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A comparative study of austenitic structure in NiTi and Fe based shape memory alloys after severe plastic deformation

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Abstract

The effect of high speed high pressure torsion (HS-HPT) was studied in NiTi and FeMnSiCr SMAs, by comparison. Severe plastic deformation was performed in austenite state for both types of alloys. The alloys subjected to HS-HPT, reduced their grain size due to microstructure fragmentation by compression and torsion. The active elements were achieved being able to support variable ranges of processing parameters like force, pressure, rotation speed and time of torsion. The evolution of microstructural refinement in the samples subjected to different deformation by HS-HPT, were studied by optical and scanning electron microscopy observation and the thermal effect was revealed using differential scanning calorimetry (DSC).

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1. Introduction

One of the new trends in industry is to stimulate the development of 'smart' systems with adaptive functions [1,2]. Shape memory alloys (SMAs) or 'smart alloy' are used as sensors, actuators and micro-controllers [3,4]. SMAs possess several interesting characteristics, such shape memory effect and superelasticity based on martensitic

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transformation [5,6]. NiTi SMAs hold the first position in the industrial market [1,7], but iron-based shape memory alloys, especially FeMnSi alloys, have also great potential in engineering field [8]. Severe plastic deformation (SPD) plays a significant role in microstructural refinement of grains contributing to enhancement of functional and mechanical properties of a material [9-11]. The high speed high pressure torsion (HS-HPT) is one of the original and reliable SPD methods to achieved advanced grain refinement, being able to perform larger disks as compared to classic HPT, applicable even in the case of difficultly deformable alloys [12,13]. During deformation by HPT method, a sample is subjected to high pressure between two anvils and slow speed, that produces (ultra)fine or a mixture of nanocrystalline and amorphous bulk metallic structure [9-11,14]. In contrast to classical HPT processing, the HS-HPT technology involves rotation speeds of the order of hundreds rpm. Due to the rather elevated rotation speed combined with high pressure, grain size is reduced down to nanostructure level. This technology is unique [15] and allows fabrication of disks with diameters over 35 mm up to 45 mm, noticeable larger as compared to classic HPT.

Data on NiTi HPT have been published, however, less attention has been paid to the FeMnSiCr SMAs, due to the brittleness and the deformation difficulty. In this study, the comparative effects of the structural changing of HS-HPT deformed NiTi and FeMnSiCr SMAs were investigated.

2. Experimental procedure

The selected NiTi alloy was commercial 50.8at% Ni. The iron based SMA, Fe₂₈Mn₆Si₅Cr mass%, was obtained through cold crucible induction melting from powders, using a Fives Celes furnace. The high speed high pressure torsion (HS-HPT) was applied on cylindrical samples of 7 mm in diameter for both SMAs. The rotation speed applied was 1795 rpm and the force was around 120 kN. The initial pressure varies between 2-5 MPa with the groups of alloys. The torsion time for NiTi samples varied between 7 to 19 seconds and 2 to 11 seconds for Fe based alloy. The deformation temperature was about 850 °C. The true plastic strain achieved in the processed material is estimated by the ration between initial and final thickness of the sample, $\epsilon = \ln(h_i/h_f)$. NiTi samples were subjected to deformation degrees of 0.92, 1.76, 2.59 and 3.00, while Fe₂₈Mn₆Si₅Cr were deformed to 0.16, 1.72, 2.04 and 2.33. The NiTi thin discs after HS HPT were up to 0.15 mm in thickness and the FeMnSiCr were 0.18 mm, respectively. The HS-HPT technology has been discussed extensively in a previous paper [12]. The observation of microstructure was carried out using optical microscope and a Zeiss scanning electron microscope (SEM). Differential scanning calorimeter (DSC) was used to detect thermal effects associated with structural transformations occurring in the HS-HPT elements. The temperature range was -40 °C to 120 °C for NiTi and to 300 °C in case of Fe based SMA.

