Surface relief and domain structure of ferromagnetic shape memory alloys

2015 IOP Conf. Ser.: Mater. Sci. Eng. 77 012045

(http://iopscience.iop.org/1757-899X/77/1/012045)

Surface relief and domain structure of ferromagnetic shape memory alloys

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Abstract. A study is made of the surface corrugation during thermal cycling of ferromagnetic shape memory alloys (FSMA). This specific feature is a property of FSMA alloys and is a consequence of martensitic phase transformations not necessarily connected with surface defects of the material. The surface relief structure was studied together with martensite and magnetic domain structure changes during thermal cycling of the samples with the aid of differential polarized light microscopy. The analysis was facilitated making use of auxiliary reference grids applied to the surface of the samples.

1. Introduction

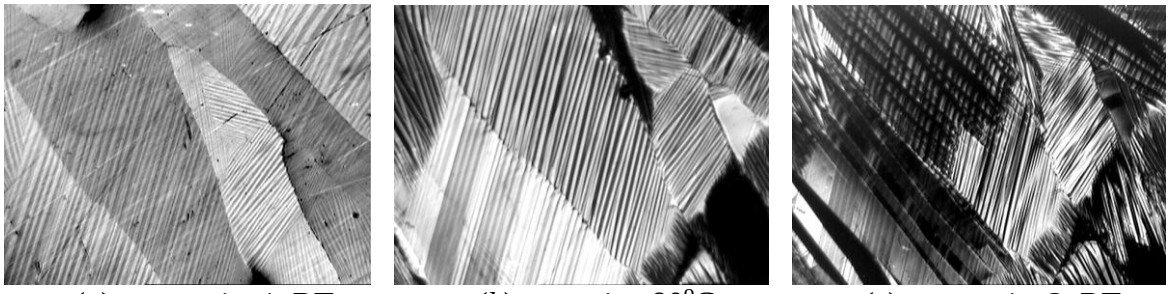
In recent studies [1-2] it was shown that the structural, thermal, magnetomechanical and mechanical properties of ferromagnetic shape memory (FSMA) alloys may be substantially affected by defects at or near the surface introduced by different surface treatments such as abrasive grinding, spark eroding, wire cutting. In the present work we examine another type of surface effects manifesting themselves in surface corrugation during thermal cycling of initially planar FSMA samples [3]. This specific feature is inherent to FSMA alloys and is basically a consequence of martensitic phase transformations not necessarily connected with surface defects of the material. The key experiments were performed by examination of the surface relief structure and magnetic domain structure during thermal cycling with the aid of differential polarized light microscopy.

2. Experimental

2.1. Corrugation of planarized surfaces of bulk samples

The experiments have revealed a very specific behaviour of FSMA samples during the metallographic preparation of planar polished surfaces. Normally the planarization is performed at room temperature when the samples are in martensite (M) state (figure 1(*a*)) (faint images of M twins are due to optical contrast in polarized light). Heating the planarized samples to temperatures above the phase transition point when they transform into the cubic austenite phase (A) causes a corrugation of the planar surface (figure 1 (*b*)). The corrugation remains upon cooling back to martensite (figure 1(*c*)); now the contrast

of the twin structure becomes much stronger due to the effect of relief. Two additional heating/cooling cycles (figure 2) have shown that the recovery of the martensite structure is not full so that each time the structural details undergo changes.



(*a*) martensite 1, RT (*b*) austenite, 90°C (*c*) martensite 2, RT

Figure 1. Microstructure of the polycrystalline $Ni_{2.19}Mn_{0.81}Ga$ sample in initial planarized state (martensite 1), heated to 90°C (austenite) and cooled back to RT (martensite 2)

martensite 3, RT martensite 4, RT

Figure 2. Residual martensite structures after two additional heatings to 95°C

During further experiments we studied the surface relief of the samples for which the standard metallographic procedures (lapping, grinding, polishing) were applied at a temperature above the phase transition point, i.e. when the samples acquire the A state. It was found that although corrugation occurs upon cooling of these samples to M state the repeated heating allows to restore the planarized surface.

Figure 3 shows the motion of the A/M phase boundary from right to left toward the cold side of the sample (see the arrow) under the action of adjustable temperature gradient produced by a differential pair of Peltier elements. It is seen that on passing across the sample the initially corrugated A/M boundary leaves the uncorrugated surface behind. This process is quite reproducible and may be repeated many times.

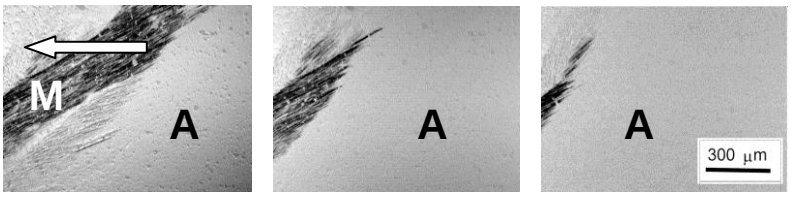


Figure 3. Video sequence showing the phase boundary motion at the surface of $Ni_{2.12}Mn_{0.88}Ga$ sample under the action of temperature gradient. Sample was planarized at 80 °C

2.2. Surface relief of thin film

As distinct from bulk samples, in film specimens the repeatibility of the structural details is much better. Figure 4 illustrates the direct and converse transformation process in a $Co_{48}Ni_{22}Ga_{30}$ thin film prepared by RF magnetron sputtering (thickness ~10 μm) starting from martensite up to austenite and back. Comparison shows that the final martensite structure coincides in detail with the starting one. Presumably this may be explained by the restriction of the degrees of freedom imposed by the large (>10) ratio of grain size/film thickness.

