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Effect of magneto-mechanical cycling on 10M Ni-Mn-Ga magnetic shape memory material

I. Aaltio[1], Y. Ge[1], X.W. Liu[1], J. Tellinen[2], O. Söderberg[1] and S.-P. Hannula[1]
[1]Helsinki University of Technology, Department of Materials Science and Engineering,
P.O. Box 6200, FI-02015 TKK, Finland
[2]Adaptamat Ltd., Yrityspiha 5, FI-00390 Helsinki, Finland

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Abstract

Martensitic Ni-Mn-Ga alloys with the five layered modulated structure (10M) provide a way to produce motion by the magnetic shape memory (MSM) effect. MSM actuation is based on alternating the twin structure by applying a magnetic field and/or mechanical stress at suitable ranges. For the actuator use, it is necessary to understand the long-term cyclic magneto-mechanical loading behavior of the actuator material. To evaluate the long-term behavior of the materials martensitic 10M Ni-Mn-Ga single crystal samples were studied in the magneto-mechanical long-term cycling tests under constant pre-stress, using a specifically designed laboratory test platform. A decrease in the magnetic-field-induced strain was observed as the number of cycles accumulated. Crack formation followed the extensive cycling. Growth of the cracks on the specimen surface due to cycling was monitored and the microstructure was studied at certain cycling intervals. The final structure of the cycled specimen was observed by XRD and SEM to detect the possible fatigue induced changes in the microstructure.

Introduction

Ni-Mn-Ga magnetic shape memory (MSM) alloys are potential actuator materials for many applications, due to high actuation strains and frequencies typical of these materials. The 10M (also called 5M) five-layered tetragonal martensite Ni-Mn-Ga alloys have been a research topic in numerous scientific publications [1, 2]. These alloys suitable for MSM use, because they can be actuated by moderate magnetic fields. In the most potential actuator applications, the performance of the MSM material at increasing number of cycles is of great importance. Previous experiments suggest that MSM actuated Ni-Mn-Ga alloys outperform the conventional (thermally actuated) shape memory alloys in withstanding the cyclic actuation [3-5]. However, there are still lack of test data on the behavior of the material in long-term actuation. According to previous studies, the cycling of the material has resulted in a decrease of the magnetic-field-induced strain (MFIS) and in crack growth. Also, a large deviation in the fatigue life of different samples has been observed. It has been proposed that part of the material is changed to state in which MSM is no-working, resulting in the decrease of MFIS [3]. To shed light on this matter, more extensive experiments are necessary. A simple magnetomechanical MSM actuator operating with a pre-stress spring [6] is suitable for many applications. The actuating element in this type of actuator experiences magneto-mechanical straining and contraction cycles in the back-and-forth strokes of its operation. In the present work, changes in the MSM material properties and structure due to magneto-mechanical cycling were studied.

Experimental

First cycling test

Single crystal Ni-Mn-Ga samples, approximately 1 x 2 x 12 mm^3, were obtained for the experiments from Adaptamat Ltd., Finland. Their surface was wet ground with grit 1200 SiC-paper and electropolished in 25 % HNO_3-ethanol solution. The chemical composition was $Ni_{50.5}Mn_{28.0}Ga_{21.5}$ established with WDS analysis on LEO-1450 scanning electron microscope. The orientation of the samples as well as their crystallography was confirmed with X-ray diffraction (XRD) measurements (Philips X'Pert MRD with Co-tube). Transition temperatures of the studied material were determined by low-field AC susceptibility method (M_s 306.8, M_f 305.5, A_s 310.6, A_f 312.4, T_c 376.0 K). A magneto-mechanical (M-M) test platform (Figure 1) was designed and constructed. It consisted of a twin coil electromagnetic circuit with a central yoke gap at right angle to the magnetic field direction, a sample holder in the gap with a moving shaft (both made of brass), and a steel pre-stress spring (compressing the sample with σ=1.0 MPa). The bipolar drive current (max. amplitude 1.3 A) varied sinusoidally. It produced a transversal alternating magnetic field changing between positive and negative values, resulting in the straining of the sample with 30 Hz frequency. The magnetic field peak value was first 0.56 T (from 0 to 55 million cycles (Mc)) and it was increased to 0.65 T (from 55 Mc on) in order to produce more strain. The magnetic field values were measured as function of the coil current in the sample's air gap. The engineering strain values were calculated from the stroke and the sample length at contracted (near-single-variant) state.

