EFFECTS OF GRAIN SIZE ON THE SHAPE RECOVERING PROPERTIES OF A STAINLESS SMA

F. C. Nascimento^{1,2}, P. R. Mei¹ and J. Otubo^{1,3}

- 1. DEMA/FEM, State University of Campinas, 13083-970, Campinas, SP, Brazil; e-mail: fabcris@ifi.unicamp.br
- 2. Applied Physics Department, Gleb Wataghin Physics Institute, State University of Campinas, 13083-970, Campinas, SP, Brazil.
- 3. ITA Technological Institute of Aeronautics 2228-900, São José dos Campos, SP, Brazil.

ABSTRACT

This work presents some results about austenite grain size effect on the shape recovery properties of a Fe-Mn-Si-Cr-Ni-Co shape memory alloy (SMA). The small grain size samples presented a higher shape recovering on the first thermomechanical treatment cycle, called training. In each cycle the sample was deformed 4% by compression (to produce ε martensite) and annealed at 873K (to induce shape recovering). The results indicated that the refined microstructure facilitates the forward and backward movement of Shockley partial dislocations. The reverse transformation of ε martensite was complete for the refined microstructure (smaller grain size). On the first cycle training the samples with average austenite grain size 75 and 129 µm presented 32 and 16% of shape recovering. The grain size reduction makes easier the reverse transformation $\gamma(fcc)\leftrightarrow\varepsilon(hcp)$.

KEYWORDS: shape memory effect, austenite grain size, stainless shape memory alloy.

1. INTRODUCTION

Fe-based shape memory alloys (SMA) have been largely investigated during the last years^{[1-3].} The shape memory effect (SME) these alloys is related with the $\gamma(\text{fcc})\leftrightarrow\epsilon(\text{hcp})$ nonthermoelastic martensitic transformation.

The SME result from the reverse motion of Shockley partial dislocation during heating. Several factors can influence on the shape recovery properties, as for example: chemical composition and microstructure (austenite grain size average).

The Fe-Mn-Si-Cr-Ni-Co shape memory alloys are very important because these materials present a good shape recovery properties when compared with others materials.

Our group have studied theses alloy since 1994 and interesting results it was obtained^[4-7]. Recently we verify that the austenite grain size average (GS) influenced strongly the shape recovery properties^[8] being in accordance with hypotheses formulated by others authors^[3, 9-11].

The results indicated that samples with refined structure (small grain size) presented the best shape recovery when compared with the large grain size samples^[8] on the first thermomechanical treatment (training).

Previous results, also obtained by our group, showed that a refined microstructure can affect the beyond the shape recovery properties also some mechanical properties: Vickers hardness, yield stress ($\sigma_{0,2\%}$) and volume fraction ε -martensite^[6].

The austenite grain size influence on the shape recovery it is a very important parameter to be studied, because can also to influence others properties.

Some authors believe that the grain size don't affect the shape recovery performance theses alloys^{[12].}

This work shows some results about the grain size influence on the shape recovery for Fe-Mn-Si-Cr-Ni-Co alloy submitted at several training cycles.

2. EXPERIMENTAL PROCEDURE

The alloy used in this work was melted in an vaccum induction furnance (VIM) and present the chemical composition (wt.%): Fe balance, 0.009%C, 8.26%Mn, 5.25%Si, 12.81%Cr, 5.81%Ni and 11.84%Co.

The material was hot rolling (1473K) and after treated at 1323K by different times to obtain different grain sizes samples, table 1.

Table 1. Austenite grain sizes obtained with heat treatment time
--

Heat Treatment Time	Grain Size Average	
(minutes)		
	ASTM	μm
10	4	75
60	3.5	106
480	3	129

To induce the $\gamma(\text{fcc}) \leftrightarrow \epsilon(\text{hcp})$ martensitic transformation the samples was submitted to six thermomechanical cycles. Such cycle correspond at 4% compression (to induce the ϵ -martensite) and heating to 873K for 30 minutes (to shape recovery) then cool to room temperature. The specimen dimensions were 9mm in length by 6mm in diameter.