3. Results and discussion

In the case of the deformation degree of 0.92, Fig. 1a shows that the grains exhibit different but apparent deformation and elongation as compared to the as-received NiTi (inside Fig. 1c) that possess a typical B2 austenite structure at room temperature [2,7]. The microstructure of NiTi alloy after HS-HPT processing with low deformation degree is characterized by the combination of dynamic recovery and dynamic recrystallization, but the dynamic recovery is dominant since the grains are obviously elongated. In Fig. 1b are presented a random formation equiaxed grains, distributed almost uniformly. With increasing of true strain to 2.59 the grain boundaries are no longer able to be observed, only an uncertain embossing it can be seen (Fig. 1c).

The microstructure of iron based SMA initial samples shows the dendritic structure (inside Fig. 2c). After HS-HPT processing the microstructure aspect were completely changed showing elongated and oriented grains even at 0.16 deformation degree (Fig. 2a). The curved flow fibering and ambiguous grain boundaries are specific for deformation degree up to 1.72 [2]. The longitudinal cross-section clearly shows defined light deformation areas along with severe deformation bands. In the areas slightly deformed some triangular morphology and two martensite plate variants are significant (Fig. 2c). The severe deformation zones are bright white and after repeatedly etching they are still not etched, resembling with amorphous areas [16-18].

A better insight of morphological changes caused by deformation, on HS-HPT structure, was obtained by SEM observations (Fig. 3 and 4). At the highest true strains achieved, generalized fibering can be observed. In transversal cross section neither grains boundaries nor observable size-individual grains are present. Also a coagulation process

of precipitates is shown. It can be emphasized that the grain refinement of Fe based SMA is higher than NiTi, for comparable deformation degrees applied, in accordance with the plasticity revealed.

The hardness are fairly consistent with microstructure of HS-HPT samples. The value of homogenized NiTi sample was 184 MPa. Increasing the thickness reduction resulted in an increase in hardness until 438 MPa for $\epsilon=3.00$. Average hardness varied from 296 MPa to 452 MPa in case of Fe based SMA.

The DSC thermograms of all specimens after HS-HPT, recorded during heating to 300 °C highlighted the presence of reverse martensitic transformation. The SMAs disks can behave as actuators, as we already presented in our previous papers [12,13]. There is a shift of peaks transformation to higher temperatures, opposite to what achieved after HPT (i.e. As of the homogenized NiTi are -25 °C and of $\epsilon=3.00$ are 13 °C).

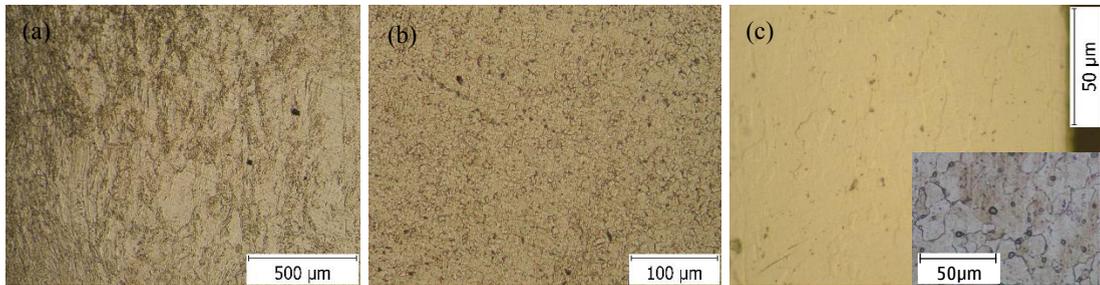


Fig. 1. Optical micrographs on the NiTi samples before (inside c) and after HS HPT process: $\epsilon=0.92$ (a), $\epsilon=1.76$ (b), $\epsilon=2.59$ (c).