This M-M cycling tests was stopped at specific times, to inspect the sample with a polarized optical microscope (Leica DMRX) and scanning electron microscope (SEM LEO 1450) as well as to measure the magnetic and the free-strain magnetic-field-induced strain (MFIS) values. MFIS is defined as the relative value of the sample length difference, after magnetizing the sample transversally and longitudinally by 1.0 T applied field at room temperature. Before the studies, the sample was cleaned with ethanol. MFIS was measured using a caliper. Magnetic properties were studied with a laboratory-made vibrating sample magnetometer (VSM) at room temperature. The sample was magnetized with 1.2 T perpendicularly applied field before each VSM measurement. The magnetization of the sample was measured before cycling, along (parallel) and perpendicular to the stick-shaped sample's wider side surfaces.

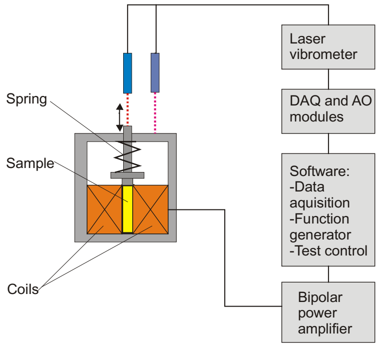


Figure 1. Test setup. Temperature as measured 4 mm below the sample was 300 ± 2 K during the test.

Second cycling tests

Another set of magneto-mechanical tests was conducted at Adaptamat Ltd. with a different test setup, in which the M-M testing device had, a similar pre-stress-spring-type operating principle as the actuator at TKK. It was also run with controlled coil current amplitude and the frequency of the magnetic field was in range of 90-150 Hz. The composition of the sample material was measured by EDS to be $Ni_{49.9}Mn_{28.4}Ga_{21.7}$ and the sample size was 1.0x2.5x16.0 mm^3. Preparation of the sample was done in the similar way as explained above. During the test, the sample was not removed from the system, and displacement was measured with a laser-interferometer sensor from the test jig push rod. The twinning stress values were calculated indirectly from the testing device actuator test results (a measurement principle developed by Adaptamat Ltd.). This method allows twinning stress monitoring and estimation during actuator use. Here, the displacement vs. current cycle of the actuator is recorded (Figure 2) for quasi-static operation.

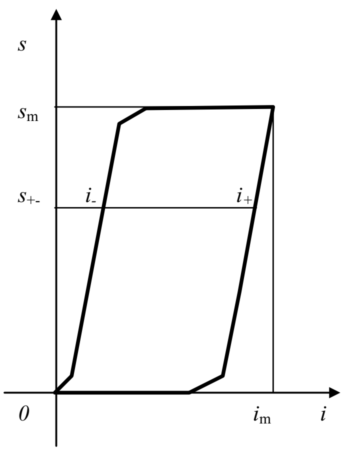


Figure 2. Schematic representation of the displacement *(s)* vs current *(i)* cycle of the MSM actuator.

