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Tensile/compressive behaviour of non-layered tetragonal $Ni_{52.8}Mn_{25.7}Ga_{21.5}$ alloy

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Abstract

The stress–strain behaviour of the alloy $Ni_{52.8}Mn_{25.7}Ga_{21.5}$ with the non-modulated tetragonal martensite (T) crystal structure was studied below M_s temperature with stresses needed for the martensite variant structure reorientation, i.e. detwinning. At ambient temperature the stress plateau of the detwinning showed a total strain of 21–22% with 18–20 MPa stress. After the first cycle, the tensile/compression cycles were repeatable. With maximum strain the single variant structure was obtained which was verified with X-ray pole figures. The tension-compression cycling decreased the stress plateau to 10–12 MPa, while the increasing testing temperature lead to a value of 6.7 MPa close to the reverse transformation temperature.
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Keywords: Ni–Mn–Ga alloys; Tensile testing; Twinning

1. Introduction

The great interest in Ni–Mn–Ga alloys is due to the large magnetic-field-induced strain (MFIS) and the magnetic shape memory (MSM), effect which appears in some of its ferromagnetic martensitic phases [1–7]. MFIS is based on the rearrangement of the twin variants by the twin boundary motion. The variants with the easy axis of magnetization along the applied magnetic field grow at the expense of the other variants ultimately reaching the single variant state. The MFIS of Ni–Mn–Ga alloys is at maximum approximately 6% in 5 M martensite [2–6] and 10% in 7 M martensite [7]. The same shape change based on the rearrangement of the twin variants, i.e. detwinning, is also obtained by the mechanical loading. In the detwinning the martensite variants favourably oriented towards the applied force grow at the expense of the other martensite variants [8]. The detwinning is indicated by a stress plateau in a stress–strain-curve. The maximum strain (ε_o) in detwinning is obtained when the material is in single variant state and the magnitude of the strain depends on the crystal lattice of the alloy. For the tetragonal Ni–Mn–Ga alloys it is $\varepsilon_o = |1\text{-}c/a|$, where c and a are the lattice parameters of martensite indicated in the cubic parent coordinates [2]. The crystal lattice of Ni–Mn–Ga martensite depends strongly on the chemical composition. The most important martensitic structures are the five layered tetragonal or slightly monoclinic martensite (5 M), the seven layered orthorhombic or slightly monoclinic martensite (7 M) and the non-modulated tetragonal martensite (T) [for example refs. [9–11]. The 5 and 7 M martensites have a short c-axis. In the T martensite the c-axis is the long one. The maximum detwinning strain is about 6% in the 5 M martensite, 10% in the 7 M martensite and approximately 20% in the T martensite [12–15]. In compression the stress required for the detwinning, called the twinning stress (σ_{tw}), varies from less than 2 MPa for the 5 M martensite to 15–18 MPa for the T martensite. The value of σ_{tw} decreases with increasing temperature [14–16]. The twinning stress in compression of the T martensite reduces to 6.5 MPa when the reverse phase transformation temperature is

