A statistical evaluation of thermomechanical loading effects on martensite plate morphology in CuZnAl SMAs

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The paper reports the effects of 100 to 500 training cycles applied to lamellar specimens of a Cu-Zn-Al Shape Memory Alloy (SMA). The training procedure consisted in the bending, during the cooling to room temperature (RT) of martensitic specimens, by the load fastened at their free end and in load lifting by Shape Memory Effect (SME) during heating up to austenitic domain. During cooling, the concave surface of bent specimens was compressed and during heating it was elongated, being subjected to tension-compression cycling, during heating-cooling, respectively. Conversely, the convex surface of bent specimens was elongated during cooling and compressed during heating, being subjected to compression-tension during heating-cooling, respectively. Considering the different loading regime of the convex and concave regions, specimens were trained under various numbers of cycles and were correspondingly prepared for analysis performed by means of atomic force microscopy (AFM). The recorded 2D and 3D micrographs were corroborated with statistical evaluations of thermomechanical loading in order to emphasize the effects of both the number of training cycles and the initial loading state (tension or compression) of the specimens on the morphology of martensite plates.

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1. Introduction

Shape memory alloys (SMAs) are presently found under five commercial systems: (i) Ni-Ti-based; (ii) Cu-Zn-Al-based; (iii) Cu-Al-Ni-based; (iv) Fe-Mn-Si-based and (v) Fe-Ni-Co-based) [1]. These alloy systems have in common the occurrence of simple shape memory effect also known as one-way effect (1WE) consisting in thermally induced reversion to parent phase (also called austenite) [2] of a stress-induced martensite phase [3]. It follows that, in SMAs, martensite can be thermally or stress induced during cooling under constant stress or during isothermal loading, respectively [4].

Cyclic functioning of SMAs requires the presence of two-way effect (2WE) consisting in spontaneous recovery of both hot (austenitic) and cold (martensitic) shapes during heating and cooling, respectively [5]. In order to obtain 2WE, SMAs must be subjected to a thermomechanical treatment called "training", consisting in repetitive passing through the same route in stress-strain-temperature space, until obtaining a closed path [6].

Due to the diversity of training routines, it is possible to vary both temperature and stress in the same time. Therefore one can no longer refer to uniquely thermally or uniquely stress induced martensite.

For instance, one of the most accessible training procedures, of soft martensitic SMA lamellas, involves repetitive lift and descent of a load, fastened at specimen's free end. Lifting is caused by work-generating 1WE and descend is due to the softening induced by direct martensitic transformation [7]. In such a case, the upper (convex) surface of bent lamellar specimens becomes elongated while the lower (concave) surface becomes compressed. During heating-cooling the former is subjected to compression-tension while the latter to tension-compression loads.

The aim of the present paper is to analyze the effects of different loading conditions of the convex and concave surfaces from the point of view of the morphology of martensite plates, by taking into consideration the influence of the number of training cycles.

2. Experimental procedure

Specimens of Cu-15 Zn-6 Al (mass. %) SMA were hot rolled to plates of 0.5 mm thickness, homogenized at 1073 K/ 18 ks and water quenched before being cut to lamellas with the dimensions 0.5×4×50 mm. As previously reported, by electrical resistance measurements during a heating–cooling cycle [8], by differential scanning calorimetry (DSC) scans [9] and by X-ray diffraction (XRD) patterns [10], the alloy under study was found to be martensitic at room temperature (RT).

Lamellar specimens, weighing approx. 0.8 g, were trained in bending with a load of 40 g, fastened at their free end, being subjected to 100, 300 and 500 heating–cooling cycles, respectively, as previously reported [7].

Atomic force microscopy (AFM) observations were carried out on etched elongated (convex) and compressed (concave) surfaces of the specimens using a NanoSurf easyScan 2 device equipped with silicon SPM cantilevers and easyScope video camera, at a scanning rate of 2×10-1 s per line. On AFM profiles, the widths and heights of martensite plates were measured with nanometer precision.