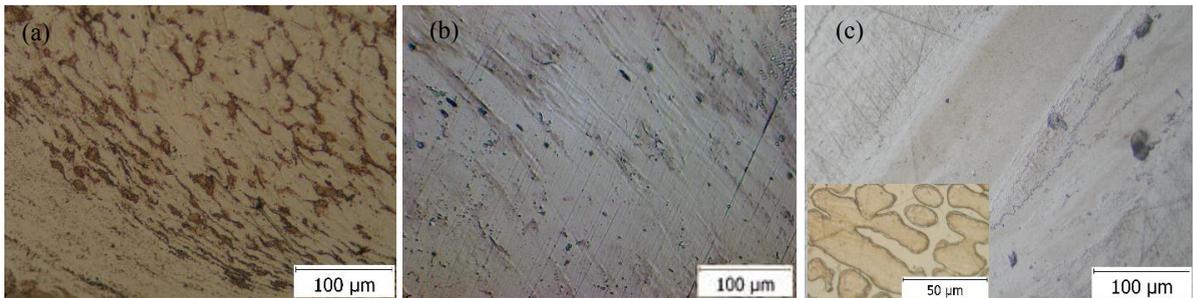


Fig. 2. Optical micrographs on the FeMnSiCr samples before (inside c) and after HS HPT process: $\epsilon=0.16$ (a), $\epsilon=1.72$ (b), $\epsilon=2.04$ (c).

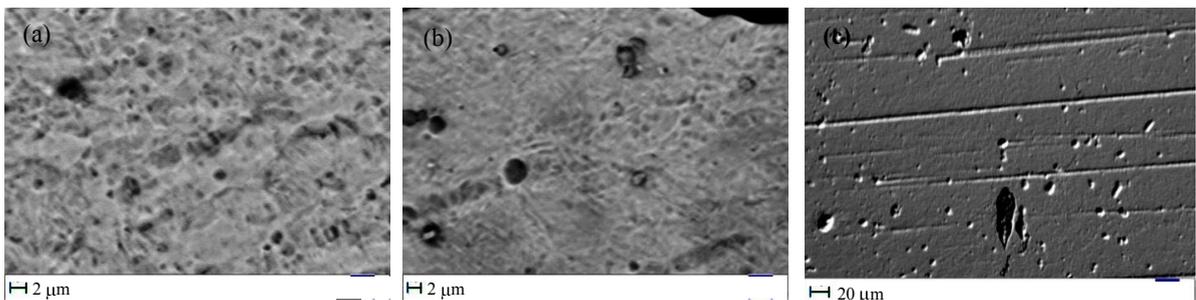


Fig. 3. SEM micrographs on the NiTi SMA after HS HPT process $\epsilon=3.00$:longitudinal (a and b) and transversal (c).

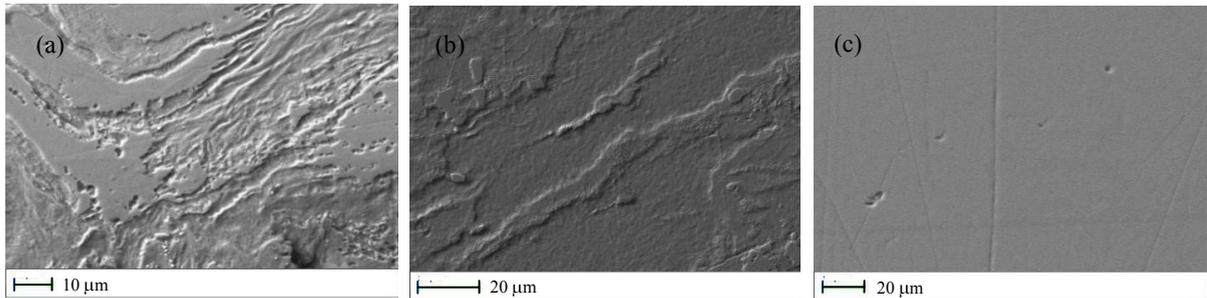


Fig. 4. SEM micrographs on the FeMnSiCr SMA after HS HPT process: $\epsilon=1.72$ longitudinal (a) and $\epsilon=2.33$ longitudinal (b) and transversal (c).

4. Conclusion

HS-HPT was applied on NiTi and FeMnSiCr SMAs, as a new attempt of severe deformation able to lead to nanocrystallization and considerably larger active elements as compared to classic HPT, in the particular case of these difficultly deformable alloys. This technology provides the possibility to obtain functional elements with rounded shapes and diameters up to 45 mm, in both types of alloys. The deformation takes place in few seconds. The way in which deformation occurs is similar; the grains exhibit an annular orientation at low true strain. By increasing the thickness reduction the grain boundaries are no longer observable.

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