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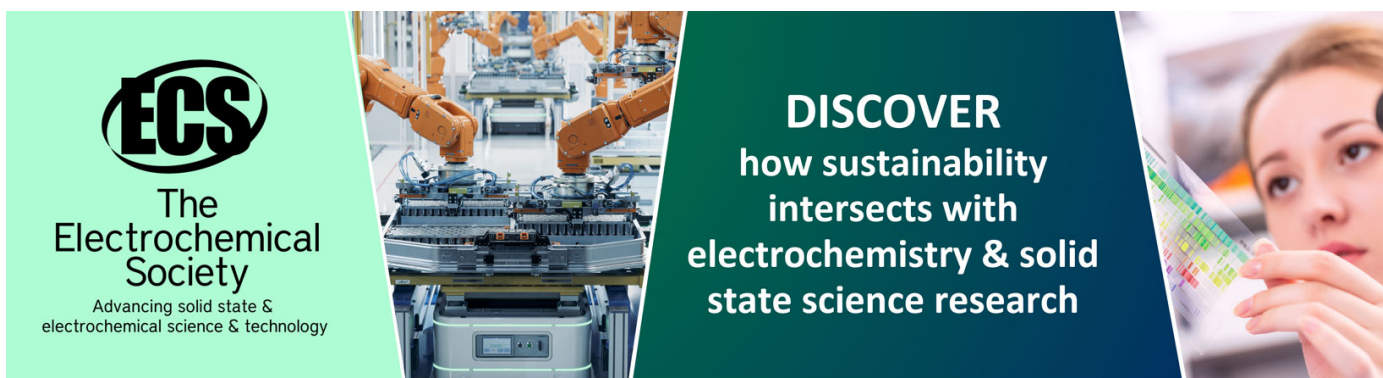
Thermomechanical processing effects on the structure and properties of Fe-based SMAs. II. Evolution of damping behavior

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Thermomechanical processing effects on the structure and properties of Fe-based SMAs. II. Evolution of damping behavior

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Abstract. The present paper analyses the effects of five different heat treatment temperatures, between 973, 1073, ... and 1373 K as well as two mechanical alloying (MA) fractions, 0 and 40 vol. %, on the evolution of damping behavior of an Fe-Mn-Si-Cr-Ni Shape Memory Alloy (SMA) obtained by powder metallurgy (PM). From the 10 types of samples, rectangular specimens were cut by wire-spark erosion for dynamic mechanical analysis (DMA) tests. DMA tests were performed by temperature scans (TS) and strain sweeps (SS). TS were applied between room temperature (RT) and 673 K, at constant amplitude, and thus enabled determining the temperatures of internal friction maxima associated with antiferromagnetic-paramagnetic transition (Néel temperature), A_{50}^N , and ϵ -martensite reversion to γ -austenite, A_{50}^ϵ , as a function of MA'ed fraction and HT temperature. SS were applied during three cycles with increasing strain amplitude and performed at three temperatures: (i) $T_1=RT$; (ii) $T_2 < A_{50}^\epsilon$ and (iii) $T_3 > A_{50}^\epsilon$. The effects of HT temperature and MA'ed fraction on storage modulus (E') values were emphasized for each of the three temperatures $T_{1,2,3}$. An E' -plateau was observed and associated with the formation of ϵ stress induced martensite, during DMA-SS.

1. Introduction

After the first paper, published in 1982, on the shape memory effect (SME) observed at Fe-Mn-Si single crystals [1], polycrystalline Fe-(28-34) Mn-(4-6.5) Si (mass. %, as all chemical compositions will be listed hereinafter) alloys with almost perfect SME were reported in 1987 [2]. With the additions of Cr [3] and Ni [4], two commercial shape memory alloy (SMA) grades were developed, as Fe-28Mn-6Si-5Cr and Fe-14Mn-6Si-9Cr-5Ni.

The first reports on practical applications of Fe-Mn-Si-based SMAs were published in 1991, introducing lock rings for bicycle frame pipes [5]. Subsequently, constrained recovery SME applications [6] were developed, comprising pipe couplings [7], fishplates for crane rail fastening [8], concrete pre-straining rods [9] and embedded stripes for beam curvature control [10]. Up to the present date, the largest SMA applications, ever reported, have been the sixteen 2-tons anti-seismic dampers made in



2014 from Fe-15Mn-4Si-10Cr-8Ni SMA and installed from the first to the fourth floors of a 196-metre skyscraper, “JP Tower Nagoya” that was completed in November 2015 [11]. These dampers exploit the general capacity of SMAs to damp oscillations [12] as well as the particular feature of Fe-Mn-Si based SMAs to retransform, from ϵ -hexagonal close packed (hcp) martensite to γ -face centered cubic (fcc) austenite, not only by heating but also by counter-directional loading [13].

