corresponds to the Shoji–Nishiyama relationship (Nishiyama 1978; Yang and Wayman 1992).

#### 4. Conclusions

In this paper, an analysis of the morphology of the *e* martensite formed in Fe-12.5%Mn-5.5%Si-9%Cr-3.5%Ni shape memory alloy was carried out by using SEM and TEM. As a consequence of furnace-cooling (slow cooling), grains of the austenite phase with a grain size between 20 and 100  $\mu\text{m}$  were observed. However, *e* martensite was observed in the austenite grain as a result of

water quenching. From the TEM observations and crystallographic investigations of this shape memory alloy, the formation of *e* martensite crystals obeyed a Shoji–Nishiyama type relationship.

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