Study of tensile behaviour of Fe base shape memory alloys during mechanical cycling

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Abstract. The governing mechanism of Shape Memory Alloys (SMAs) relays on a reversible martensitic transformation between parent (austenite) and stress induced martensite phases. Martensite transformation is a first order phase transition which involves the presence of an invariant (habit) plane which serves as an interface between the two transforming phases. In the specific case of FeMnSi-base SMAs the formation and reversion of ε-hexagonal close 7packed (hcp) stress induced martensite to γ-face centred cubic (fcc) austenite proceeds by stacking fault migration. However, the reaction is not perfectly reversible, in such a way that an amount of retained stress induced martensite, that does not revert to austenite during unloading, is accumulated after each loading-unloading cycle and this evolution is directly related to that of permanent strain. The present paper investigates the relationship between macroscopic and microscopic effects of mechanical cycling. In this purpose, tensile loading-unloading tests were applied to specimens prepared from Fe-18Mn-3Si-7Cr-4Ni SMA (mass. %) by powder metallurgy routine. The tensile tests were applied by means of two experimental test protocols: (i) with constant maximum stress and (ii) with constant maximum strain. In each case, the effect of the number of applied cycles was investigated, while monitoring the evolution of total and permanent strains and of maximum stress and permanent strain, respectively. By means of dedicated software, the variation tendencies of the above mechanical parameters, with the number of cycles have been determined. Finally, the evolution of stress induced martensite has been discussed as a result of microstructural analysis.

1. Introduction
Fe-Mn-Si based shape memory alloys (SMAs) were discovered by Prof. A. Sato et al. [1], developed by Prof. M. Murakami et al. based on Fe-30 Mn-6 Si (mass. %) [2] and optimized by Prof. H. Otsuka, under the form of two potential candidates for low price (as compared to NiTi base alloys) SMA applications: Fe-28 Mn-6 Si-5 Cr [3] and Fe-14 Mn-5 Si-9 Cr-5 Ni [4].

The mechanism of shape memory effect (SME) is represented by the thermally induced reversion of ε-hexagonal close-packed (hcp) stress-induced martensite to γ-face centred cubic (fcc) austenite [5]. Besides ε-hcp, α′-body centred cubic (bcc) martensite can also be induced by cooling or straining, at Mn contents or intense deformation [6], with detrimental effects on SME magnitude [7].

One accessible example of stress induced formation of ε-hcp is the mechanical loading during tensile testing which is defined as the most effective method of assessing material’s behaviour upon deformation [8].

The macroscopic behaviour during a tensile loading-unloading cycle depends upon the initial structure of the specimen. If the initial structure is internally-twinned martensitic, then the deformation
process occurs as illustrated in figure 1.

![Figure 1](image1.png)

**Figure 1.** Tensile behaviour of CuZn based martensitic SMA specimen with internally twinned structure: (a) stress-strain variation during a loading-unloading cycle of a Cu-14.86 Zn-5.81 Al (mass %) SMA; (b) microstructural mechanism of gradual detwinning of a self-accommodated group of four martensite plate variants [9, 10].

During tensile loading, the following phenomena occur: 0A-detwinning; AB-elastic straining; BC-plastic deformation. During unloading, the specimen shrinks back elastically on CD and finally becomes twinned again, on DE [9]. The mechanism of gradual detwinning is shown in figure 1(b). Considering that initially there are four martensite plate variants (1 to 4) the gradual increase of stress enhances the growth firstly of variants 3 and 4 and finally only of variant 4 which has the most favourable orientation with respect of stress axis, from the point of view of Schmidt’s law [10].

If the initial structure martensitic structure is face centred cubic (fcc) austenitic than the gradual increase of tensile stress would gradually induce hexagonal close packed (hcp) ε martensite, according to the mechanism illustrated in figure 2.