Most of these applications use bulk materials obtained by ingot metallurgy (IM). However, powder metallurgy (PM) accompanied by mechanical alloying (MA) have the potential to overcome most of drawbacks of IM, basically by improving the control of chemical composition and grain size, as succeeded in the case of other SMAs, such as Ti-Ni, or Cu base alloys [14].

Nevertheless, PM Fe-Mn-Si based SMAs, were only scarcely reported in literature. On the other hand, some of present authors have studied quinary PM-MA'ed Fe-Mn-Si-Cr-Ni SMAs, ever since 2009. Thus, it was proved that PM specimens experienced higher stress levels than IM ones, due to larger amounts of α' thermally induced martensite [15], which was additionally stress-induced with the increase of the number of mechanical cycles [16], the amount of MA'ed powder fraction [17] and pre-straining degree [18]. In addition, specific damping capacity experienced an increasing trend with pre-straining degree and MA'ed fraction [19]. Yet, the dynamic behavior of PM-MA'ed Fe-Mn-Si-Cr-Ni SMAs during strain sweeps performed by dynamic mechanical analysis (DMA) was never analyzed and reported. The evolution of dynamic stiffness is particularly important in the case of vibrating pipes, due to the turbulence of transported fluids that can be connected by coupling rings made from PM-MA'ed Fe-Mn-Si-Cr-Ni SMAs, the constrained recovery of which can be controlled by the deformation and thermomechanical training processing technologies [20].

The present paper investigates the effects of heat treatment (HT) and mechanical alloying (MA) on the evolution of damping behavior of an Fe-Mn-Si-Cr-Ni Shape Memory Alloy (SMA) obtained by powder metallurgy (PM).

2. Experimental details

Two groups of specimens, with nominal chemical composition 66Fe-14Mn-6Si-9Cr-5Ni (mass %), were pressed and sintered under argon atmosphere, being designated as: (i) 0_{MA}, from as-blended elemental powders and (ii) 40_{MA} comprising 40 vol. % MA'ed powders, obtained after 4 hrs. high-energy ball milling under Ar atmosphere in order to inhibit the chemical reactivity of oxygen-sensitive alloying elements. After blending, pressing and sintering, further increase of compactness was achieved by six consecutive hot rolling passes at 1373K, until samples' thickness decreased from 4 to 1 mm. By spark-erosion wire cutting, specimens with “dog bone” (gauge dimensions 1×4×20 mm) configuration were prepared for dynamic mechanical analysis (DMA). Heat treatment (HT) was applied with 5 min-holding at five temperatures, 973, 1073, ... 1373K, followed by water quenching. In this way, 10 different sets of specimens, with different MA'ed powder fraction and HT temperature were obtained, designated by MA'ed powder fraction and HT temperature (in °C).

DMA tests were performed on a NETZSCH DMA 242 Artemis, equipped with “three-point bending” specimen holder, under two variants: (i) temperature scans (TS) and (ii) strain sweeps (SS).

TS were applied between RT and 673 K, with a heating rate of 5 K/min and a frequency of 1 Hz. The present authors pointed out that, in PM-MA'ed Fe-Mn-Si-Cr-Ni SMAs, a second internal friction maximum occurs on TS-DMA diagrams recorded on heating, compared to only one found in literature. This low-intensity $\tan\delta$ maximum, located at a temperature designated as A_{50}^N , could be associated with antiferromagnetic-paramagnetic transition characterized by Néel temperature (TN) [22]. In order to confirm the antiferromagnetic-paramagnetic transition, thermomagnetic measurements were done, on a Lake Shore VSM 7410 Vibrating Sample Magnetometer. Temperature variation rate was 2 K/min, applied magnetic field was $H = 20$ Oe and temperature range was RT-723 K.