For such cycle, the dimensions of the samples it was measures (after deformation and recovery shape), figure 1.



Figure 1. Treatment thermomechanical cycle.

The states presented in the figure 1 represent: (1) the initial state, (2) deformed state by 4% compression ($\gamma \rightarrow \epsilon$), (3) unload – Elastic Recovery (E_R) and (4) Shape Recovery (S_R) – by heating . The shape recovery S_R it was measure using the equation (1):

$$\mathbf{S}_{\mathbf{R}} = (\mathbf{h}_3 - \mathbf{h}_2) \tag{1}$$

The elastic recovery (E_R) it was analyzed using the equation (2):

$$\mathbf{E}_{\mathbf{R}} = (\mathbf{h}_2 - \mathbf{h}_1) \tag{2}$$

The total shape recovery (T_{SR}) is represent by E_R and S_R contribution:

$$T_{SR} = (E_R + S_R) \tag{3}$$

X-ray diffraction (XRD) may be used do detect and quantify ε -martensite using CuK α radiation and optical microscopy for to available the morphology of these phase.

3. **RESULTS**

The shape recovery as a function grain size it was observed for the samples with different grain sizes average: 75,106 and 129µm obtained during the heat treatment, table 1.

The figure 2 present the typical curve obtained during compression test. Similar curve was obtain for all samples studied. The segment AB represent the load for induce the ε -martensite. During the unload (BC) have a elastic recovery of material, segment BC'.



Figure 2. Curve obtained during the compression test to induce ε -martensite.

The presence of ε -martensite induced during the compressive test it was observe on the X-ray diffraction measurements, where the three main peaks had been identified: $(10.0)_{\varepsilon}$ and $(10.1)_{\varepsilon}$ for all grain size average.

Figure 3 shows the X-ray diffraction pattern of the GS= 75µm samples submitted at (1), (3) and (6) training cycles. The identification of the phases by X-ray diffraction confirms the presence of ε -martensite. In these figure we can see the evolution of phase transformation after some cycles. In this case, was observe $\varepsilon_{\%}$ = 65%.

Using XRD measurements, it was possible quantify the martensite for deformed and recovery states for all grain size and number of cycles. Figure 4 present the volume fraction ε -martensite for the cycles: (1), (3) and (6).



Figure 3. XRD spectra show the evolution of stress-induced ε -martensite for (1), (3) and (6) training cycles, GS = 75 μ m.



Figure 4. Volume fraction ε -martensite as a function grain size for the cycles: (1), (3) and (6), deformed state.

The figure 4 present the volume fraction ε -martensite as a function grain size for the cycles: (1), (3) and (6) before shape recovery (deformed state). The microstructure refinement showed that samples with small grain size can induce more easily the transformation $\gamma \rightarrow \varepsilon$, the boundary grain size it is a point of nucleation of ε -martensite. The result presented in the figure 4, contributed for a better performance on the shape recovery. The results indicated that samples with small grain size facilitate the transformation of ε -martensite.

When the samples are recovery, occur the reverse transformation $\varepsilon(hcp) \rightarrow \gamma(fcc)$ resulting on the SME. Figure 5 present the volume fraction martensite after heating for (1), (3) and (6) cycles. This result shows that grain size reduction also facilitated the reverse transformation, that is result of movement reverse of Shockley dislocation. For small grain size this movement would be facilitated.



Figure 5. Volume fraction ε -martensite as a function grain size for the cycles: (1), (3) and (6), recovery state.

The influence of microstructure refinement on the shape recovery is presented in figure 6. On the first cycle training the samples with average austenite grain size 75 and 129 μ m presented 32 and 16% of shape recovering.

The improving on the shape recovery for small grain size sample, it is related with the fact on that the grain size reduction makes easier the reverse transformation γ (fcc) $\rightarrow \epsilon$ (hcp).