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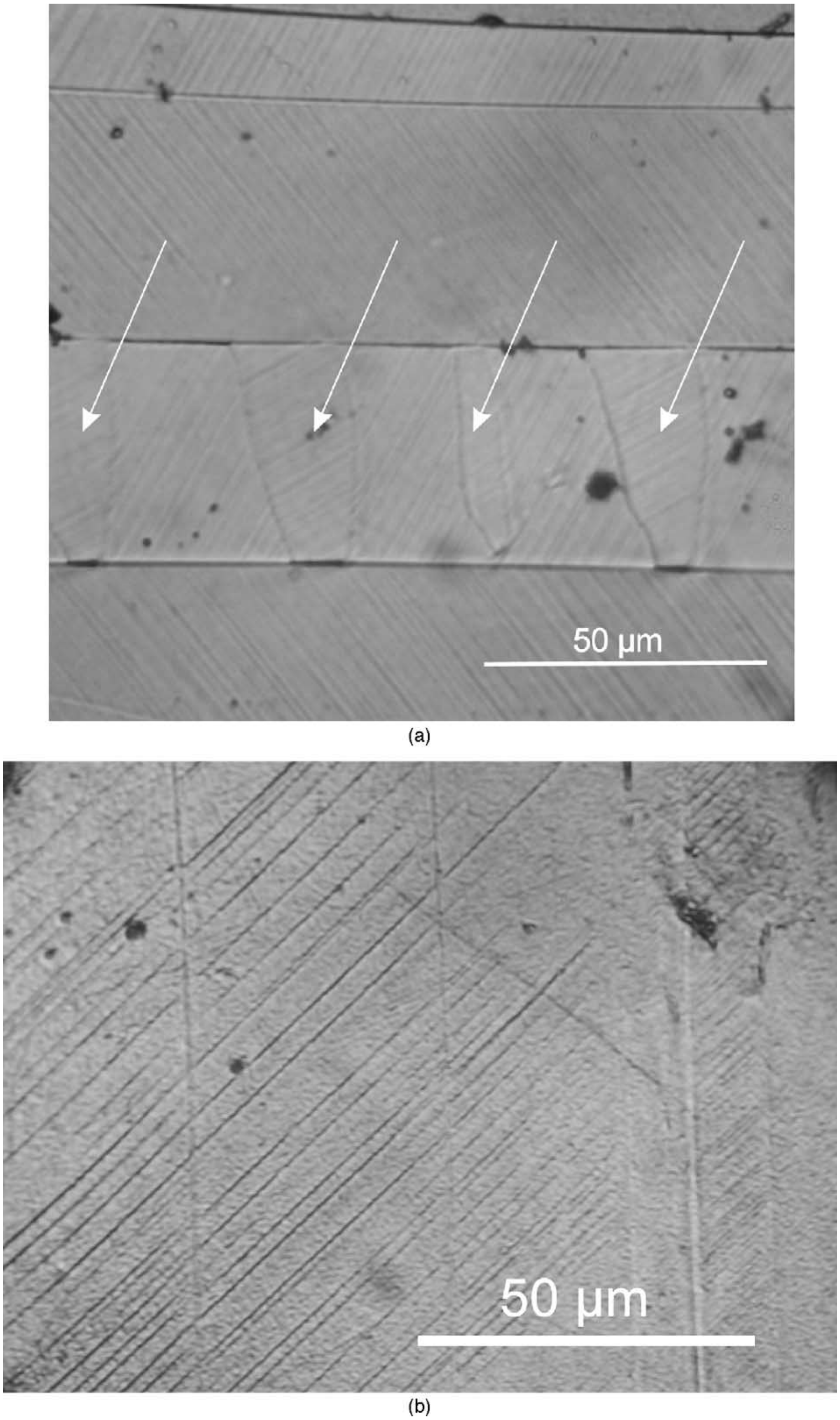

Fig. 1. The morphological structure of a non-modulated tetragonal martensite in the alloy $Ni_{52.8}Mn_{25.7}Ga_{21.5}$ at ambient temperature. (a) Twinned multi-variant martensite structure with smaller separate twin domains inside the larger twin marked with arrows. (b) The single-variant martensitic structure after tensile testing.

approached [15]. The MSM effect requires that the magnetically induced force exceeds the twinning stress of the material [17]. Consequently, this high value excludes the possibility for MSM, while the magnetically induced force (σ_{mag}) in the T martensite is approximately 0.7–1 MPa [15,18].

The tensile behaviour of T martensitic alloys has not been studied to a greater extent. Martynov and Kokorin [10] applied the tensile load to study the stress-induced phase transformations of a Ni–Mn–Ga alloy. The internal friction study [19] with a moderate tensile load confirmed the high mobility of the twin boundaries in two alloys with T structure. As the T martensitic Ni–Mn–Ga alloys possess good mechanical and chemical properties [14,20–22] and some of them have potential for the magnetocalorimetric applications due to the co-occurrence of the magnetic and the structural phase transition [23,24], it is important to obtain more information about their tensile behaviour. The present work studies the behaviour of the T martensitic alloy $Ni_{52.8}Mn_{25.7}Ga_{21.5}$ in the tensile/compressive loading.

