Influence of Austenite Grain Size on Mechanical Properties of Stainless SMA

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This paper presents experimental results relating the initial austenite grain size to bulk hardness, compressive yield stress (σ₀.2%ε) and volume fraction of stress-induced ε martensite. It is shown that the bulk hardness obeys quite closely the Hall-Petch equation while the yield stress, σ₀.2%ε, decreases with decrease of grain size, indicating that the induction of ε martensite mechanically is easier for the materials with finer grains. This fact is corroborated by the observed increase of the volume fraction of stress-induced ε martensite with decrease of grain size. Inversely, after shape recovery heating, the volume fraction of residual stress-induced ε martensite increases as the grain size increases, e.g. the increase in grain size hinders the reversible martensitic transformation.

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1. Introduction

Study of stainless shape memory alloys started in the 1990’s, 1–6 and since that time intensive work has been carried out worldwide with the goal of the shape memory effect improvement. The stainless shape memory alloys can be considered as an alternative to the more expensive NiTi shape memory alloys, in such applications as pipe couplings, because its shape recovery upon pseudoplastic deformation is about 3 to 4%. Several researchers, including the present authors, have shown that the degree of shape recovery depends on the chemical composition and on the thermomechanical treatment, such as training cycles. The process of training in terms of practical application is not feasible due to two main reasons: it increases the production cost and also, depending on the application, it is sometimes impossible to perform the training operation. Therefore it is important to have a material with good intrinsic shape recovery with no need for training. Ogawa and Kajiwara7 who were the first to obtain 80% shape recovery after 4% strain without cycling, attributed the good shape recovery to formation of a mixed of FCC and HCP structures of nanometric scale. Later, Otubo et al.8,9 studied two stainless shape memory alloys, Fe–Mn–Si–Cr–Ni–C and Fe–Mn–Si–Cr–Ni–Co–C, and showed that, after the same thermomechanical treatment, the alloy without Co addition, always showed a better shape recovery, almost 80% shape recovery after 4% tensile strain, while the Co-containing alloy displayed around 65% shape recovery for the same amount of pre-strain. Besides the difference in chemical composition that could influence the shape recovery, another striking difference was in their grain size. After the same thermomechanical treatment, the alloy without Co addition always produced a finer grain structure, and one of the hypotheses explaining its better performance was attributed to this fine grain. Another aspect that should be analyzed is the relation between formation of secondary α′ martensite and austenite grain size. As shown by Otubo et al.,9 the alloy without Co addition generated the secondary α′ martensite in the localized areas of coarse grain where the primary ε martensite bands were formed, indicating that the transformation sequence is γ → ε → α′. Formation of α′ martensite was not observed in the areas with the fine grain structure. The formation of α′ martensite is undesirable in terms of shape recovery due to its hindering effect on the reverse transformation of ε → γ.10,11 Moreover, when the reverse transformation occurs, the α′ → γ transformation takes place directly, skipping the ε phase formation.

Only a few data regarding austenite grain size, shape memory effect, mechanical properties, volume fraction of ε martensite, etc., have been so far reported for the stainless shape memory alloys and some of them are controversial. Murakami et al.12 working on polycrystalline Fe–32Mn–6Si stated that no difference in terms of shape recovery with the grain size varying from 20 to 200 µm has been detected. Later Shiming et al.13 using bending test on a polycrystalline Fe–30Mn–6Si sample, showed that the shape recovery decreased from 75% for the material with the 10 µm grain size to 20% for the one with 60 µm grain size. This decrease in shape recovery was attributed to the generation of perfect dislocations in the sample with coarser grain structure. In order to verify these conflicting findings, we selected the alloy with Co addition from which several samples with various initial austenite grain size were prepared. The objective is to analyze the influence of the grains size on the mechanical properties such as compressive yield stress, hardness, and their relation to stress-induced ε martensite.

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2. Experimental Procedure

A stainless Fe-based shape memory alloy ingot used in this work, of which the composition (mass%) was Fe (balance)–0.044C–7.8Mn–5.16Si–13.02Cr–5.74Ni–11.85Co, was produced by conventional vacuum induction melting (VIM). The ingot of 65 × 65 mm² was heated at 1450 K for 7.2 ks, hot forged into bar of 40 × 40 mm² and longitudinally sectioned into 20 × 20 mm² bars. These bars were heated to 1370 K for 3.6 ks, hot rolled into a 10 mm in diameter rod, solution treated at 1320 K for 3.6 ks and then quenched into water. The solution treated rod was cold swaged with 40% in area reduction, cut into several parts, and then, each part was annealed at 1320 K for different periods of time in order to obtain different initial grain sizes.

Mechanical properties for each rod with a different initial grain size were evaluated by compression tests and hardness measurements. The material was subjected to six thermomechanical cycles which consisted of 4% deformation by compression, cut into several parts, and then, each part was annealed for 3.6 ks, hot rolled into a 10 mm in diameter rod, solution treated at 1320 K for 3.6 ks and then quenched into water. The solution treated rod was cold swaged with 40% in area reduction, cut into several parts, and then, each part was annealed at 1320 K for different periods of time in order to obtain different initial grain sizes.

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As it has been shown in the earlier work the transformation temperatures $M_S$, $A_S$ and $A_F$ for this alloy are 312, 368 and 468 K (the grain size was not measured in that work). Therefore the alloy is partially martensitic at room temperature. Actually, the volume fraction of thermally induced $\varepsilon$ martensite is around the 4%. The final annealing at 1320 K for 1.8 ks, and then cooling to room temperature. The sample dimension for compression test was 9 mm in length by 6 mm in diameter. The load used for Vickers hardness was 49 N. The volume fraction of $\gamma$–FCC austenite phase and $\varepsilon$–HC martensite phase were determined using X-ray diffraction.