![Figure 2](image2.png)

**Figure 2.** Structural-morphological changes accompanying stress induced formation of martensite in FeMnSi based SMAs specimens with initial austenitic structure: (a) stress-strain variation during a tensile failure test of a Fe-28Mn-6Si-5Cr (mass %) SMA; (b) microstructural mechanism of γ (fcc) ↔ ε (hcp) martensitic transformation at atomic, plate variants and surface relief levels [11, 12].
The shape of the tensile failure curve shown in figure 2(a) is typical for the materials that undergo transformation induced plasticity (TRIP) [11] the mechanism of which is best described by the schematized pile of martensite plate variants illustrated in the middle left side of figure 2(b). Each additional increase of stress caused a supplementary augmentation of tensile strain. By cumulating all these individual shifts, ultimate strains over 80 % can be reached, as in the example shown in figure 2(a). Figure 2(b) shows that, by means of a single atomic shuffle fcc structure can change to hcp [12].

The present paper aims to investigate which is the effect of mechanical cycling on the tensile behaviour of FeMnSiCrNi SMAs processed by powder metallurgy.

2. Experimental procedure

By means of powder metallurgy routine, comprising pressing and sintering at 1120°C under cracked ammonia (75 % N₂ + 25 % H₂), samples were produced from commercial powders with zinc stearate binder with the chemical composition Fe-18Mn-3Si-7Cr-4Ni (mass. %). Homogenization annealing (1100°C/ 1 hr/ water) and hot-rolling 1000°C to 1mm thickness were applied to increase chemical homogeneity and compactness, respectively. Tensile specimens were cut by wire spark erosion, with gauge dimensions 1×4×20 mm [13].

Tensile loading-unloading cycles were applied with an INSTRON 3382 tensile testing machine at a deformation rate of 2.77×10⁻⁴ sec⁻¹. Two tensile test protocols were applied: (i) with a constant maximum stress of 600mMPa and (ii) with a constant maximum strain of 4 %.

After tensile testing, scanning electron microscopy (SEM) microstructural analysis was performed on a SEM–VEGA II LSH TESCAN scanning electron microscope, coupled with an EDX – QUANTAX QX2 ROENTEC detector.

3. Experimental results and discussion

3.1. Tensile mechanical cycling

The stress-strain curves recorded during 100 tensile loading-unloading cycles, up to 600MPa, are shown in figure 3.

![Figure 3. Tensile stress-strain curves during 100 tensile cycles up to 600 MPa maximum stress.](image-url)
Both the beginning of loading and the end of unloading present the same characteristic portions which were associated with detwinning-twinning, according to the tensile curve shown in figure 1(a). In addition, the shape of the curve does not reveal the same roundness observed in figure 2(a), which is characteristic for TRIP behaviour [13]. During common tensile tests, the specimens failed above 16 % strain.

It is obvious that both total and permanent strains increased during mechanical cycling. The detail reveals the agglomeration of tensile curves during the last cycles which suggests the stabilization of tensile behaviour.

The stress-strain curves recorded during 20 tensile cycles up to the maximum strain of 4 % are shown in figure 4.

In this case, as well, the contour of the last loops became more accentuated, suggesting the stabilization tendency of tensile behaviour.

The recorded values of total and permanent strains, during 100 tensile cycles up to 600MPa, were plotted as a function of the number of cycles. The result is shown in figure 5.

Obviously, both total and permanent strain increase with the number of cycles but this increase experiences a saturation tendency. For this reason, the polynomial fit gave very good results with a second order polynomial, under the form equation (1):

\[ e = B_2N^2 + B_1N + I \]  

where \( e \) – total or permanent strain; \( B_2, B_1 \) and \( I \) (intercept) are the coefficients determined by ORIGIN software, while performing the second order polynomial fit and \( N \) – number of cycles.

The plots of the two polynomial fits are rather parallel which suggest that the two parameters tend to vary in the same way, with the number of tensile cycles.

In addition, the standard errors, listed in the tables inserted in figure 5, are very small which proves the validity of the theoretical variations.