2. Experimental

The present study was carried out with the non-stoichiometric $Ni_{52.8}Mn_{25.7}Ga_{21.5}$ alloy that was manufactured by AdaptaMat Ltd. with a Bridgman-type crystal growth furnace. The ingot was annealed at 1273 K for 48 h and at 1073 K for 72 h in a vacuum quartz ampoule. The chemical composition in at.% was determined with an energy-dispersive spectrometer (EDS) connected to a scanning electron microscope LEO-1450 SEM. The crystal structure together with the orientation of the ingot slice and the test specimens were measured using a Philips X'Pert X-ray diffractometer equipped with a Co-tube. The transformation temperatures and the Curie point were determined by the low field ac magnetic susceptibility and by the differential scanning calorimeter Linkam 600. The heating/cooling speed in the measurements was 2 K/min. Optical micrographs were taken with the back-scattered light using an Euromet microscope.

Samples for tensile tests were cut from the oriented slice of the ingot with spark cutting. They were wet ground and electropolished at ambient temperature in 100 ml H_2NO_3 + 300 ml ethanol electrolyte. The dimensions of the two single crystalline samples were approximately 3 mm^3 × 3 mm^3 × 30 mm^3 and their edges were nearly parallel to the [1 0 0], [0 1 0] and [0 0 1] directions of the martensite crystal lattice, presented in the cubic parent phase coordinates. Tensile tests were carried out at Institute of Physics, Prague, with an INSTRON 1362 mechanical testing machine. The gauge length in all cases was 9 mm, i.e. 21 mm of the sample length was in the grips. Straining of the samples was measured from the movement of the grips. Consequently, the strain amplitudes are slightly overestimated as the part of the sample inside the grips also transforms. The samples were heated with an electrical heating chamber and the testing temperature was measured with a thermocouple attached to the specimen surface in the middle of the gauge length. The temperature distribution over the sample length was rather uneven as the holding grips cooled the specimen ends.

3. Results and discussion

At ambient temperature the studied alloy $Ni_{52.8}Mn_{25.7}Ga_{21.5}$ has the non-modulated tetragonal martensite structure with the lattice parameters $a = b = 0.546$ nm and $c = 0.657$ nm (presented in cubic parent phase coordinates). The lattice distortion is $c/a = 1.203$. The morphological structure of the alloy with the larger twins consisting of a couple of separate internal twin domains is presented in Fig. 1a.

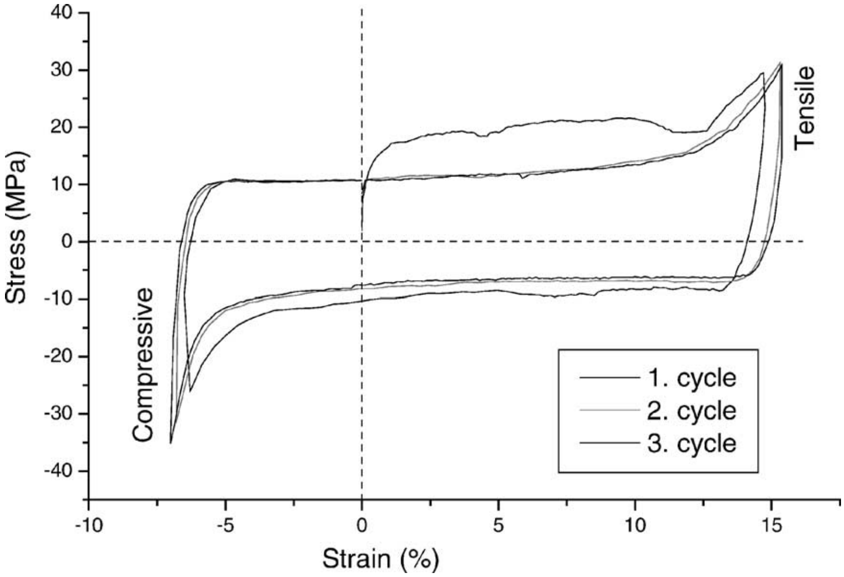

Fig. 2. The complete tensile/compression stress–strain cycles showing the detwinning of the martensitic structure.

The phase transformation temperatures determined with the DSC were for the martensitic transformation M_s = 390 K and M_f = 367 K, and for the reverse transformation A_s = 377 K and A_f = 404 K. The ac magnetic susceptibility measurements showed that the ferromagnetic transformation is gradual with the mean Curie temperatures in cooling T_{Cc} = 378 K and in heating T_{Ch} = 386 K. The hysteresis of the magnetic transition is explained in [12,23,24] due to the co-occurrence of the magnetic and the structural phase transitions.