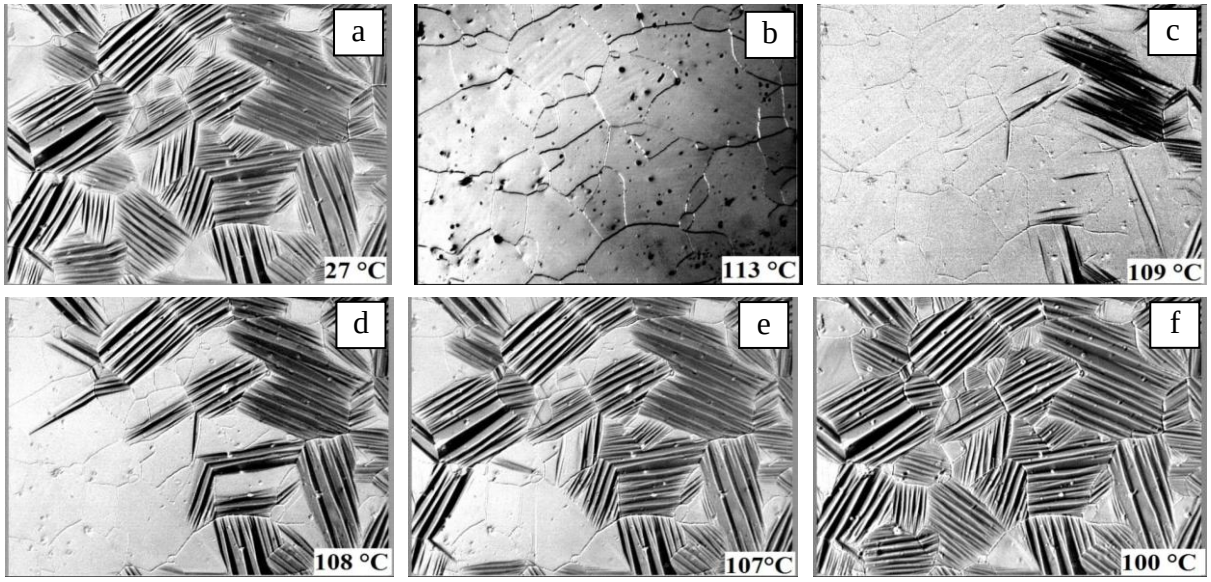
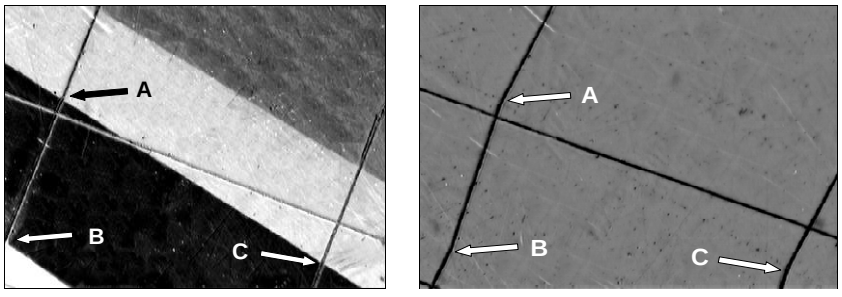


Figure 4. Dynamics of the martensite transformations of a $Co_{48}Ni_{22}Ga_{30}$ thin film. (*a*) starting point at RT (100% martensite); (*b*) 100% austenite state at 113 ^{o}C; (*c*), (*d*) and (*e*) – intermediate states; (*f*) – final return to 100% martensite

2.3. Observation of local deformation

Optical observations provide a possibility to perform quantitative measurements of local deformations caused by martensitic transformation. To this end the sample is supplied by some kind of reference marks. In the present work we used a diamond indentor of a microhardness testing instrument to apply a rectangular grid of light scratches on the sample's surface. Figure 5(*a*) shows these scratches applied at RT when the sample is in martensite state.



(*a*) (*b*)

Figure 5. Microstructure of $Ni_{2.16}Mn_{0.84}Ga$ FSMA alloy observed at RT (martensite state) (*a*) and after transformation into the austenite state at T = 370 K (*b*). The arrows show the points of martensite twin boundary intersections with a square grid of scratches in (*a*) and the points of the grid inflection in (*b*)

By using polarized light we were also able to observe the martensite twins. On passing to the austenite state by heating the polarized contrast of the martensite disappears because the cubic austenite phase is optically isotropic. The deformation is characterized by the changes in the geometry of the grid by the kink of scratch lines in characteristic points of intersection with the martensite boundary labeled as A, B, and C in figure 5. Comparison of the two images provides quantitative information on the localized deformation values of the material during martensitic transformation.

