Effect of training and the reduction of the austenitic grain size on the morphology of the stress-induced ε-martensite in stainless SMAs

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Abstract. *The shape memory effect in stainless shape memory alloys is a physical phenomenon related to the fcc(γ)↔hcp(ε) transformation and reversion. The morphology of the martensitic phase is a function of such factors as chemical composition, number of training cycles and microstructure refinement; these are very important because they influence strongly the shape recovery properties. In this work, the martensitic phase was induced in a stainless shape memory alloy by applying training cycles (deformation by compression followed by heating) to samples with average austenitic grain sizes varying between 75 and 129μm. It will be shown using optical microscopy that the martensitic morphology is a function of the grain refinement and of the training. Width of the martensite plates and their distribution within the prior austenite grains will be correlated with the data already gotten by the shape memory group (DEMA/FEM/UNICAMP) and presented in international literature.*

Keywords. *shape memory, stainless steel, microstructure refinement*

1. Introduction

The shape memory alloys (SMA) present the capacity to recover its original form, after plastic deformation, by heating above A_F (final temperature of martensite in austenite transformation) (Funakubo, 1987).

The shape memory effect (SME) in the Fe-Mn-Si alloys is related to the transformation of the austenite (γ), of the face centered cubic structure, in martensite (ε), of the hexagonal compact structure, and the posterior reversion of this process. The ε (hcp) → γ (fcc) reversion occurs by heating and results in recovering the memorized shape (Nascimento, 2002a; Otubo, 1996; Jost, 1999; Liu, 2000).

The identification of the phases on structure is the first step for a study more complete of the γ→ε transformation on SMAs. The analysis of martensite morphology depends on factors as chemical composition, number of training cycles and microstructural refinement is very important because they influence strongly the shape recovery properties (Nascimento, 2002a; Meng, 2001). Further, with the analysis of different phases, others factors can be explored, such as: mechanical properties and SME.

The present work, gives continuity to the studies initiated in 1998 with the Fe-Mn-Si-Cr-Ni-Co alloy with SME (Nascimento, 2002a-b), and its main objectives are quantify the phases for each grain size (GS) and training cycle by development of a specific etchant for the used alloy and for each condition. After that the hardness of ε-martensite, for each condition, will be determined by nanoindentation technique (Bueno, 2002).

2. Experimental details

2.1. Alloy and samples

The investigated alloy was melted in a vacuum induction furnace. The chemical composition of the alloy is showed in Tab. (1). At previous work of F. C. Nascimento (Nascimento, 2002a), this alloy passed for a termomechanical treatment (reduction of area around 40% and annealing to 1050ºC in different times) resulting samples with grain size (GS) values differentiated. Afterwards, the samples were submitted to the training cycles.

Table 1 – Chemical composition (wt %)

C	Si	Mn	P	S	Co	Cr	Ni	Mo	Cu	O$_2$*	N$_2$*
0,009	5,25	8,26	0,002	0,006	11,84	12,81	5,81	0,001	0,16	50	52

* percentage in pfm

Each complete cycle corresponds to 4% of deformation by compression followed by heating at 600ºC for 30 minutes, and then until room temperature. For some cycles, a half of the specimen was kept in the deformed state and the others were recovered. The samples final characteristics are showed in Tab. (2).

Table 2 – Samples characteristics

Grain Size (µm)	Annealing 1050ºC (min.)	Number of training cycles	Status
75 +/-13	10	2	DEFORMED
		4	
		5	
106 +/-17	60	4	
		5	
129 +/-18	480	2	

2.2. Metallographic preparation

This work had beginning with the metallographic preparation of the samples by processes of mechanical polishing and sanding. After that they were tested to optimize the specific reagent $K_2S_2O_5 + NH_4HF_2$. According Bergeon et all (Bergeon, 1997), this reagent reveals the martensitic phase with a specific color (color etching).

The metallographic preparation is an essential stage in this work and it is being accomplished based in the literature (Jang, 1995; Bergeon, 1997, 1998a-b; Beraha, 1977; Vander Voort, 1984, 1985) and in studies performed by F. C. Nascimento (Nascimento, 2002a-b) considering the specific parameters of the alloy, such as: chemical composition, grain size, number of training cycles' and volumetric fraction of the phases.

The tests with the reagent $K_2S_2O_5 + NH_4HF_2$ were performed varying and time of attack and the concentration in distilled water.

2.3. Optical analysis of microstructure

The images were acquired using the optical microscope NEOPHOT. This equipment allows to identify and to quantify the existing phases in the material through distinct colors. Because that, is very important to optimize the color etching.

Optic microscopy uses is fundamental in this analysis, being complementary and decisive in already obtained results, where one of the hypotheses is that the martensite precipitates in the austenitic grain boundary (Nascimento, 2002a).

The obtained results will allow to identify and to quantify the martensitic phase, complementing the results obtained in previous jobs of the group using x-rays.

