Storage modulus and internal friction variations in a Fe-28 Mn-6Si-5Cr (mass. %) shape memory alloy analyzed by three-point-bending DMA

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Abstract: An Fe-28Mn-6Si-5Cr (mass. %) shape memory alloy (SMA) was investigated, observing the behavior of the material when subjected to dynamic mechanical analysis (DMA) cycling performed as a function of temperature and as a function of amplitude. Strain sweeps were performed at three temperatures: \( T_1 = RT \), \( T_2 = A_{SO} \); and \( T_3 = T_N \), where \( A_{SO} \) and \( T_N \) are the critical temperatures for the middle of reverse transformation of martensite to austenite and for antiferromagnetic-paramagnetic transition. During temperature scans, a modulus increase, with several GPa, and two internal friction maxima were observed on heating. The temperature scans revealed that internal friction increased with increasing the frequency as compared to similar data presented by other authors where a decreasing tendency was noticed.

1. Introduction
Fe-Mn-Si-based shape memory alloys (SMAs) represent a high-output cheap promising alternative [1] to more expensive less stable Ni-Ti-based SMAs [2]. The microstructural mechanism of Fe-Mn-Si SMAS relays on the stress induced formation of \( \varepsilon \)-hexagonal close-packed (hcp) martensite and on its thermally induced reversion (during heating) to \( \gamma \)-face centered cubic (fcc) austenite.

Among Fe-Mn-Si-based SMAs, Fe-28Mn-6Si-5Cr (mass. %) represents a commercial grade alternative developed for constrained recovery [3] and vibration mitigation applications [4]. By constrained recovery shape memory effect, this SMA develops force (stress) during a heating-cooling cycle in deformed state. Constrained recovery applications include pipe couplings [5], fishplates for crane rails [6] and stress pre-straining rods [7]. Vibration mitigation applications are based on the high values of internal friction, which enabled the development of passive anti-seismic dampers [8].

In this work, the variation of internal friction values, of an Fe-28Mn-6Si-5Cr SMA was determined as a function of temperature, strain amplitude and vibration frequency, using a dynamic mechanical analyzer (DMA).

2. Experimental procedure
An Fe-28Mn-6Si-5Cr (mass. %) alloy was obtained in a FIVE CELES levitation induction furnace (25 kW-power, maximum melting temperature – 2273 K, primary vacuum - \( 10^{-4} \) mbar and secondary vacuum - \( 3 \times 10^{-8} \) mbar) and cast into copper molds. The ingots with a diameter and height of 20 mm and 45 mm, respectively, were longitudinally cut and hot rolled (1370 K) until their thickness decreased to 1.2 mm. Rectangular specimens, 0.7 x 4 x 25 mm, were cut on a spark erosion machine, and subjected to dynamic mechanical analysis (DMA) in a DMA E Artemis device using a three-point-bending specimen holder. The DMA tests were done under two variants: (i) temperature scans...
and (ii) strain sweeps. DMA temperature scans were performed with multiple frequency \((1, 5, 10\) and \(20\) Hz) at \(20\) \(\mu\)m amplitude, from room temperature to \(673\) K with a heating rate of \(5\) K/min. DMA strain sweeps were performed at constant frequency of \(1\) Hz, a strain amplitude range from \(0.1\) to \(20\) \(\mu\)m divided in \(21\) equal steps and three different temperatures: (i) \(T_1 = \) room temperature (RT); (ii) \(T_2 = A_{50}^c\) and (iii) \(T_3 = T_N\), where \(A_{50}\) and \(T_N\) are the critical temperatures for the middle of reverse transformation of martensite to austenite and for Néel temperature of antiferromagnetic-paramagnetic transition, respectively.

3. Experimental Results and Discussion

Temperature scans

Figure 1, which presents the variation of internal friction \((\tan \delta)\) as a function of temperature, reveals the presence of two internal friction maxima at \(A_{50}^c\) and \(T_N\).

![Figure 1. Internal friction (\(\tan \delta\)) variation recorded during heating on a hot rolled Fe-28Mn-6Si-5Cr using four different frequencies: 1, 5, 10 and 20 Hz.](image)

In every case, a monotonic increase of \(\tan \delta\) values, with increasing frequency was observed. Thus, the first maxima experienced a \(283\%\) total increase, from \(0.012\) to \(0.034\), and the second maxima a \(215\%\) increase, from \(0.02\) to \(0.043\). The temperatures of the two maxima increased with frequency, except for a small fluctuation, up to \(15\) K, observed at the first maximum when the frequency increased from \(1\) Hz to \(5\) Hz and at the second maximum when changing the frequency from \(5\) to \(10\) Hz. The variations of storage modulus with temperature are presented in Figure 2. It is known that a high internal friction peak \((\tan \delta)\) [9] and a step-like modulus \((E')\) variation with obvious modulus increase \((\Delta E')\) [10] are associated with the hcp \(\rightarrow\) fcc transition, in the case of Fe-Mn-Si-based SMAs [11]. In our case, storage modulus increase \(\Delta E'\), corresponding to a modulus hardening, is only observed in the case of the second maxima, which is associated with Néel temperature.