SS, up to 20 μm -amplitude, were applied at three temperatures: (i) $T_1 = \text{RT}$, (ii) $T_2 < A_{50}^\epsilon$ and (iii) $T_3 > A_{50}^\epsilon$, at constant frequency of 1 Hz. DMA-SS diagrams were recorded under the form of storage modulus vs. (strain) amplitude, at the frequency of 1 Hz.

3. Experimental results and discussion

3.1. Heat treatment and mechanical alloying effects on the response observed by DMA thermal scans

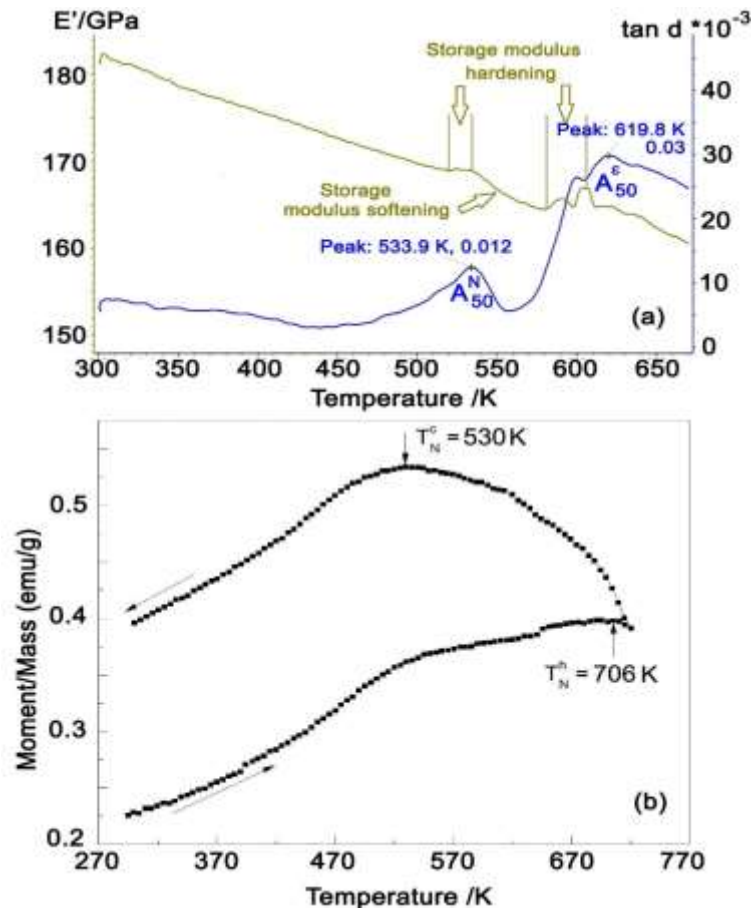


Figure 1. Temperature scan-DMA diagrams, recorded for specimen 0-MA_700, illustrating the variations with temperature of: (a) storage modulus (E') and internal friction ($\tan \delta$); (b) magnetization, during a heating-cooling cycle

One typical example for the variation of storage modulus and internal friction, during DMA-TS on heating, is illustrated in figure 1(a). Considering that during paramagnetic $\rightarrow$ antiferromagnetic transition the change in storage modulus is abrupt but the correlated internal friction peak height is small, because magnetic ordering relaxes much faster than the moving array of atoms associated with the martensitic transformation, it was assumed that the low intensity $\tan \delta$ maximum could correspond to antiferromagnetic $\rightarrow$ paramagnetic transition. In addition, internal friction value is larger in austenite than in martensite because the former possesses a larger number of close packed planes. Therefore the second $\tan \delta$ maximum, which has always larger internal friction values after the peak than before it, could fairly correspond to $\epsilon(\text{hcp}) \rightarrow \gamma(\text{fcc})$ reverse martensitic transformation. Figure 1(b) displays magnetization variation, during heating, for the same specimen.

Martensite reversion to austenite, during heating, is accompanied by a local drop in storage modulus (E'), since martensite is harder than austenite. Consequently, two modulus hardening segments were identified by arrows in figure 1(a). It is noticeable that the temperatures of modulus hardening segments coincide with the first halves of internal friction maxima. The beginning of second increase of E' (larger), during heating, can be associated with the $\epsilon(\text{hcp}) \rightarrow \gamma(\text{fcc})$ reverse martensitic transformation, in figure 1(a). In figure 1(b), above Néel temperature (T_N) the subsequent magnetization decrease on heating corresponds to the paramagnetic state. One can argue that, due to the low amplitude of applied

magnetic field, magnetization increase with temperature could be caused by thermal fluctuations that occur at high temperature or the formation of a new phase. Nevertheless, considering that a similar magnetization maximum was observed during cooling as well, we assume that the presence of the magnetization maxima can be related to antiferromagnetic-paramagnetic transition [23].