3. Results and discussion

3.1. General characteristics of ε-martensite

It was possible to obtain the revelation of the grain boundaries and present inclusions in the samples for shorter times of etching. The images show heterogeneity of the grain size and the great quantity of inclusions as it shows the Fig. (1).
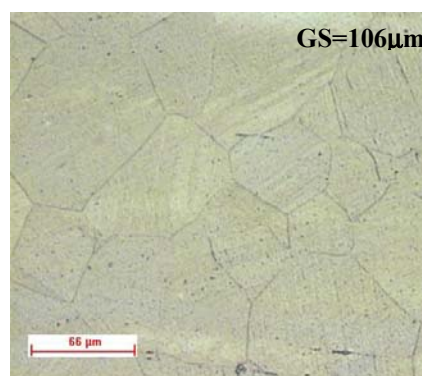

GS=106µm

Figure 1 – Optic microscopy showing the grain size heterogeneous and the inclusions. Etching: 2,0g $K_2S_2O_5$ + 0,5g NH_4HF_2 + 100ml H_2O, t = 5 min.

The great variation in the morphology of the austenitic grain, clearly noticed in the Fig. (1), justifies the standard deviation values in GS's data obtained by F. C. Nascimento (Nascimento, 2002a) and presented in the table 2.

An analysis of the size of the inclusions, accomplished in previous work of the group (Nascimento, 2002a), found values between 2,5 and 5 microns of diameter. This analysis is necessary, because the great quantity of inclusions can change the study of the morphology by optic microscopy.

The martensite plates were distributed inside the austenitic grain, being this characteristic independent of the GS and/or the training. The Fig. (2) presents this characteristic of the morphology of the ε-martensite for different conditions. According to Bergeon et all the dimensions of ε-martensite plates are nanometrics (Bergeon, 1997).
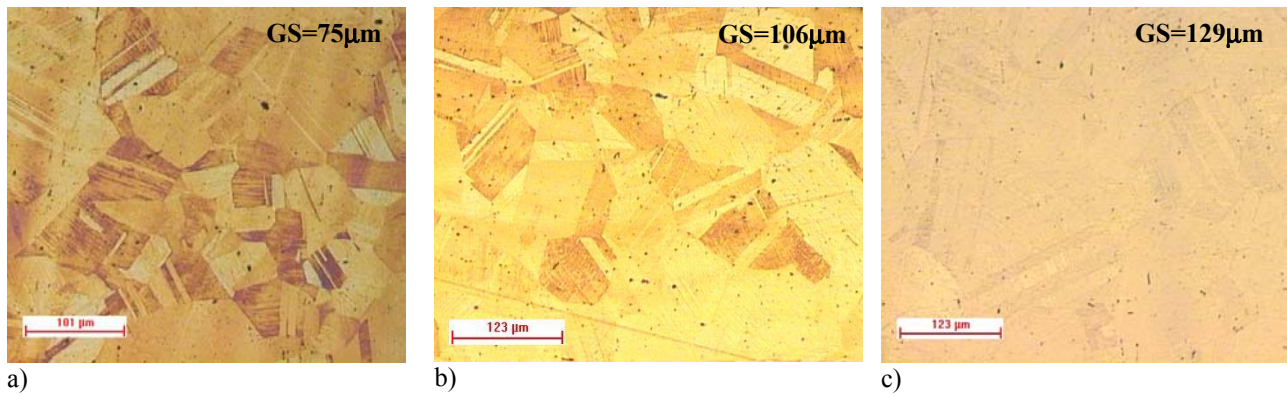

a)   b)   c)

Figure 2 – Optic microscopy showing the morphology of the ε-martensite inside the austenitic grain. a) Etching: 2,0g $K_2S_2O_5$ + 0,5g NH_4HF_2 + 50ml H_2O, t = 18 min; b)Etching:2,0g$K_2S_2O_5$:0,5gNH_4HF_2:50mlH_2O; t = 12 min. c)Etching: 2,0g $K_2S_2O_5$ + 0,5g NH_4HF_2 + 100ml H_2O, t = 8 min;

As shows in Fig. (3), other characteristic of the microstructure of the analyzed samples is the great quantity of twins, characteristic of materials with low stacking fault energy.
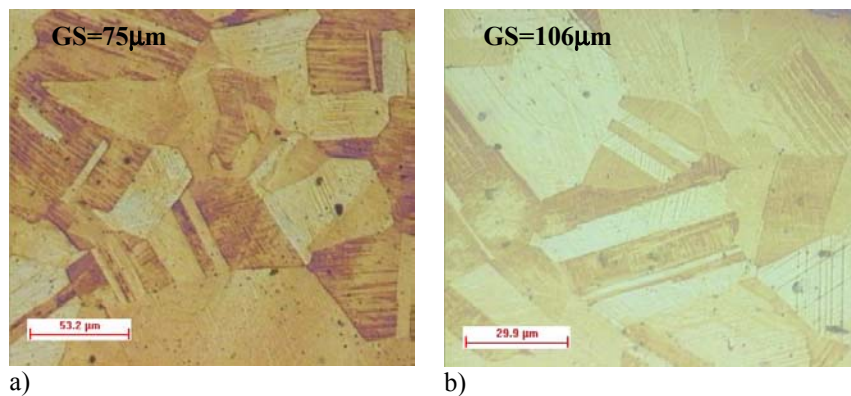

a)   b)