Figure 6. Shape recovery (S_R) as a function grain size for the cycles (1)-(5).

Previous works showed that the yield stress $\sigma_{0,2\%}$, start $\gamma \rightarrow \varepsilon$ transformation, decreased with the grain size reduction, being more easy to induce these phase.

According to authors for the samples with GS=75 and 129 μ m, had a $\sigma_{0,2\%}$ =246 and 322MPa^{[8].} In other words, the microstructure refinement (grain size reduction) create conditions for the appearance of the martensite phase.

In terms of elastic recovery, the alloy presented 25% of elastic recovery, approximately, on the first cycle for the small grain size and 21% for large grain size. It was not observed great variations in this property.

The contribution of E_R and S_R resulted on the total shape recovery (T_{SR}). The sample with 75µm (GS) presented 57%, while that the for 129µm (large grain size) the shape recovery it was 37%. The figure 7 shows the shape recovery for all grain size conditions and number of cycles.



Figure 7. Total shape recovery as a function austenite grain size.

4. CONCLUSION

We can conclude that the austenite grain size reduction makes easier the transformation and reversion $\gamma(\text{fcc})\leftrightarrow\epsilon(\text{hcp})$ resulting on the improvement shape recovery for these alloy on the first cycle, result very important for the practical applications.

The grain size effect on the transformation $\gamma \rightarrow \varepsilon$ it's related with the fact that small grain size sample have a more points of ε -martensite nucleation.

During heating the reverse transform $\varepsilon \rightarrow \gamma$ also is facilitated for small grain size where the movement of Shockley dislocation is more easy.

On the first compression cycle the sample with $GS = 75\mu m$ presented a shape recovery 50% upper when compared with the condition of $GS = 129\mu m$, therefore we can affirm that the austenite grain size reduction improvement the shape recovery properties on the first cycle training.

5. ACKNOWLEDGMENTS

The authors would like to thank CNPq, FAPESP, AEB Vilares Metals S.A for supporting the shape memory development project.

6. REFERENCES

[1] S. Kajiwara. Materials Science and Engineering, Vol. A273, pp. 67-88, 1999.

[2] N. Bergeon, S. Kajiwara, T. Kikuchi, Acta Materialia, Vol. 48, pp. 4053-4064, 2000.

[3] A. Sato, T. Masuya, M. Morishita, S. Kumai, A. Inoue, *Shape Memory Materials*, pp. 223-226, 2000.

[4] J. Otubo, P.R. Mei, S. Koshimizu, Journal de Physique IV, Vol.5, pp. 427-432, 1995.

[5] J.Otubo, P.R. Mei, S. Koshimizu, AH. Shinohara, C.K. Suzuki, C. K, *Materials Science and Engineering*, Vol. A273, pp. 533-537, 1999.

[6] J. Otubo, F.C. Nascimento, P.R. Mei, L.P. Cardoso, M. Kaufman, *Materials Transactions*, Vol. 43, n.5, pp. 916-919, 2002.

[7] F.C. Nascimento, J. Otubo, F.V. Sorrila, P.R. Mei, *Acta Microscópica*, Supplement A, pp. 177-178, 2002.

[8] F.C. Nascimento, Thesis: State University of Campinas, Unicamp, Brazil, 2002, pp. 138.

[9] T. Shiming, L. Jinhai, Y. Shiwei, Scripta Materialia, Vol. 25, pp. 2613-2615, 1991.

[10] J. H. Jun, C. S. Choi, *Materials Science and Engineering*, Vol. A257, pp. 353-356, 1998.

[11] T. Masuya, N. Yoneyama, S. Kumai, A. Sato, *Shape Memory Materials*, Vol. 327-3, pp. 267-270, 2000.

[12] M. Murakami, H. Suzuki, Y. Nakamura, *Materials Transaction ISIJ*, Vol. 27, pp. 87, 1987.