The measuring procedure, involves selecting five characteristic groups of primary plates with lengths greater than 50 micrometers, in different grains on the surface of each specimen under study and five parallel plates within each group. In order to evaluate each of the five plates, four profiles were measured at equal distance, d=10 micrometers, along the respective plate. On each profile, right-side and left-side heights, as well as top and bottom widths were measured, based on which the mean height and width was determined. These 100 width and 100 height values, for each surface, were used in order to illustrate the average profile of martensite plates, characteristic to each specimen and loading type. Finally, a statistical evaluation was performed by dividing the measured values of widths and heights into ranges of 100 nanometers.

3. Results and discussion

The first series of characteristic AFM micrographs, illustrating training-cycle effects on martensite plate morphology on the elongated (convex) surface of specimens is shown in Fig. 1.

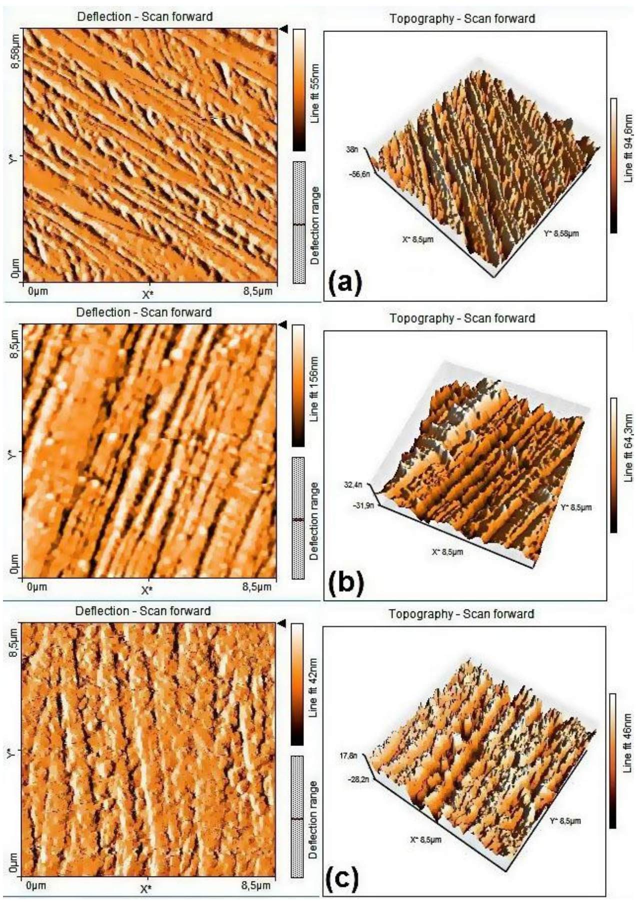

Fig. 1. Typical 2D and 3D AFM micrographs illustrating the effect of the number of training cycles on the morphology of martensite plates in elongated (convex) area of specimens, after: (a) 100; (b) 300; (c) 500 cycles.

The three micrographs reveal a gradual decrease of martensite plate heights which is more noticeable in 3-D images. With increasing the number of training cycles, from 100, Fig. 1(a) to 300, Fig. 1(b), the plates became more oriented and the surface relief decreased.

It is expectable that martensite plates with single orientation prevent the occurrence of intersected structures which have the potential to hinder their mobility, thus reducing the magnitude of 1WE. Finally, after 500 cycles, Fig. 1(c) shows highly oriented plates with the lowest surface relief.

Similar effects characteristic to compressed (concave) area of trained specimens are revealed in Fig. 2.