Figure 3 – Optic microscopy showing the twin boundaries. a) Etching: 2,0g $K_2S_2O_5$ + 0,5g NH_4HF_2 + 50ml H_2O, t = 18 min; b) Etching: 2,0g $K_2S_2O_5$ + 0,5g NH_4HF_2 + 50ml H_2O, t = 12 min.

3.2. Characteristics of ε-martensite observed in Fe-Mn-Si-Cr-Ni-Co alloy

Besides the general characteristics previously mentioned, we can also observe in the Fig. (4) accommodation of variants of ε-martensite which occurred with larger frequency in samples with smaller GS.
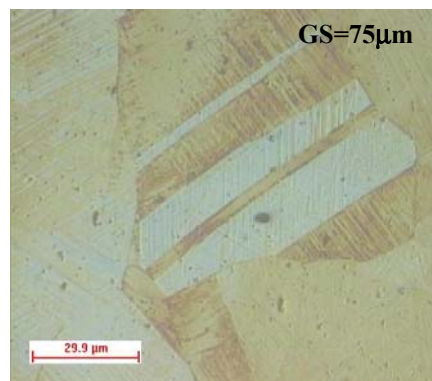

Figure 4 – Optic microscopy showing the orientation change of the ε-martensite; Etching: 2,0g $K_2S_2O_5$ + 0,5g NH_4HF_2 + 50ml H_2O, t = 18 min.

New ε-martensite orientations with crossing of plates occurs in samples with larger GS as showed in Fig. (5). In this regions occurs precipitation of α'-martensite of body centered cubic structure as can be seen in the Fig. (5). This phase was detected, at previous work, by x-rays difraction for samples with larger deformations (Nascimento, 2000).

Already for samples used in this work the quantitative analysis of the α'-phase by x-rays diffraction was not possible due to its volumetric fraction smaller than 5%. The α'-phase is characterized by particle shape or rod shape inside the ε-phase. The length of these short particles is about 9μm, value near to the obtained by J. Otubo (Otubo, 1996) which was about 10μm.

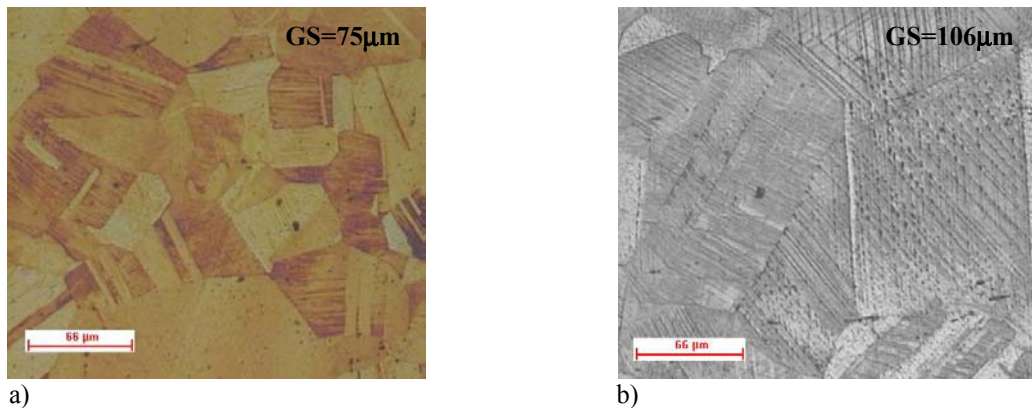a)   b)

Figure 5 – Optic microscopy showing the α'- martensite inside the ε-martensite plates in the samples of larger GS; a) Etching: 2,0g $K_2S_2O_5$ + 0,5g NH_4HF_2 + 50ml H_2O, t = 18 min; b) Etching: 4,0g $K_2S_2O_5$ + 1,0g NH_4HF_2 + 50ml H_2O, t = 6 min.

4. Conclusions

Samples of stainless shape memory alloy with grain size varying from 75 to 129μm were investigated. The main results obtained in this work by metallography and optic microscopy are:

(1) In the samples with larger grain size (129μm), appear α'-martensite that is characterized by rod shape inside the ε-phase with length about 9μm;

(2) Crossing plates and new orientations of ε-martensite occurs more times in samples with larger GS;

(3) The martensite plates were distributed inside the austenitic grain and this characteristic is independent of the microstructure refinement and the training;

(4) For all grain sizes, was observed a great quantity of twins, this is a characteristic of materials with low stacking fault energy.

5. Acknowledgement

6. References

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