2.4. Absorption and desorption of liquids at the surface of FSMA

Another unusual feature of the FSMA was discovered during examination of metallographically processed samples. It was found that heating the sample polished at RT to a temperature above A/M transition (~80 ^{o}C) is accompanied by an appearance (desorption) of liquid drops with a diameter of 10 to 20 μm (figure 6). Surprisingly the effect appeared to be reproducible during at least 50 cycles of cooling and heating resembling the work of some kind of micropump.. The origin of the oil is definitely from the diamond polishing pastes which are prepared on the base of oil. The same behavior was observed with other polycrystalline FSMA compositions, in particular, $Ni_{2.19}Mn_{0.81}Ga$.

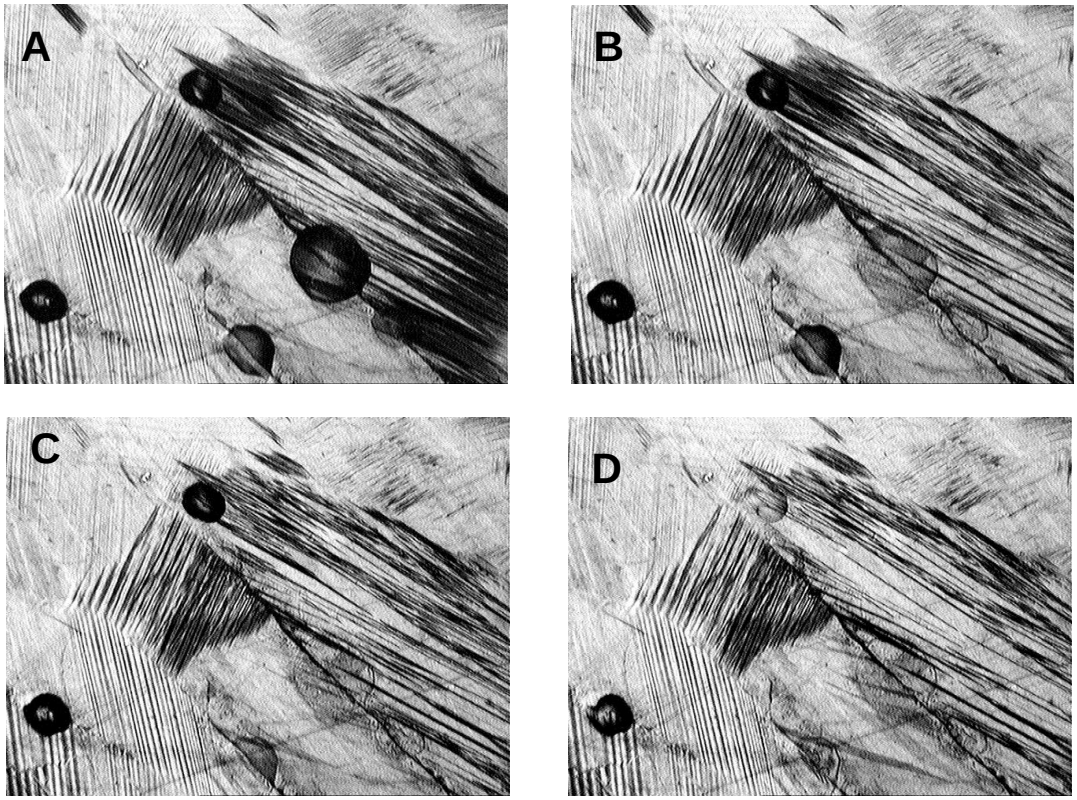


Figure 6. Desorption of four drops of oil at the grain boundaries of a heated polycrystalline $Ni_{2.16}Mn_{0.84}Ga$ sample heated to ~80 ^{o}C (A) and their one-by-one absorption during further cooling to 60 (B), 40 (C) and 22 ^{o}C (D). On next heating the drops arrive again one-by-one at the same places. The whole process may be repeated many times (up to 50 cycles were made in the experiment)

4. Conclusion

An experimental study is made of the effect of sample preparation on the surface relief of shape memory alloys of the Heusler family and its behaviour in the course of direct and converse martensite-austenite transformations. The transition from tetragonal twinned martensite to a cubic austenite causes a corrugation in the planar surfaces. Performing the planarization of the samples at elevated temperatures in the austenite state enables to conserve the planarity at this state, but reverse is not true for the samples planarized in the martensite state. In addition to the experiments on planarizing and ridging of the samples an unusual effect of reversible absorption and desorption of liquids by the surface of the specimen was found to exist. This effect may be useful for understanding the mechanical behaviour of shape memory alloys.

Acknowledgement

The work was carried out with financial support in part from the Ministry of Education and Science of the Russian Federation in the framework Program of NUST "MISIS" (No. K-2014-008) and RFBR Research Project No. 14-07-00888 A.

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