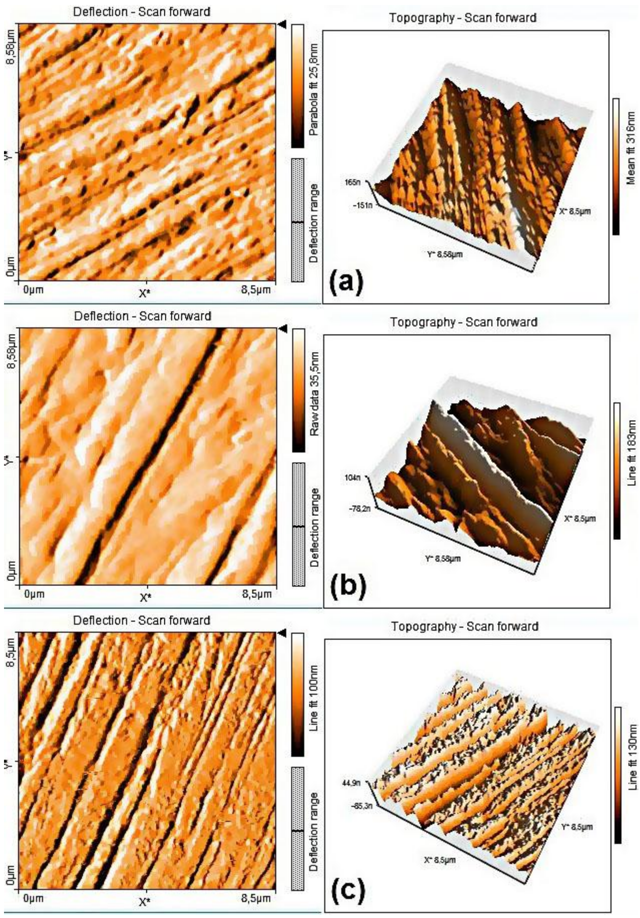

Fig. 2. Typical 2D and 3D AFM micrographs illustrating structural effects of the number of training cycles on the morphology of martensite plates in compressed (concave) area of specimens, after: (a) 100; (b) 300; (c) 500 cycles.

The difference between the effects of tensile and compression loading is evident by comparing Figs.1 and 2. In compressed area, the plates are both wider and deeper, however the refining tendency, caused by training cycles, is still noticeable.

For a more accurate comparison, quantitative measurements were performed as listed in Table 1.

Table 1. Minimum, maximum and average values of width and height of martensite plate profiles measured by AFM, for two loading modes.

Loading	No of cycles	Dimension (nm)	Min	Max	Mean
Elongation	100	Width	66	299	184
		Height	33	235	89
	300	Width	67	199	133
		Height	33	166	79
	500	Width	33	133	91
		Height	33	101	53
Compression	100	Width	268	830	529
		Height	34	603	272
	300	Width	214	583	346
		Height	34	302	165
	500	Width	105	214	165
		Height	34	201	111

The mean values in Table 1 represent the average of plate heights and widths, which were determined for each characteristic martensite plate profile.

In order to compare the two profiles from elongated and compressed areas, Fig. 3 provides a schematic illustration of the contours described by using the mean values of height and width of the plates, listed in Table 1.

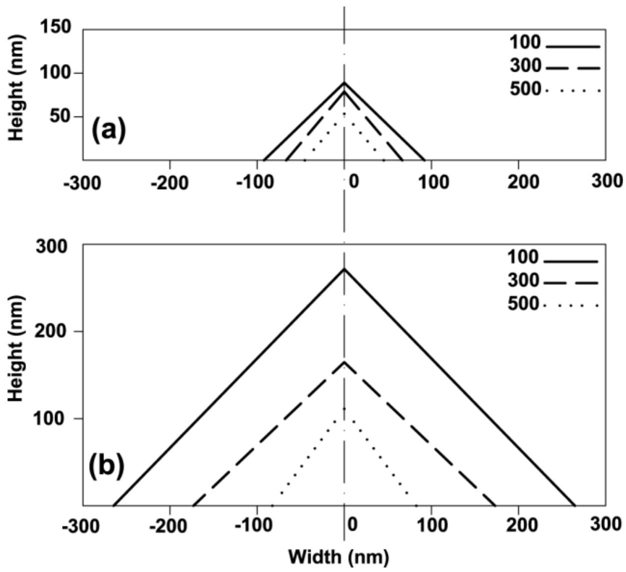

Fig. 3. Schematic illustration of the profiles, defined by average widths and heights of martensite plates, revealing training cycle effects on the areas subjected to different loads: (a) elongation; (b) compression.

Solid-line profiles correspond to the specimens subjected to 100 training cycles. It is obvious that average height increased from 89 nm in elongated area, Fig. 3(a) to 271 nm, in compressed area, Fig. 3(b). Likewise, average width increased from 184 to 529 nm, respectively.

After 300 training cycles, the average height and width, depicted with dash-line, increased from 78 and 133 nm to 164 and 346 nm, respectively.

Finally, the refining effect of 500 training cycles can be observed at the average height and width corresponding to dotted-line profiles of elongated area, which decreased to 53 and 91 nm, respectively, in Fig. 3(a). In compressed area Fig. 3(b) the corresponding average values have been 111 and 165 nm, respectively.