In the cycle, the specific points shown in Figure 2 (s_{+-}, i_- and i_+) are determined. The point s_{+-} should be selected as close as possible to the value that corresponds to the $\varepsilon_0/2$. Using them, the twinning stress value σ_{tw0} follows from Equations 1 to 5,

$$H_S = \frac{B_{iS}}{(\mu_{hd} - 1)\mu_0}; \qquad (1)$$

$$E_a = \frac{B_{iS} H_S}{2}; \qquad (2)$$

$$\sigma_a = \frac{E_a}{\varepsilon_0}; \qquad (3)$$

$$i_S = \frac{l_\delta H_S}{N}, \qquad (4)$$

$$\sigma_{tw0} = \frac{\sigma_a}{2}\left(\frac{i_+ - i_-}{i_S}\right)\left(2 - \frac{i_+ + i_-}{i_S}\right) \qquad (5)$$

where:
H_S is magnetic field strength at which easy direction and hard direction magnetization curves cross each other;
μ_{hd} is relative permeability of the MSM material along the magnetic hard axis;
μ_0 is absolute permeability of the vacuum ($4\pi \times 10^{-7}$ H/m);
E_a is anisotropic energy density of the MSM material;
B_{iS} is saturated value of the intrinsic flux density;
σ_a is the maximum anisotropic magnetic-field-induced force density of MSM material;
ε_0 is free strain of the MSM material;
l_δ is effective value of the gap in which MSM material element is located;
N is turn number of the actuator coil.
Numerical values for the material properties can be found in, e.g., [7]. The surface of the sample was studied by SEM before cycling to find pre-existing defects and cracks and after the cycling for detecting their development during the cycling.

Results

It was confirmed with XRD pole figures that the crystallographic orientation of the sample surfaces in the first M-M- cycling test deviated approximately 6° from the (100) plane of the martensite within the cubic phase coordinate system. The θ-2θ XRD proved the 5-layered (10M) martensite structure. Some spherical inclusions were found, typically of 20 µm diameter, originating from the manufacturing of the material. A few pre-existing crack-like defects were found, with 5 to 40 µm length, mainly close to the [100] and [001] directions of the martensite.
Figure 3 shows the development of the peak-to-peak stroke of the M-M test system (TKK), converted to relative strain of the sample, as a function of number of cycles. In the stroke values, the sample was under prestress. Statically measured MFIS values at no prestress are also inserted to figure (squares connected with the dashed line). A clear decrease of MFIS was observed in the beginning (before 7 Mc), but after that the MFIS was relatively constant.
The sample could slide sideways during cycling in the sample holder causing eventually mechanical contact between the sample (a dynamic surface) and the magnetic circuit (a static surface), resulting in wear of the sample surface (Figure 4). The scratch marks caused by this wear were inducing microcracks during the cycling (Figure 5). The trace of these microcracks was analyzed by comparing the SEM image to the measured crystallographic orientation (XRD) of the scratched and microcracked area. The studies showed that the cracking took place close to [011] (in seven microcracks) and [001] (in one microcrack) directions. It was

confirmed with the SEM inspections that the pre-existing surface cracks did not evoke crack growth in the same way as the surface scratches.

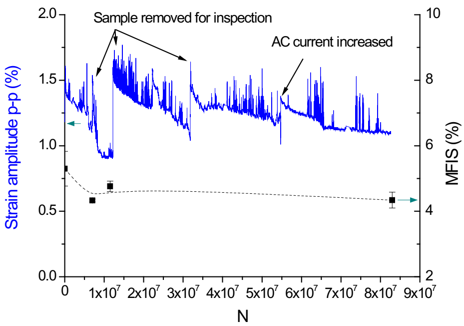
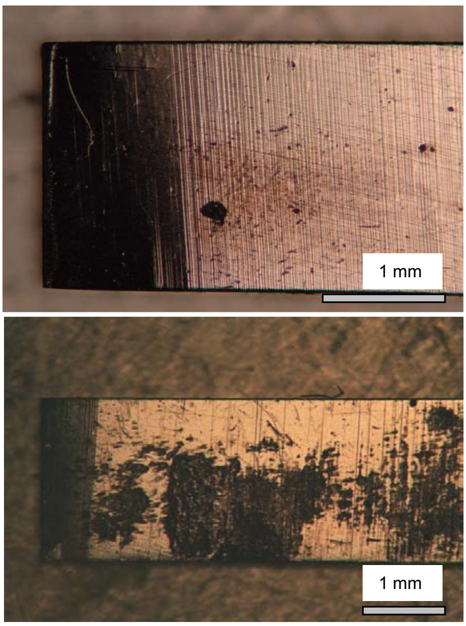


