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## Optical Microscope Study of the $\gamma(\text{FCC}) \leftrightarrow \varepsilon(\text{HC})$ Martensitic Transformation of a Fe-16%Mn-9%Cr-5%Si-4%Ni Shape Memory Alloy

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**Abstract.** The  $\gamma(\text{FCC}) \leftrightarrow \varepsilon(\text{HC})$  transformation is studied by light optical microscopy and scanning electron microscopy in a polycrystalline Fe-Mn-Si-Cr-Ni shape memory alloy. Thermal and stress-induced martensites are both studied to point out differences. A color etching method permitted to clearly observe morphological evolutions during the transformation and its reversion. Deformations of a golden microgrid deposited on austenitic samples are observed by SEM during the transformation. This technic has led to point out microscopic differences concerning the two kinds of martensite. SEM results are used to explain light optical microscopy observations.

### 1. INTRODUCTION

Fe-based shape memory alloys have been largely investigated during the last fifteen years. The shape memory effect observed in these alloys is related with the  $\gamma(\text{FCC}) \leftrightarrow \varepsilon(\text{HC})$  martensitic transformation. The transformation is produced by the introduction of stacking faults every second compact plane of austenite. The faults are created by the motion of  $a_\gamma \langle 112 \rangle$  type Shockley partial dislocations. The relationships between the two phases are clearly known:

$$\begin{array}{l} \{111\}_\gamma // \{0001\}_\varepsilon \\ \langle 110 \rangle_\gamma // \langle 1120 \rangle_\varepsilon \end{array}$$

Four compact planes exist in the FCC structure thus defining four shear bands where  $\varepsilon$  plates can be created. Three directions of Shockley dislocations exist in each band thus defining three  $\varepsilon$  variants per band.

The transformation can be activated by simple cooling or by stress. These two kinds of transformations are investigated in a Fe-Mn-Si-Cr-Ni polycrystal using light optical microscopy and scanning electron microscopy. The aim of this study is to observe microstructural differences between thermal and stress-induced martensites.

### 2. EXPERIMENTS

#### 2.1. Samples preparation

The alloy is made by Ugine-Savoie with a composition of Fe-16Mn-5Si-9Cr-4Ni (weight %). It was initially cold-rolled and the thermomechanical treatment proposed by Federzoni and al. [1] has been used to leave the alloy in a microstructural state favourable to thermal  $\varepsilon$  martensite production. This treatment is : 850°C annealing, 30 mn + 7% deformation + 700°C, 30 mn annealing. Austenitic samples were then mechanically and electrolytically polished.

The thermal martensite ( $\varepsilon_{\text{th}}$ ) is produced by simple cooling and the stress-induced martensite ( $\varepsilon_{\text{st}}$ ) by tensile elongation. The samples are then cut into two pieces : one piece is studied in this state, the other

one is heated up to 200°C before study. The martensite fractions are measured by thermoelectric power [1].

Sample	Treatment	$\epsilon_{th}$ (%)	$\epsilon_{st}$ (%)	Treatment	$\epsilon_{th}$ (%)	$\epsilon_{st}$ (%)
T1	-35°C, 5 mn	24	0	---	---	---
T1R	-35°C, 5 mn	24	0	200°C, 15 mn	2	0
T2	-196°C, 5 mn	66	0	---	---	---
T2R	-196°C, 5 mn	66	0	200°C, 15 mn	3	0
D1	deformation 3%	0	26	---	---	---
D1R	deformation 3%	0	26	200°C, 15 mn	0	6
D2	deformation 7%	0	~50	---	---	---
D2R	deformation 7%	0	~50	200°C, 15 mn	0	~45

## 2.2. Light optical microscopy

A color etching method developed by Qin and al. [2] is used to reveal  $\epsilon$  and  $\alpha'$  martensites. The phases are clearly distinguished by different colors. The composition of the color etching solution is 1,2%  $K_2S_2O_5$  + 0,5%  $NH_4HF_2$  in distilled water.  $\gamma$  matrix appears as brown ;  $\epsilon$  martensite appears as clear except thin plates which appear as dark lines ;  $\alpha'$  martensite appears as blue dark particles.

## 2.3. Scanning electron microscopy

In-situ tensile experiments have been realised to observe the microstructure evolution during stress-induced transformation. A technique developed by Thollet and al. [3] has been used to depose a square golden grid onto the material surface before martensitic transformation. The grid has a 2  $\mu m$  step and a 0,2  $\mu m$  linewidth. The thermal transformation has also been observed by this method.