The variations of storage modulus and magnetization during heating, for the two fractions of MA'ed powders, corresponding to the lowest (973 K) and highest (1373 K) HT temperatures, were summarized in figure 2. It is noticeable that on most of the thermograms storage modulus increased before magnetization reached its maximum. This delay could be caused by the low value of applied magnetic field.

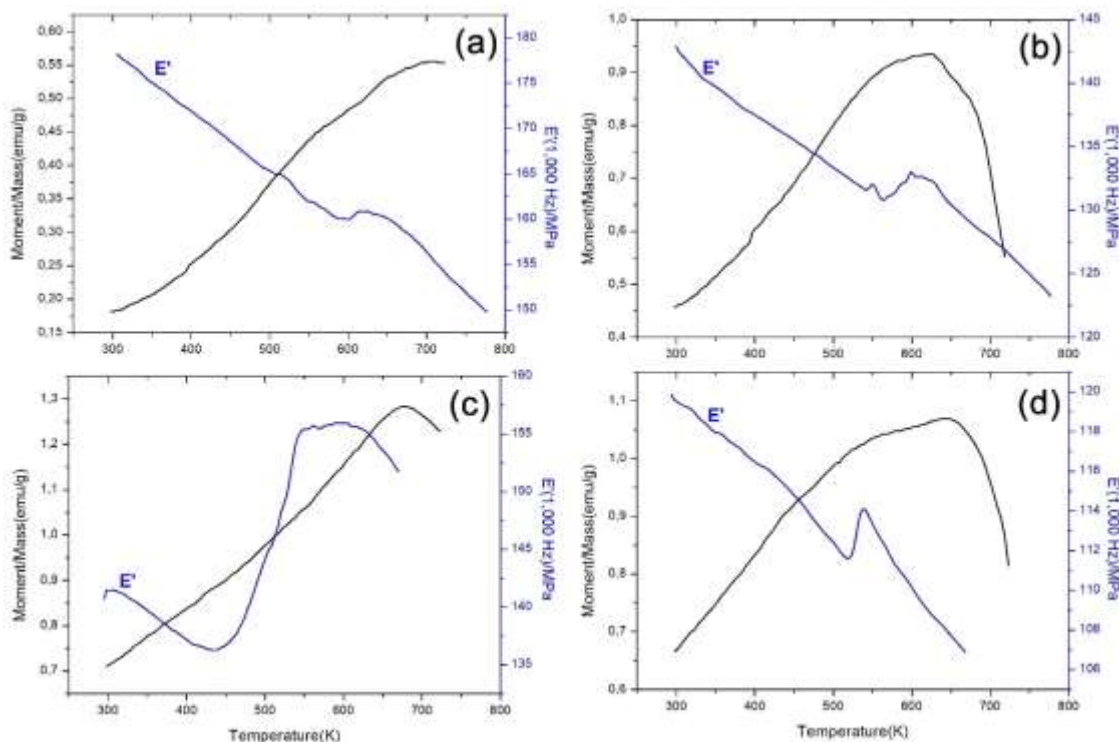


Figure 2. Heat treatment temperature and MA'ed powder fraction effects on the evolution of storage modulus and magnetization during heating: (a) 0_MA_700; (b) 0_MA_1100; (c) 40_MA_700; (d) 40_MA_1100.

The influence of HT temperature, on the positions of the two peaks, is not substantial because dynamic recrystallization was quite active during 1373 K-hot rolling. With increasing MA'ed powder fraction, both A_{50}^N and A_{50}^E experience a global tendency to decrease.

3.2. Heat treatment and mechanical alloying effects on the response observed by DMA strain sweeps

As mentioned above, DMA-SS were performed at three different temperatures. In each case, three SS cycles were applied. A typical SS-DMA diagram comprising storage modulus (E') variation with strain amplitude, is shown in figure 3, corresponding to specimen 0_MA_700, tested at $T_1 = RT$.