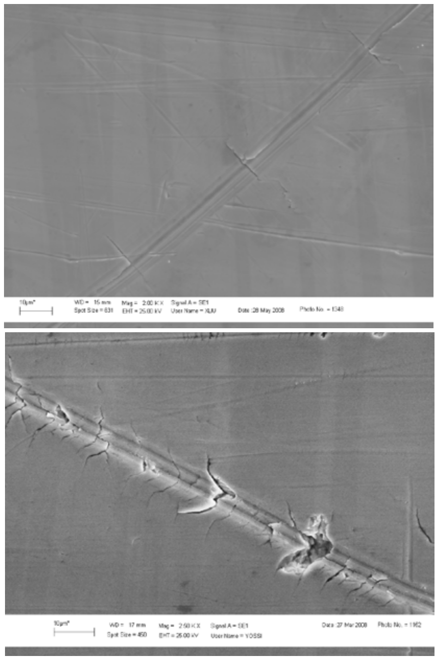
Figure 3. Strain (continuous line) and MFIS (squares with dashed line) as a function of straining cycles N. After 83 million cycles the test was stopped.



(a)

(b)

Figure 4. Optical microscope image of the sample surface (a) after 7 and (b) 83 million cycles, showing the wear of the surface.



(a)

(b)

Figure 5 (a) A SEM image of the scratched area after 83 million cycles, showing microcracks. The long axis of the sample is in the horizontal direction.(b) A scratched area of another test sample after 200 million cycles (right).

Results of the VSM measurements in the field direction parallel to sample, at free stress, are shown in Figure 6. The characteristic “step” due to reorientation of the martensite twin structure starts at 0.14 T applied parallel field (switching field e.g., the necessary magnetic field for moving the twin boundaries), while in the perpendicular direction, the MSM step started at 0.27 T (perpendicular curve not shown). After 7 million cycles (Mc) the reorienting field in the parallel direction increases. However, the curve after 83 Mc resembles more the non-cycled sample than that measured at 7 Mc.

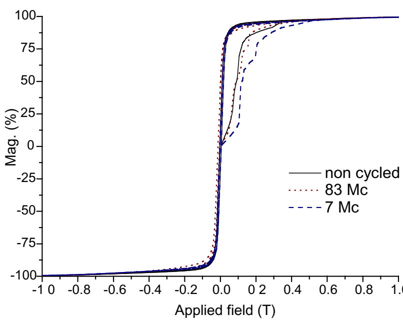
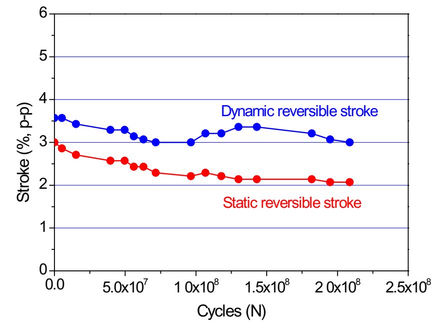


Figure 6. Magnetization curves of the non-cycled sample, after 7 and 83 million cycles.

Results of the second cycling test

Results of the AdaptaMat cycling test up to more than 200 million cycles are shown in Figure 7. In Figure 7a, the lower curve describes the static case (test was then paused and the coil current slowly increased and decreased), whereas the upper curve shows the dynamic reversible stroke of the sample. A decrease of the static stroke is strongest from 0 to about 20 million cycles, and it has a stabilizing trend. The dynamic stroke shows a decrease from about 3.5 to 3.0 % in the entire testing up to over 200 million cycles. In Figure 7b, is shown the development of the twinning stress for the tested sample.

