

# Microstructure observation in Fe-Mn-Si-Cr-Ni-(Co) type stainless alloys with shape memory effect using scanning electron microscope (SEM)

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## 1. Introduction

The Shape Memory Effect (SME) is the capacity that some materials have to recuperate your shape or original state during the heating after plastic deformation. These effect are associated with the non thermoelastic  $\gamma(\text{fcc})$  to  $\epsilon(\text{hcp})$  martensitic transformation<sup>[1-3]</sup>. Near the  $M_s$  (martensitic transformation starting temperature) the martensite could also be stress induced.

The study of Fe-Mn-Si-Cr-Ni-(Co) type stainless alloys with (SME) started at Unicamp in 1994. These alloys present a good shape memory performance, high mechanical strength, and with low stacking fault energy of the austenitic phase<sup>[4]</sup>.

A lot of techniques are used for microstructural observation at different scales of the stress induced  $\epsilon$  martensite<sup>[2,5-6]</sup>. That's because the microstructure, thermomechanical treatment, and chemical composition have a strong influence on the shape memory effect.

In this work we studied the influence of different austenitic grain sizes on the volumetric fraction of phases presents after several compression cycles and shape recovery.

## 2. Experimental procedures

The material used had the following composition : Fe ( balance ) / 0.044%C / 7.81%Mn / 5.16Si / 13.03%Cr / 5.74%Ni and 11,85%Co (in wt. % ). The ingot produced was cold rolled with 40% of area reduction , and then cut in various parts. Each part was heat treated at 1323K for : 10 minutes, 1, 5, 8 and 16 hours, to obtain different austenitic grain sizes. The thermomechanical treatment was composed by six cycles. Each cycle correspond to the deformation at 4% compression and heating at 873K for 30 minutes for shape recovery.

The microstructure was observed after the last cycle ( non-recuperated, only deformed ) with Light Optical Microscope and Scanning Electron Microscope (SEM). The surface was electropolished and etched with 2ml HCl + 2ml HNO<sub>3</sub> to observe the lamellar structure.

## 3. Results and discussion

### a)- Heat treatment

The heating at 1323K for 10 minutes, 1, 5, 8 and 16 hours resulted in different austenitic grain sizes: 35, 48, 65, 71 and 88 $\mu\text{m}$ . This variation on the grain size with the heating can be seen in Figure 1.

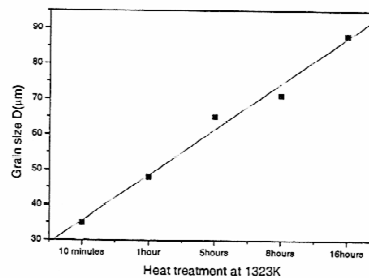


Figure 1. Variation of grain size with the time for heat treatment at 1323K.

### b) X-ray diffraction

The results of X-ray diffraction using  $\text{CuK}\alpha$  radiation confirmed the presence of  $\gamma(\text{fcc})$  and  $\epsilon(\text{hc})$  phases after compression cycles. We observed that samples with small grain size presented a higher volumetric fraction of  $\epsilon$  martensite when compared with large grain size samples.

After the shape recovery at 873K we observed an increasing of the volumetric fraction of  $\gamma$  (fcc) phase . This indicates that the reversion  $\epsilon(\text{hc}) \rightarrow \gamma(\text{fcc})$  occurred during the heating and provoked a change on the microstructure of this alloy that can be observed with light optical microscope and SEM.

### c) Microstructure

The microstructure observation in this alloy using light optical microscope and SEM, showed that the austenitic grain size can affect the volumetric fraction of stress induced  $\epsilon$  martensite.

In all the conditions it was observed a lamellar structure with a mixture of hcp and fcc phases (Figures 2 and 3).

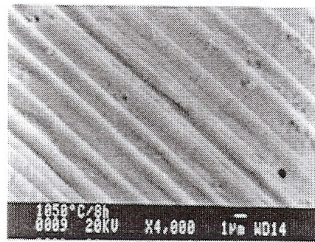


Figure 2. SEM- Micrography of the sample heat treated at 1323K for 8 hours after the last cycle of compression.

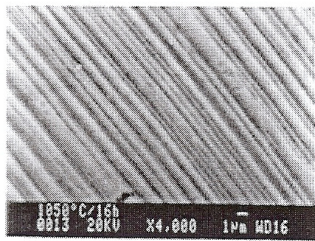


Figure 3. SEM- Micrography of the sample heat treated at 1323K for 16 hours after the last cycle of compression.

The influence of austenitic grain size in the morphology of the stress-induced  $\epsilon$  martensite was remarkable. We observed with light optical microscope that the samples with smaller grain size presented the  $\epsilon$  martensite lamellas oriented in only one direction inside the grain. But, when the grain size was large we observed several orientations inside the grains. This shows that samples with smaller grain sizes present better accommodation for  $\epsilon$  martensite lamellas.

The SEM confirmed the presence of lamellar structure of stress induced  $\epsilon$  martensite in all conditions. The  $\epsilon$  martensite lamellas presented a width of some microns.

Using SEM associated with other techniques we want to continue the study of the influence of austenitic grain size in the:

- shape memory performance;
- formation of the  $\alpha'$ - martensite (that is formed preferentially on the  $\epsilon$  martensite plates)<sup>[2,6-8]</sup>;
- the influence of the  $\alpha'$ - martensite on the shape memory performance, that according to some authors promote a blocking effect on the reversion of  $\epsilon$  martensite<sup>[6-7]</sup>.

### 3. Conclusions

The results of light optical microscope showed that the austenitic grain size influences the volumetric fraction of stress induced  $\epsilon$  martensite.

Samples with small grain size present only one direction of martensite lamella inside the grains. For large grain size, many directions of these lamellas inside the grains were observed.

Using SEM we observed that in all conditions a lamellar structure was obtained with lamellas width of some microns.

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