The negative values of \( B_2 \), the coefficient of the second order independent variable (N), requires a maximum value of strain, according to the polynomial model. The number of cycles where this maximum is reached is given by:
Figure 5. Evolution of total and permanent strains during 100 tensile cycles up to 600 MPa maximum stress, according to the results shown in figure 3 and second order polynomial fit.

\[ N_{\text{max}} = -\frac{B_1}{2B_2} \]  \hspace{1cm} (2)

By introducing the corresponding values from the tables of figure 5 it follows that \( N_{\text{max}} \approx 75 \) cycles and \( N_{\text{max, ep}} \approx 74 \) cycles. These results suggest that there is a synchronization between the evolutions of total and permanent strains during tensile cycling.

The variation of the values of maximum stress and permanent strain, recorded during 20 tensile cycles up to 4% maximum strain, are plotted as a function of the number of cycles in figure 6.

Figure 6. Evolution of total stress and permanent strains during 20 tensile cycles up to 4% total strain, according to the results shown in figure 4 and second order polynomial fit.
In this case, as well, the reduced value of standard errors supports the validity of second order polynomial fit. By applying the parameters from the table inserted in figure 6 within equation (2) it follows that \( N_{\text{max,exp}} \approx 15 \) cycles.

It is obvious that both values of the number of cycles corresponding to maximum strains are irrelevant for the actual evolution of the specimen during mechanical cycling. Normally, if there were a maximum, strain would have to decrease during further increase of the number of cycles. Or this behaviour was not observed during testing. Permanent and maximum strains continue to increase due to the occurrence of fatigue phenomena, which are beyond the purpose of the present paper. The theoretical presence of the respective maxima must be considered as a technological limit, beyond which the increase of the number of cycles does not have any marked structural effects on the specimen.

3.2. Microstructural analysis

The representative SEM micrographs of a specimen in initial and mechanically cycled conditions are shown in figure 7.

![Figure 7. SEM micrographs illustrating mechanical cycling effects: (a) initial state; (b) after 75 cycles up to 600 MPa.](image)

Figure 7(a) illustrates the presence of scarce martensite plates at the specimen in initial state. After 75 cycles, the number of martensite plates markedly increased, in spite of the barriers represented by grain boundaries and pores which were filled with zinc stearate that was volatilized during sintering [14].

4. Conclusions

By correlating mechanical cycling and microstructural analysis results, the following conclusions can be drawn, regarding the tensile behaviour of Fe-18Mn-3Si-7Cr-4Ni (mass. %) SMA produced from commercial powders with zinc stearate binder:

- the experimental material revealed a tensile stabilized behaviour which enabled the application of 100 loading-unloading cycles up to 600 MPa;
- during 100 mechanical cycles up to 600 MPa, both total and permanent strains experienced similar variation tendencies, theoretically approximated by second order polynomials that reached the maximum values after 74-75 cycles;
during 20 mechanical cycles up to the maximum strain of 4 %, maximum stress decreased and permanent strain increased emphasizing the same stabilization tendency approximated by a second order polynomial;

- the three second order polynomials, that model the saturation tendencies of strain:
  \[ \varepsilon_{600\text{MPa}} = -6.87402 \times 10^{-5} N^2 + 0.01029 N + 5.18953, \]
  \[ \varepsilon_{p600\text{MPa}} = -1.18843 \times 10^{-4} N^2 + 0.01747 N + 2.0689 \]
  \[ \varepsilon_{p4\%} = -6.03362 \times 10^{-4} N^2 + 0.01776 N + 1.8567 \]
  are valid only until reaching number of cycles (N) that correspond to the maximum values, namely 75, 74 and 15 cycles respectively;

- an obvious increase of the number of martensite plates was noticed after mechanical cycling.

5. References

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Acknowledgments

This work was supported by UEFISCDI through project code PN-III-P4-ID-PCE-2016-0468, contract no. 76/2017.