(a)



(b)

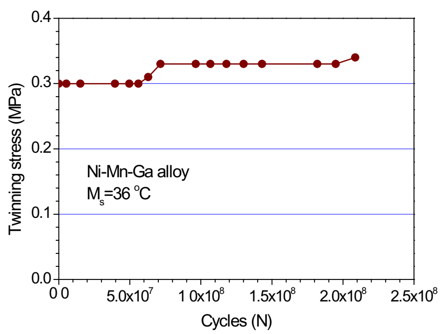


Figure 7 (a) The stroke of the actuator as a function of magneto-mechanical cycles. (b) Results of the twinning stress measurements of the cycled sample (Adaptamat test).

Discussion

It is obvious that the mechanical contact between the sample and the magnetic circuit caused friction and affected to the observed decrease of the peak-to-peak strain (Figure 3). This mechanical contact occurred to larger extent in the TKK test assembly than in the Adaptmat test, due to the slightly different constructions. While the specimen was taken out and reinstalled in the TKK study, the friction was decreased, and the majority of the MSM strain was recovered. This suggests that the mechanical contact have a significant contribution to the observed relative stroke. In the results of the AdaptaMat test (Figure 7a), the decrease of the static stroke is strongest from 0 to about 20 million cycles, and it has a stabilizing trend. Here, the local changes of the twinning stress in the sample would occur at those places, which are touching static surfaces and, thus, experiencing friction. This contact would perhaps only appear at relatively small area, near moving end of the sample. Wear is highest in the beginning, when the uneven surfaces are in contact. The observed decrease of the MFIS supports this assumption (Figure 3). Later on, the sample area already in contact with the static surface would support the sample, and the worn area would not increase at high rate as before, resulting in the stabilizing trend. The dynamic stroke decreases from about 3.5 to 3 % during the entire testing up to over 200 million cycles. Here, the stroke decrease is larger than in the case where the sample was removed in between the cycling, and the mechanical friction was less. The calculated twinning stress increased from 0.30 to 0.33 MPa in the cycling (Figure 7b). The twin boundary movement could be hindered by the microcracks (and possible twin variant structure disturbance) or mechanical wear of the sample (Figure 5). The growth of the microcracks at a scratched site was found to be in most cases near the [011] direction at the surface, which is an indication that the crack occurred at twin variant boundary. [011] direction is trace direction of twin boundary (011) on the (100) plane.,. Based on these observations, the crack is very likely to be on the (011) plane. If the direction of the straining is considered, this corresponds with the direction of the maximum resolved shear stress, which suggests that the crack growth mechanism would be related to twinning/detwinning in addition to stress state, corresponding with the maximum resolved shear stress. . The increase of the twinning stress is in accordance with the higher switching field observed after 7 million cycles (Figure 6), However, the decrease of the switching field again after 83 million cycles can be partly or wholly due to differences in the twin variant structure. Another reason may be related to possible difference of supporting stress of the sample holder. Since the twinning stress of the material is low, small stresses can have an influence, despite that the samples were carefully attached. Since the magnetic measurements and the twinning stress measurement are of different experiments, conclusions of their relation can not be directly made.

Conclusions

In the magneto-mechanical cycling for 10M martensite Ni-Mn-Ga samples with two test procedures, the samples were magnetically elongated and mechanically contracted up to over 200 million cycles (Mc) without failure. A decrease of the dynamic stroke had the magnitude of 0.5 % relative to the sample length in both tests. An increase of 10 % in the twinning stress was found when cycled about 200 Mc. The microstructural examinations of the sample surfaces showed that microcracks were developed in the cycling at the locations of the pre-existing surface scratches of the sample. The actual effect of the mechanical wear of MSM material surface will be studied in more detail in further studies.

Acknowledgements

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