The first three tensile-compression cycles of the material are shown in Fig. 2. The stress plateau of the detwinning appears in both the tensile and the compression loading. The tensile twinning stress values are slightly higher than those on the compression side, while the strain on the tensile side is larger than that of the compression side. During the first tensile-compression cycle, the stress values are clearly higher than in the latter ones and the curve is rather uneven. This is due to the multi-variant structure of the thermally formed martensite (Fig. 1a). When it is strained, the tetragonal martensite variant MV1 with its long axis along the stress direction starts to grow. At the beginning the stress needed for this growth is approximately 20 MPa while in the end the stress value is lowered to 18 MPa. When the single-variant state is obtained in the tensile deformation, the total strain is close to 14%. In the subsequent compression the stress plateau is decreased to 12 MPa indicating that the stress needed for changing the single variant structure of MV1 to another single variant is lower than the stress needed for thermally formed multi-variant structure to become a single variant. During compression MV1 variant gradually changes to another tetragonal martensite variant MV2 which has one of its short axes along the loading direction. The total shape change of 21% in the first tensile/compression cycle consists of the change of the multivariant structure to MV1 (14%) and the additional strain in the formation of the other single-variant state, MV2 (approximately 7%). The stress–strain curve during the second and third cycle has stabilized. It indicates that the sample is altering between the single-variants MV1 and MV2. The stress needed for the twin variant change is approximately 10.5 MPa in tensile loading and 6 MPa in compression. The full deformation is 22% including the 15% strain of the tensile side and the 7% change of the compression side. This exceeds to some extent the theoretical maximum, $\varepsilon_0 = |1-(0.657/0.546)|$ = 20.3%. The difference is due to the experimental inaccuracy caused by the measurement of the strain using the grip movement.

The twin structure of the sample was studied by X-ray diffraction pole figures before and after the tensile loading (Fig. 3a and b). The figures show the poles of the (4 0 0) and the (0 4 0) planes at ambient temperature. Fig. 3a before tensile testing shows clearly four points in the middle with a maximum deviation of 9°. This splitting may be caused by a small deviation from the tetragonality or it may indicate the existence of the low angle boundaries within the martensite twins. The peaks in the centre have the highest intensity. The lower intensity peaks on the edges confirm the existence of other variants. The angle between the variants is approximately 80°, which agrees well with the theoretical value $\delta = 2\,\mathrm{arc}\,tg(a/c) = 79.5^\circ$ using the lattice parameters of the martensite and assuming (1 0 1)[1 0 $\bar{1}$] the twinning system. After the tensile loading of 40 MPa (Fig. 3b) there is only one peak with a very high intensity. Its position is deviated 6° from the centre of the pole figure. This single peak suggests that the martensite has reached a true single