## 3. RESULTS

### 3.1. Thermal martensite

#### 3.1.1. Light optical microscopy

From the beginning of its formation, the  $\epsilon$  martensite appears as large domains (fig. 1a). Increasing martensite rate induces an increase of the number of grains containing martensite (fig. 1b). Only one or two band orientations are activated in a grain. The  $\alpha'$  martensite is not observed even for high  $\epsilon$  fractions.

Reversion is almost complete after heating up to 200°C.

#### 3.1.2. SEM

Figure 2 shows that large domains of martensite appears as a very tight juxtaposition of thin plates. These domains do not create any global deviation of grid lines but some local alternate shears are present which are created by each thin plate. All these shears are accommodating to cancel the global deformation. This is coherent with Yang and al. TEM observations [4].

### 3.2. Stress-induced martensite

#### 3.2.1. Light optical microscopy

As shown in figure 3a, the first step of stress-induced transformation is characterised by the formation of thin  $\epsilon$  plates randomly distributed through the material. Almost no large domain is observed. Only one or two band orientations are generally activated in a grain. Thus, the surface relief is composed of many

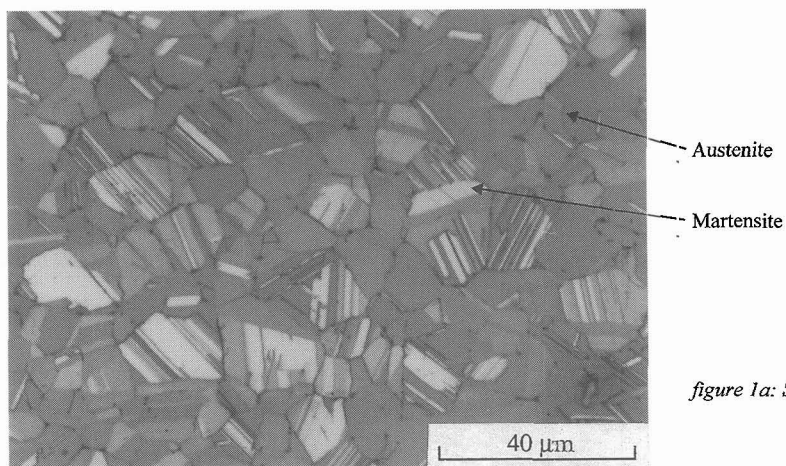


figure 1a: Sample T1 (24%  $\epsilon_{th}$ )

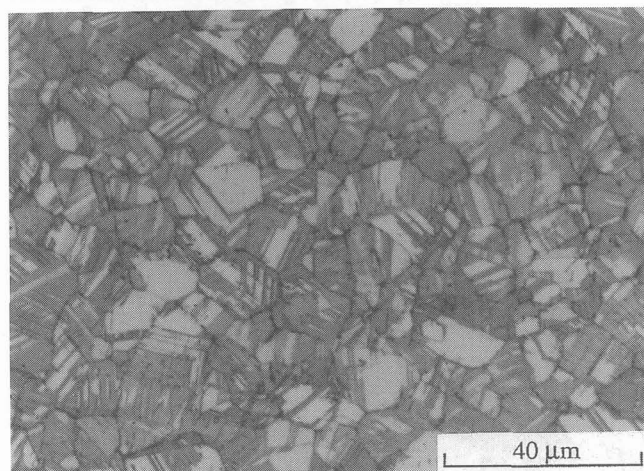


figure 1b: Sample T2 (66%  $\epsilon_{th}$ )

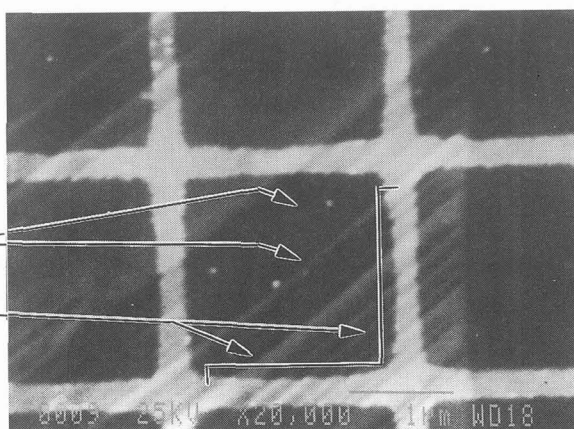
figure 1: Microstructure of T1 and T2 after color etching showing  $\gamma$  matrix (grey) and  $\epsilon$  martensite (white).

figure 2:  
SEM observations  
of a sample containing  $\sim 70\%$   $\epsilon_{th}$

Microgrid is deposited before  
the transformation.

Local deviations created by thin plates

Thin  $\epsilon$  plates



parallel scratches. After heating up to 200°C, thin plates disappear almost completely but figure 3b shows that some slight surface relief remains.