In most of the DMA-SS diagrams recorded at RT, E' experienced a plateau between 0.02 and 0.06 % strain amplitudes. This plateau became longer in the second and third SS cycles. Since storage modulus remains constant, instead of continuously increasing with strain amplitude (as expectable in the case of work-hardenable materials), it can be assumed that the plateau can be associated with ϵ -hcp stress induced formation during dynamic loading. In order to have a better perception on storage modulus evolution during SS cycles, an insight of E' values at 0.02 % strain amplitude was shown in figure 3 comprising the three cycles. It appears that E' markedly increased from the first to the second cycle and almost stabilized in the third.

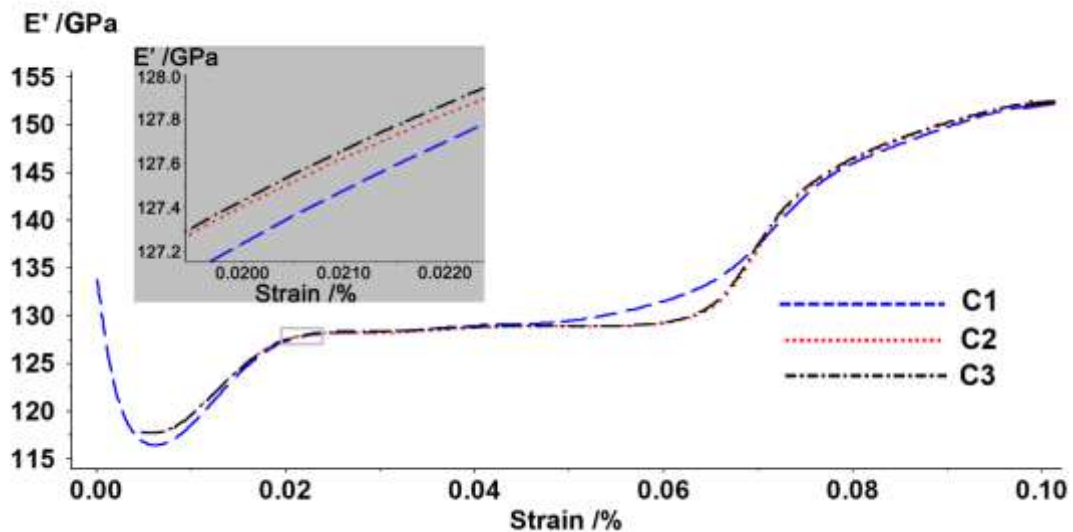


Figure 3. Variation of individual values of storage modulus during three cycles of DMA-strain sweep of 0_MA_700 specimen, tested at $T_1 = RT$ with inset at 0.02 % strain amplitude.

At the specimens tested at T_1 : (i) the values of storage modulus tend to saturate with increasing strain amplitude, displaying a first E' plateau, at most of the specimens; (ii) further increase of strain amplitude caused additional E' saturation, yielding a second plateau (exceeding 200 GPa at specimen 0_MA_1000), which is noticeable only at a part of specimens.

At the specimens tested at higher temperatures (T_2 and T_3), most E' variations display a single plateau or two shorter plateaus at higher MA'ed powder fractions (40_MA).

4. Summary and conclusions

- PM-MA'ed Fe-Mn-Si-Cr-Ni SMAs had porosity degrees between approximately 17 % (at as blended powders) and 5 % at specimens with 40 vol. % mechanically alloyed powders, which contributed to the obtainment of mass magnetization values below 4.5 emu/ g at RT;
- DMA temperature scans revealed two $\tan\delta$ maxima: a low-intensity one located between 463-572 K and a high-intensity one located at 532-650 K. In the opinion of the authors, the former was ascribed to antiferromagnetic $\Rightarrow$ paramagnetic transition, at A_{50}^N and the latter to $\varepsilon(\text{hcp}) \Rightarrow \gamma(\text{fcc})$ reverse martensitic transformation, at A_{50}^ε .
- With increasing MA'ed powder fraction, the temperatures of both A_{50}^N and A_{50}^ε tend to decrease, during DMA-temperature scan tests.
- Storage modulus (E') increased with strain amplitude and cycle number, experiencing a plateau at 0.02-0.06 % (associated with ε -hcp stress induced formation) during DMA-strain sweep tests. The presence of this modulus plateau recommends PM-MA'ed Fe-Mn-Si-Cr-Ni SMAs as potential candidates for RT-dampers, under the form of coupling rings of vibrating pipes.

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