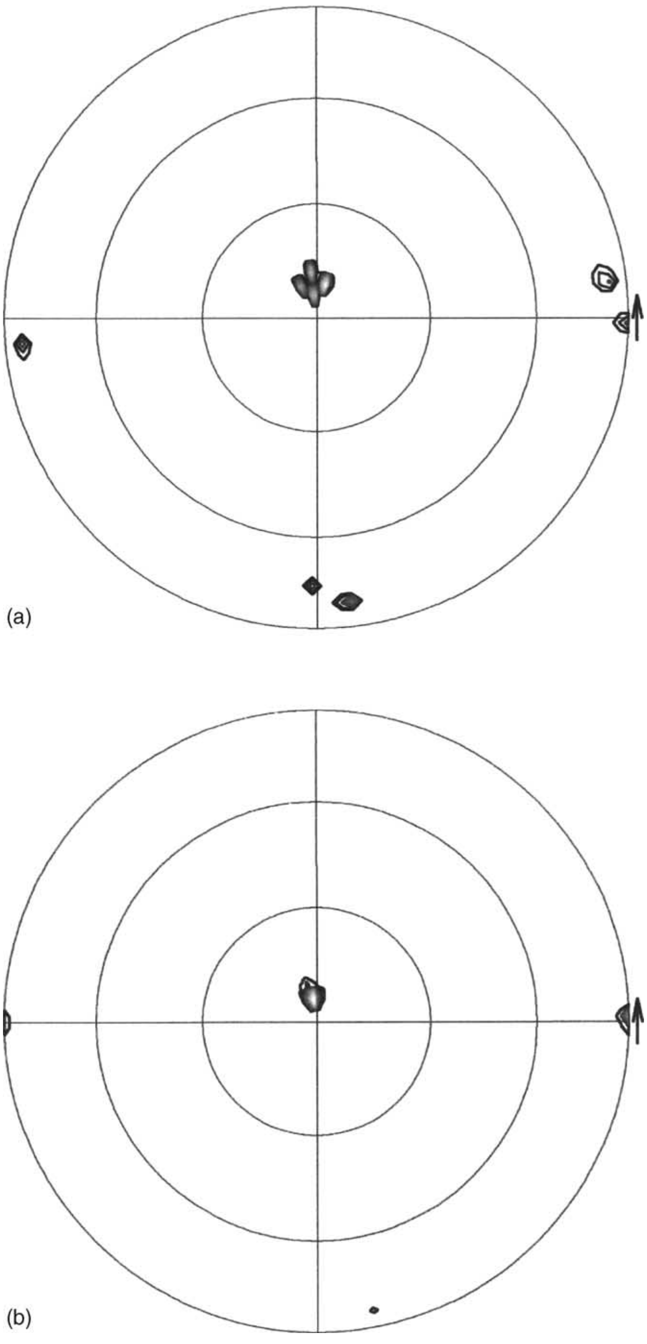

Fig. 3. The 2D pole figure of the (4 0 0) and (0 4 0) planes (lattice constant a_M) in Schmidt projection obtained by texture measurements. (a) The initial structure of the sample after cooling (transformation from parental phase) – the maximum intensity is 190 000 counts/s. (b) After the tensile deformation of the sample (40 MPa) – the maximum intensity is 520 000 counts/s.

variant state. According to literature, after compression the middle peak might be still to some extent split which indicates that with compressive loading it is not possible to eliminate totally all the irregularities of the structure. However, also Fig. 3b shows some very weak peaks at the bottom of the circle of the pole figure indicating the presence of very small amount of residual martensite variants. According to Fig. 3b, the observed martensite single variant has its plane (4 0 0) approximately perpendicular both to the sample surface and the tensile stress axis. The lattice parameters $a = b = 0.546$ nm and $c = 0.656$ nm are approximately the same with the original structure which indicates that the observed strain is indeed due to the favourably oriented twin variant growth.

Testing continued with the same sample by applying two different partial cycling load procedures (Fig. 4). In the first partial cycling the elongation was increased cycle by cycle (Fig. 4a). During the first cycle the total shape change was 4%, in the second cycle 8% and in the third one 12%. The recovery of the structure during the cycles is complete and the stress levels are the same as in the full cycling shown in Fig. 2. The increase of the compressive stress in the end of the third quadrant of Fig. 4a occurs at the same strain value as in Fig. 2. This indicates that the sample has obtained approximately the single-variant structure MV2. The second partial cycling was started with compression to 5% and elongating the sample back to 0% strain (Fig. 4b). With the small cycles of approximately 1% strain carried out inside the 5% cycle it was confirmed that total area of the small cycles was close to the area of the big cycle. This points out that the energy connected to the shape changes is approximately the same in spite of the deformation path.

Another sample was subjected to mechanical testing at different temperatures (Fig. 5a). Even though the highest test temperatures seemed to exceed the given reverse