Increasing stress induces the appearance of large  $\epsilon$  domains (fig.4). These large stress-induced  $\epsilon$  domains are quite different from large thermal  $\epsilon$  domains : they rarely invade all the grain, the austenite/martensite interfaces are not well defined from a geometric point of view and the martensite plates often do not reach the grain boundaries. There is a lot of  $\alpha'$  martensite which appears as little particles in thin plates tangles or as long lamellas in large domains. The reversion is low at 200°C but the number of large domains clearly decreases. At the same time, the appearance of zones of thin parallel  $\epsilon$  plates containing no  $\alpha'$  martensite is to be noticed. This morphology may result from the incomplete reversion of large  $\epsilon$  domains.

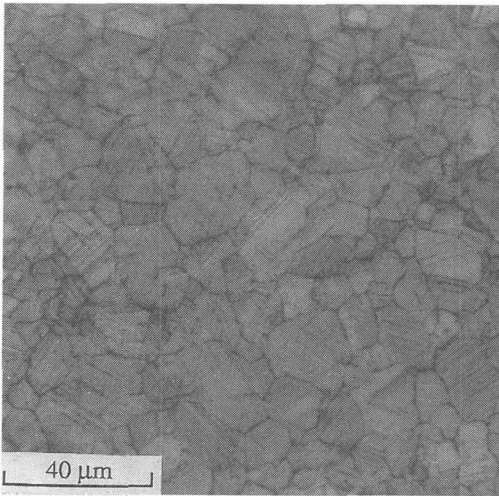


figure 3a: Sample D1

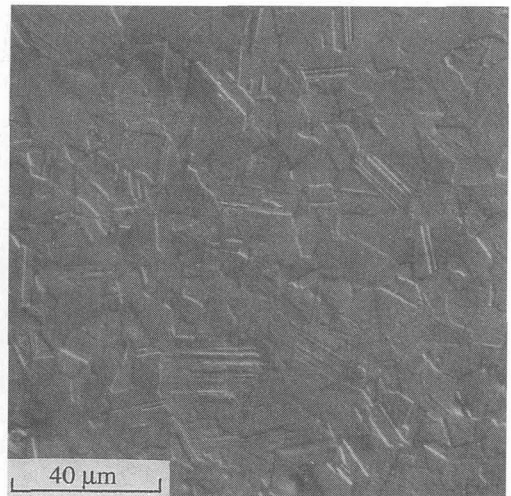


figure 3b: Sample DIR (relief observation)

figure 3: Microstructure of D1 and DIR after color etching showing  $\gamma$  matrix (grey) and thin  $\epsilon$  plates (dark lines)  
Dark lines associated to martensite plates have disappeared in DIR but relief lines remain.

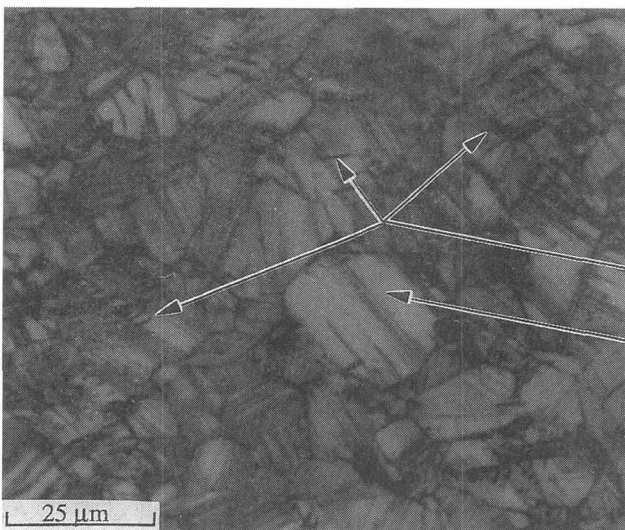


figure 4: Microstructure of D2 after color etching showing  $\gamma$  matrix (grey),  $\epsilon$  plates (white or dark lines) and  $\alpha'$  particles (dark).