Storage modulus hardening, reaching several GPa, varies with frequency. Thus, when using different frequencies the following \(\Delta E'\) values were obtained: for \(1\) Hz, \(\Delta E' = 2.69\) GPa, for \(5\) Hz \(\Delta E' = 2.59\) GPa, for \(10\) Hz \(\Delta E' = 2.44\) GPa and in the case of \(20\) Hz \(\Delta E' = 2.55\) GPa.
Figure 2. 3D variation of storage modulus (E’) recorded during heating on a hot rolled Fe-28Mn-6Si-5Cr using four different frequencies: 1, 5, 10 and 20 Hz.

Strain sweeps
In order to emphasize the effect of strain amplitude on the mechanical behavior of our samples, strain sweeps were performed at three different temperatures using the frequency of 1 Hz. The results are presented in Figures 3 to 8.

Figure 3. Tan d variation, comprising five cycles, recorded during strain sweep performed at room temperature.
Figure 4. Storage modulus variation, comprising five cycles, recorded during strain sweep performed at room temperature.

Figure 3 and 4 present the variation of internal friction and storage modulus during strain sweeps performed at room temperature, 296 K. As can be seen from Figure 3, the sample presents a maximum of internal friction of about 0.054, in the first cycle. This behavior is not observed in the following cycles, meaning that the maximum was influenced by the hot-rolling process that the sample was subjected to. Increasing the number of cycles lead to a stabilization of the internal friction to around 0.015 with no obvious maxima.

Figure 5. Tan δ variation, comprising five cycles, recorded during strain sweep performed at $T_2 = A_{50}^5$. 
The same tendency is observed in Figure 4, where the variation of storage modulus at room temperature is presented. The first cycle displays a different behavior than the rest and with increasing the number of cycles, it seems that the sample presents a storage modulus increasing tendency.

**Figure 6.** Storage modulus variation, comprising five cycles, recorded during strain sweep performed at $T_2 = A_{50}^\varepsilon$.

The variation of internal friction and storage modulus, during the strain sweeps performed at $T_2 = A_{50}^\varepsilon$, are presented in Figure 5 and 6. In this case, the maximum strain amplitude only reached 0.032 as opposed to 0.042 reached at RT that means that the maximum applied stress of 12 N was reached sooner at $T_2$ temperature. Tan d variation, at this temperature, presented a very scattered behavior that can be due to the structural changes in the material related to martensite reversion to austenite.

**Figure 7.** Tan d variation, comprising five cycles, recorded during strain sweep performed at $T_3 = T_N$. 
Figure 8. Storage modulus variation, comprising five cycles, recorded during strain sweep performed at \( T_3 = T_N \).

Although tan δ variation is very scattered it seems that increasing the number of cycles lead to a decrease of internal friction, the lowest variation being obtained in the 5th cycle.

Storage modulus variations, presented in Figure 6, displays a similar curve shape to that from RT sweep but larger variations from cycle to cycle can be seen in this case with the highest storage modulus obtained in the 1st cycle and the lowest in the 2nd one.

Internal friction variations at Néel temperature are presented in Figure 7 where a similar curve shape to that obtained at RT can be seen, although in this case the variations are from 0.023 to 0.03 compared to 0.015 obtained at RT.

Figure 8 present the storage modulus variation at \( T_3 \) and although no clear tendency can be observed at this temperature, the highest initial values were obtained of approx. 145 GPa compared to approx. 131 GPa at \( T_2 \) and 27.5 GPa at RT.

4. Summary and conclusions

An Fe-28Mn-6Si-5Cr (mass. %) SMA was subjected to DMA cycling as a function of temperature and amplitude at constant temperature. The sweeps were performed at three temperatures: \( T_1 = \text{RT}, T_2 = A_{fo}^\epsilon \) and \( T_3 = T_N \). During temperature scans, a modulus increase with 2.69 GPa was observed on heating and two internal friction maxima, the first attributed to the reversion of \( \epsilon \) martensite \( \rightarrow \gamma \) austenite and the second to Néel temperature of antiferromagnetic-paramagnetic transition. In addition, internal friction increased with increasing the frequency, in contrast to similar data presented by other authors where a decreasing tendency was noticed [7], which makes this material a promising candidate for earthquake dumpers. Increasing the temperature of the strain sweeps lead to the obtainment of a 0.023-0.030 internal friction values at \( T_3 \) while at \( T_2 \) the variations were very scattered due to the structural changes in the material related to martensite reversion to austenite. At RT only a value of 0.015 was obtained with a peak of 0.054 in the first cycle.
5. References
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