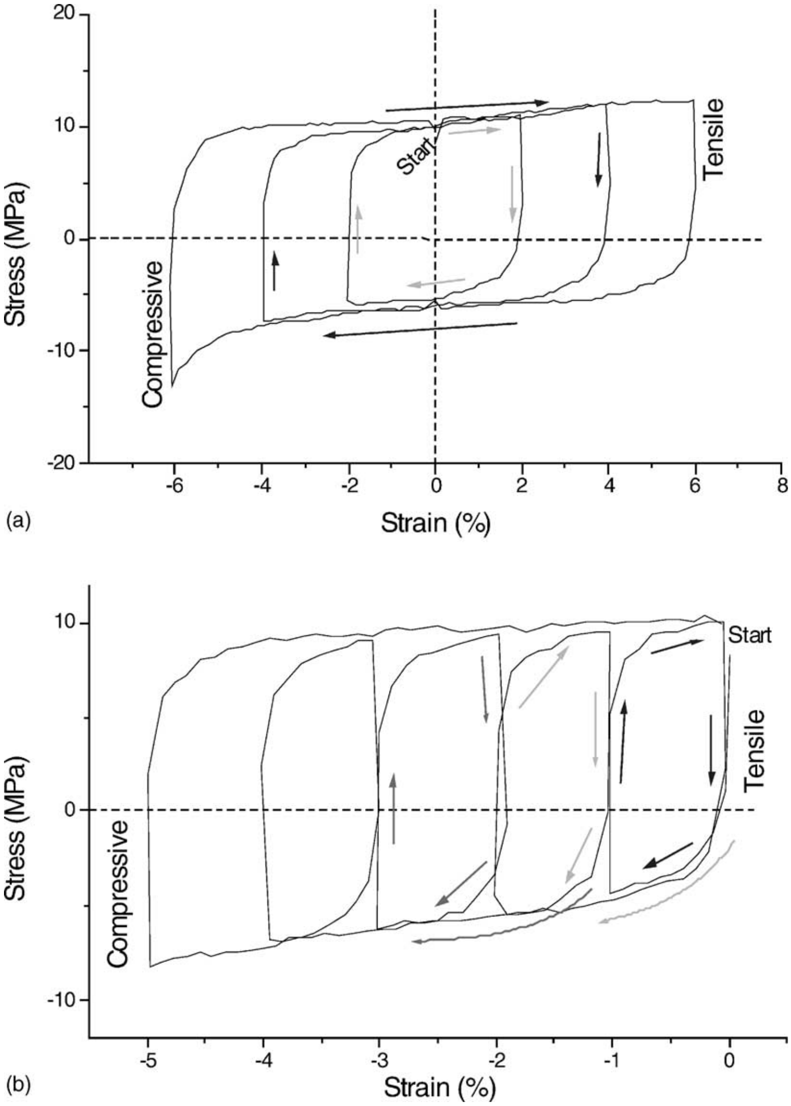

Fig. 4. The partial loading experiments. (a) The increase of elongation cycle by cycle and (b) step-wise cycling compared to a corresponding full single cycle.