$\alpha'$  martensite

$\epsilon$  martensite as large domains

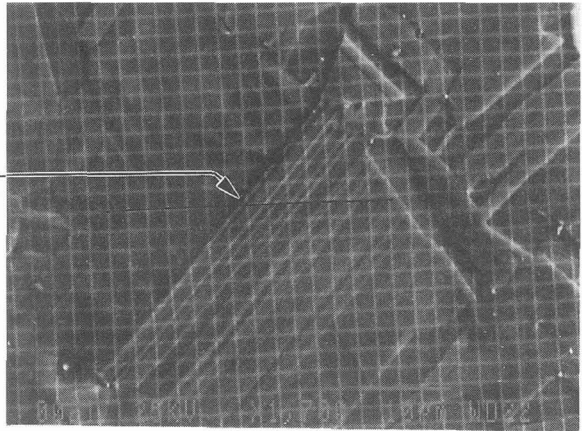
### 3.2.2. SEM

The large  $\epsilon$  domains previously observed appear as a tight juxtaposition of thin plates (fig.5). A global deviation of grid lines crossing these domains is clearly observed. Each thin plate creates a little local shear and the superposition of all these shears induces the global deviation observed.

Thin parallel  $\epsilon$  plates outside large domains create local deviations of grid lines.

*figure 5:*  
SEM observations of a 8% deformed sample

A global deviation of linegrid crossing martensite is observed.



## 4. DISCUSSION

### 4.1. $\epsilon$ martensite formation

Many macroscopic morphological differences between thermal and strain-induced  $\epsilon$  martensites have been noticed. SEM observations point out that these differences are associated with different dislocations stackings. The strain accommodation is perfect in thermal martensite on a scale of the order of 150 nm. On the same scale, there is an accumulation of little strains in stress-induced martensite. These large net deformations induce stress concentrations at plates ends and a high energy state which limits plates growth. This fact explains that large domains are not spontaneously created under stress and are not observed for low martensite fractions.

Formation kinetics are also different. Thermal martensite immediately appears as large domains which do not develop more ; stress-induced martensite grows band by band and large domains seem to result from extension of bands initially present as thin plates.

All these phenomena suggest different microscopic formation process.

### 4.2. $\alpha'$ martensite formation

$\alpha'$  (CC) formation is caused by a double shear mechanism [5,6].  $\gamma \rightarrow \epsilon \rightarrow \alpha'$  and  $\gamma \rightarrow \alpha'$  transformations can exist but, in the present case,  $\alpha'$  formation seems to result from  $\gamma \rightarrow \epsilon \rightarrow \alpha'$  transformation. Indeed,  $\alpha'$  martensite is observed in large  $\epsilon$  domains or at intersections between several  $\epsilon$  bands.

The  $\alpha'$  formation at the intersections of two bands is a well-known phenomena [5,6]. Coalescence between  $\alpha'$  crystallites creates quite long  $\alpha'$  particles.

$\alpha'$  is also observed inside large domains where only one band orientation is activated. Fujita and al.[6] have developed a model to explain this phenomena. Large domains are formed by a tight

juxtaposition of thin plates. Front partial dislocations make a stair-rod type cross-slip and hit a neighboring  $\epsilon$  crystal to form  $\alpha'$  crystallites. The propagation of this mechanism associated to coalescence induces creation of long  $\alpha'$  lamellas. The observation of long parallel lamellas crossing large  $\epsilon$  domains supports this explanation.

### 4.3. $\epsilon$ martensite reversion

The  $\alpha'$  martensite has a strong effect upon the  $\epsilon$  martensite reversion.

In the case of thermal martensite, reversion is complete at 200°C.

In the case of low fractions of stress-induced martensite, no  $\alpha'$  martensite is observed. The nearly complete reversion leaves only a slight surface relief. That means that the reversion does not always correspond to a simple come-back of partial dislocations : the microstructure memory is partial [7].

Increasing stress induces  $\alpha'$  formation and stress-induced  $\epsilon$  reversion strongly decreases. The  $\epsilon$  plates are pinned. However, the reversion of large  $\epsilon$  domains containing no  $\alpha'$  martensite is observed ; it leads to large austenitic domains containing only few thin  $\epsilon$  plates. This last case corresponds to a good reversion and microstructure memory. It suggests a forward reversion of plates by partial dislocations come-back. This can be explained by the model of  $\epsilon$  stress-induced martensite formation developed by Guénin [8].

## 5. CONCLUSION

This study pointed out many differences between thermal and stress-induced martensites. They are clearly related to the microscopic net deformations associated to the transformation.

In thermal martensite, self-accommodation allows the creation of large  $\epsilon$  domains and reduces stress concentrations. Thus, no  $\alpha'$  martensite is created and reversion is good (no martensite plates and no surface relief remain).

In stress-induced martensite, there is a large shape change. Thus,  $\epsilon$  martensite is rather created as thin plates to limit stress concentrations at plate ends. When the stress (strain) is low, the reversion of these thin plates is good. But when the martensite rate increases, large domains appear composed of many thin plates. In this case, the large stress concentrations favour the creation of  $\alpha'$  martensite which in turn reduces drastically the reversion.

### Acknowledgements

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