Therefore in both elongation and compression training contributes to the refinement of martensite plate profile. Plate profile refining by training cycles was also noticeable in compression area but to a lower intensity than in elongation area.

For a statistic evaluation of the effects of both training cycles and loading mode on martensite plate morphology, the 100 measured values of width and height of each specimen were divided into ranges of 100 nanometers and representative 3-D diagrams were built up, as shown in Fig. 4.

When comparing the widths of martensite plates in elongation area, Fig. 4(a) and compression area, Fig. 4(c), it is obvious that the dimension of the profiles diminished with increasing the number of training cycles. For instance, in the elongation area, 2% of plate widths were lower than 100 nm and 63 % between 100 and 200 nm after 100 training cycles. After 300 cycles, 12 % plate widths were below 100 nm and the rest of 88 % between 100-200 nm. Finally, after 500 cycles 70 % of plate widths were below 100 nm and 30 between 100 and 200 nm.

In compression area, these figures are summarized in the following. 100 cycles: 1 % widths between 200-300 nm and 9 % between 300-400 nm. 300 cycles: 45 % between 200-300 nm and 26 % between 300-400 nm. 500 cycles: 94 % plate widths between 100 and 200 nm and 6 % between 200-300 nm. Therefore plate widths are obviously larger in compression than in elongation area. A similar situation is observed when analyzing plate heights.

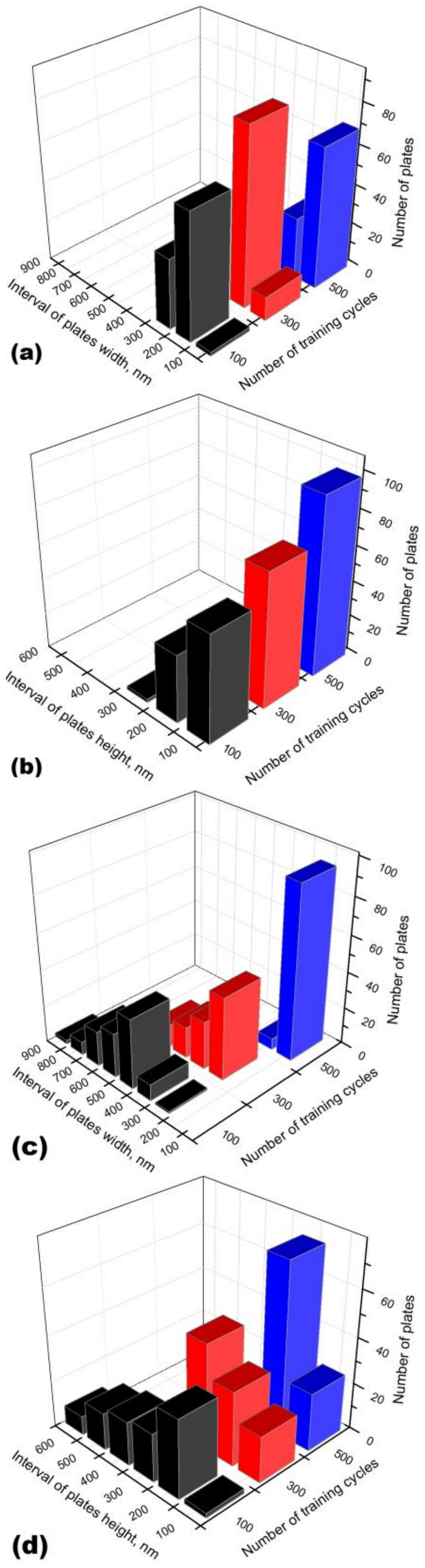

Fig. 4. Statistical evaluation of 100 AFM measurements of martensite plate profiles, illustrating the effects of loading mode and number of training cycles on: (a) width values in elongation area; (b) height values in elongation area; (c) width values in compression area; (d) height values in compression area.

4. Conclusions

The evaluation of martensite plate profiles on the elongated and compressed areas of trained specimens of Cu-Zn-Al SMA revealed a definite refining tendency with increasing the number of training cycles.

In all situations, martensite plate profiles were larger in compression than in elongation area of trained specimens.

After 500 training cycles martensite plate profiles were refined to nanometric domain (i.e. bellow 100 nm) in a proportion of up to 70 %.

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