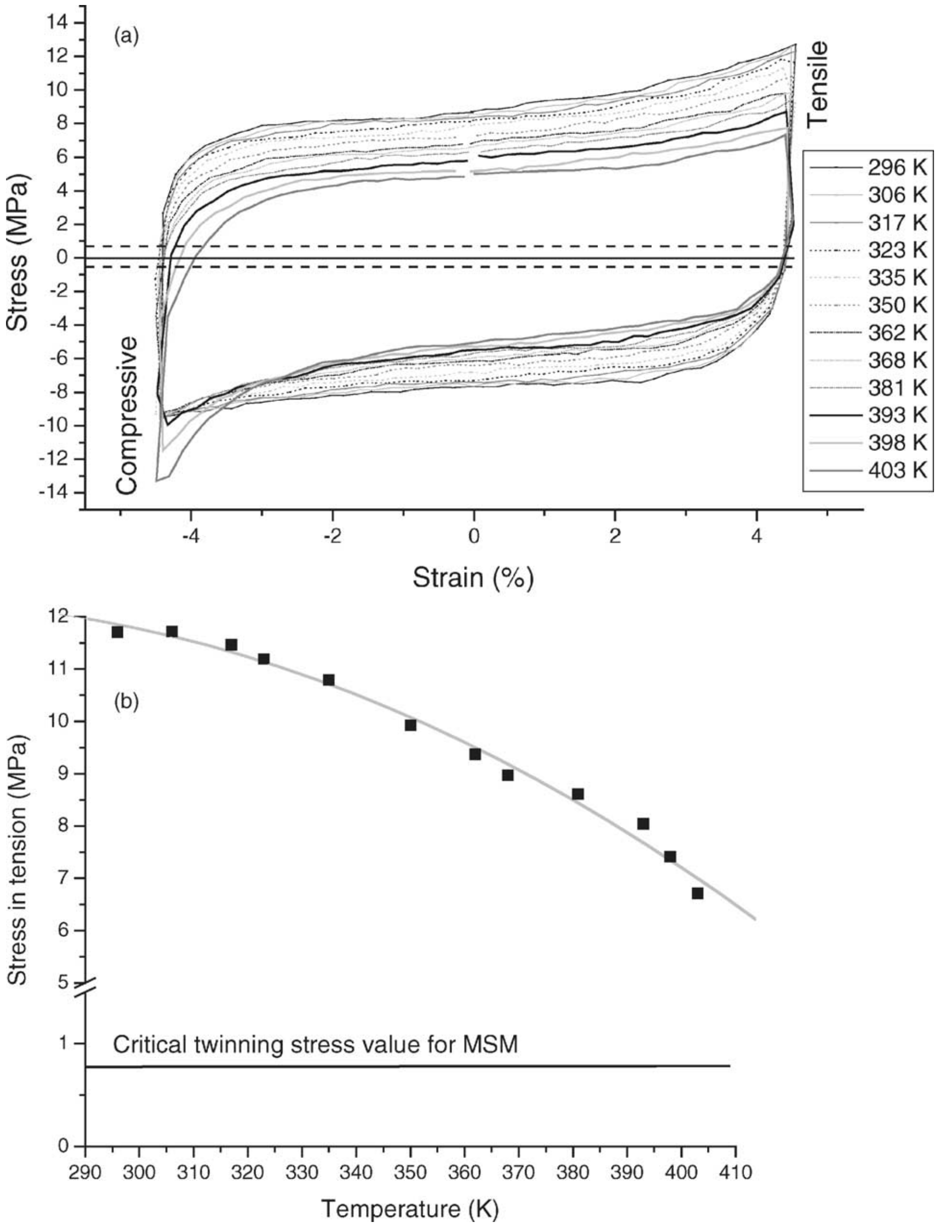

Fig. 5. The effect of the temperature on the mechanical cycling behaviour. (a) Straining cycles at different temperatures. (b) The plateau stress values at the 4% straining in tension.

transformation temperature, all the curves indicated the martensitic behaviour with detwinning at a low stress level. If the material had turned to the parent phase and the stress plateau had been connected to the stress induced martensite formation, the stress level would have been approximately ten-fold higher [25]. The discrepancy of the testing temperatures 381–403 K and the transformation temperatures can not be explained with the increase of the transformation temperature in loading, since this is small in Ni–Mn–Ga alloys, only 3 K in 4–10 MPa compression [26,27]. Therefore, the explanation is more likely the temperature variation in the sample and the cooling effect of the sample grips. Actually, the attempt to carry out the test above 405 K destroyed the specimen, possibly due to a shape change connected to the reverse transformation.

Fig. 5b shows the stress values of the 4% straining as a function of temperature. The twinning stress decreases from the value of 12 MPa at ambient temperature to 6.7 MPa close to reverse transformation (at test temperature 403 K). This correlates well with the approximately 6.5 MPa twinning stress obtained in the compressive test close to reverse transformation [15]. However, the observed high twinning stress exceeds remarkably the magnetically induced stress 0.7–1 MPa of the Ni–Mn–Ga T martensite [15,18] and this excludes the possibility of the MSM effect also in the tensile/compression cycled alloys.

4. Conclusions

In the present work, the tensile behaviour of one non-modulated tetragonal martensitic Ni–Mn–Ga alloy was studied. Based on these studies, the following conclusions can be drawn:

(i) The non-modulated T martensitic structure of the studied alloy is suitable for applications based on the tensile loading. The material shows repeatable behaviour after a couple of tensile/compression cycles. The shape of the tensile/compression curve is symmetric and it is possible to evaluate the full stress–strain pattern with the measurements carried out only in a half of a cycle.

(ii) The X-ray study confirms that one could obtain a true single variant state of the non-modulated martensitic structure in tension. This is important for applications where a true fully oriented structure is needed.

(iii) The twinning stress of the material decreases with cycling and with increasing the temperature. After cycling the original detwinning stress 18–20 MPa decreases down to the value of 10–12 MPa. The lowest detwinning value of 6.7 MPa will be obtained while the testing temperature is close to the reverse phase transformation region.

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