3. Results

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Figure 1 presents the compressive yield stress (hereafter called simply yield stress), $\sigma_{0.2\%}$, as a function of number of cycles, and each curve represents a different austenite mean grain size (hereafter called grain size). It is clearly seen that the yield stress decreases as the number of cycles increases mainly on the first few cycles due to training effect. Another aspect that should be pointed out is that, for a fixed cycle, the yield stress increased with increase of the grain size. Figure 2 highlights this behavior for the first compressive loading showing the yield stress (solid dots) as a function of grain size.

![Fig. 1 Compressive Yield Stress, $\sigma_{0.2\%}$, as a function of the number of the thermomechanical cycles for various austenite grain sizes.](image1)

![Fig. 2 Compressive Yield Stress, $\sigma_{0.2\%}$ (the first cycle) and Vickers Hardness (just annealed) as a function of austenite grain size.](image2)

The yield stress increased from 226 MPa at 35 $\mu$m to 242 MPa at 88 $\mu$m. Since the yield stress is the stress necessary to mechanically induce the martensite ($\gamma \rightarrow \varepsilon$), it means that the induction of $\varepsilon$ martensite is hindered by increase in grain size. The same figure also shows the hardness (open dots) as a function of grain size for the solution treated samples (the samples that are in the same state as the samples before the first compression loading). Here it can be observed that the hardness decreases as the grain size increases following closely the classical Hall-Petch behavior. That is, the load used was sufficient to deform the sample plastically generating perfect dislocations (work hardening).

Figure 3 shows the results of hardness measurements on the sixth thermomechanical cycle just after 4% compression (solid dots), and after shape recovery heating at 873 K for 1.8 ks (open dots). For the 4% strained samples, the observed behavior is similar to that shown in Fig. 2, i.e., the hardness decreases as the grain size increases. The opposite behavior is seen after shape recovery heating, i.e., the hardness increases as the grain size increases, almost reaching the value observed in the as-deformed sample. This result indicates that the larger the grain size is, the lower is the degree of the reverse transformation of stress-induced $\varepsilon$ martensite, that will result in increase of hardness. This is corroborated by mea-

![Fig. 3 Vickers Hardness after 4% strain at the 6th cycle and after shape recovery annealing at the 6th cycle.](image3)
The decrease in yield stress, \( \sigma_{0.2\%} \), and the increase in stress-induced \( \varepsilon \) martensite as grain sizes decrease is an indication that the \( \gamma \rightarrow \varepsilon \) transformation is facilitated by grain refinement. Even though unverified, based on the above results, it can be inferred that the martensitic transformation temperature, \( M_S \), should increase as the grain size decreases. These results are rather opposite to the well-known effect of \( M_S \): temperature increase and the yield stress decrease as the grain size increases. \(^{15-19}\) One explanation for our results could be the following: the forward movement of Shockley partial dislocations to induce the HCP band martensite generates shear strain that should be accommodated at the grain boundary. This accommodation could be favored by the sample with a smaller grain size since the net boundary surface area is much larger. Besides, assuming that the Shockley partial dislocations, once nucleated, should travel all the way to the grain boundary, the travel path would be smaller, the smaller the grain size is. As shown by Bergeon et al., \(^{14}\) if the deformation is large or the grain size is large, as in this work, more than one variant needs to be activated to accommodate the shear strain. The consequence is the necessity of higher stress to induce the martensite and, for the same amount of macroscopic deformation, the volume fraction of stress-induced martensite would be lower in the samples with larger grain size, as observed in this work. If only one variant is activated, the backward movement of Shockley partial dislocations would be easier. The research accomplished in the present work shows that no stress-induced martensite was left in the sample with the smallest grain size, while in those with the largest grain sizes the content of the residual stress-induced \( \varepsilon \) martensite was about 20\%. Therefore, it can be concluded that the shape recovery should be better for the samples with small grain sizes. Unfortunately, for this set of samples it was not possible to record the amount of shape recovery due to lack of precision in measuring the sample length, which was too short (9 mm). A new set of samples of 20 mm in length was prepared and the preliminary results indicate that the smaller grain size enhances the shape recovery, which corroborates the initial hypotheses. The influence of grain size on the martensitic transformation temperature on stainless shape memory alloys will also be analyzed to complement above results.

5. Summary

Samples of a stainless shape memory alloy with the grain size varying from 38 to 88 \( \mu \)m were investigated. The main results of this work are:

(1) For the same grain size, the compressive yield stress, \( \sigma_{0.2\%} \), decreases as the number of training cycles increases, mainly during the first few cycles due to the training effect.

(2) The compressive yield stress, \( \sigma_{0.2\%} \), increases with increase of the grain size demonstrating that the larger the grain, the more difficult it is to stress-induce the \( \varepsilon \) martensite. This fact was corroborated by measurement of the volume fraction of the stress-induced martensite, which decreased with increase of the grain size.

(3) The reverse transformation of stress-induced \( \varepsilon \) martensite was complete for the samples with the smallest grain size, while for those with larger grain sizes some
amount of stress-induced $\varepsilon$ martensite remained after shape recovery heating.

(4) The hardness of the as-annealed samples, and also of the ones that have undergone six thermomechanical training cycles (before shape recovery heating), decreased as the grain size increased following closely the Hall-Petch behavior.

(5) The shape recovery should be improved by decrease in grain size, as has being shown by the preliminary results obtained by this group using longer samples. Further work on the latter issue is under way, and the results are soon to be published.

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REFERENCES