A R C H I V E S O F M E T A L L U R G Y A N D M A T E R I A L S
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AN INVESTIGATION OF MECHANICAL INSTABILITY OF Ni-Mn-Ga SINGLE CRYSTALS COMPRESSED AT ROOM TEMPERATURE

BADANIA NIESTABILNOŚCI MECHANICZNEJ MONOKRYSZTAŁÓW Ni-Mn-Ga ŚCISKANYCH W TEMPERATURZE OTOCZENIA

In order to obtain some information of the room temperature deformability of Ni-Mn-Ga single crystals, known from the literature as those revealing very strong magnetic shape memory effect [1], the uniaxial compression test of the single crystals of different crystallographic orientation and chemical composition was performed. In each case of the tested crystal it was found that: (1) – the elastic/super-elastic range of the stress-strain characteristics is always interrupted by the entry of heavy mechanical instability accompanied by strong and irreversible strain localization; (2) – the instability leads rather rapidly to the sample failure, which may or may not be preceded by the occurence of the second order mechanical instability – strain localization in the already localized zone of crystal deformation. The x-ray measurements and optical microscopy observations show that the tested crystals are of cubic symmetry and the structural mechanism of the instability may consist of the cooperative shear transformation events taking place on the {001} type plane. Further measurements obtained by means of the scanning electron microscope (EBSD method) show that the localized zone of crystal deformation is still of cubic symmetry and the crystallographic relationship between the zone of strain localization and the matrix can be described by a rotation of the crystal lattice around the <110> type direction, and the axis of rotation is as much as possible perpendicular to the compression axis. Moreover, it was established that the angle of rotation may be of several degrees and its exact value depends on the initial orientation of the compression axis and the magnitude of total crystal deformation. Finally, the crystallography of second order mechanical instability has been also established.

Keywords: .....

W celu uzyskania informacji na temat odkształcalności w temperaturze otoczenia stopów Ni-Mn-Ga, wykazujących magnetyczną pamięć kształtu, wykonano próby ściskania monokryształów tych stopów, które posiadały rożną orientację krystalograficzną osi ściskania oraz różny skład chemiczny. Przeprowadzone próby ściskania wykazały że: (1) – w każdym przypadku obszar sprężysty charakterystyki naprężenie-odkształcenie monokryształów kończy się gwałtownym pojawieniem się niestabilności mechanicznej, której towarzyszy silna lokalizacja odkształcenia; (2) – niestabilność ta szybko prowadzi do zniszczenia próbki, która może zostać poprzedzona niestabilnością mechaniczną drugiego rodzaju tj. zlokalizowanym odkształceniem w paśmie pierwotnej lokalizacji odkształcenia. Pomiary rentgenowskie oraz obserwacje mikroskopii optycznej wykazały, że analizowane monokryształy Ni-Mn-Ga posiadają symetrię regularną oraz że aktywna płaszczyzna odkształcenia plastycznego jest typu {001}. Dalsza analiza krystalograficzna uzyskana przy pomocy skaningowej mikroskopii elektronowej (metoda EBSD) pokazała, że obszar zlokalizowanego odkształcenia posiada również symetrię regularną. Relacje krystalograficzne pomiędzy osnową, a pasmem zlokalizowanego odkształcenia można opisać jako obrót sieci krystalicznej wokół kierunku <011>. Kierunek krystalograficzny osi obrotu jest zawsze ten z rodziny <011>, który jest najbardziej prostopadły do kierunku osi ściskania. Ponadto, w pracy zostało pokazane, że kąt obrotu może wynosić nawet kilkanaście stopni, a jego wartość jest zależna od orientacji początkowej osi ściskania oraz stopnia makroskopowej deformacji monokryształu. W końcu przeprowadzono również krystalograficzną analizę zaobserwowanej niestabilności mechanicznej drugiego rodzaju.

1. Introduction

Recently, there has been much attention paid to the Ni-Mn-Ga alloys because of their strong Magnetic Shape Memory effect. By the application of a magnetic field grater than 0,7 Tesla the deformation effect of even 6% can be achieved in single crystal specimens [2], which is several times greater compared to other MSM materials [3]. Especially, the Ni_2MnGa intermetallic compound, due to low temperature martensitic transforma-

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tion in which the austenite phase (cubic structure) turns into tetragonal phase structure, does reveal the greatest MSM effect among other Ni-Mn-Ga alloys. Studies of the properties of Ni-Mn-Ga alloys mainly focused on the changes of physical properties of the material caused by the transformation process. The affect of chemical composition on the critical temperatures of the martenstitic and magnetic transformations was also thorougly studied [4,5]. On the other hand there is lack of fundamental information on mechanical properties of the Ni-Mn-Ga alloys, especiallly those concerning low and room temperature plastic deformability. This work brings first experimental data on the mechanism of room temperature deformation of Ni-Mn-Ga single crystals, not yet reported in the literature. The main aim of the present work is to show the crystallographic features of a mechanical instability which is observed during room temperature compression of Ni-Mn-Ga single crystals.

2. Experimental procedure

Ingots of Ni-Mn-Ga alloys were first prepared from materials of purity: Ni(99,999), Mn(99,98), Ga(99,999), in electrod arc furnace in argon atmosphere, and then they were remelted three times to ensure good homogeneity. Furthermore, from the remelted ingots single crystals of the squared cross-section of 3×3 mm^2 and 50 mm long were grown in a vacuum better than 10^{-4} hPa by modified Bridgman method using vertical temperature gradient furnace and the boron nitride crucibles. The orientations of the single crystal specimens of final rectangular shape 3×3×5 mm^3 were checked by x-ray method by taking the {220} inverse pole figures measurements. The orientations of the compression axis and the chemical composition of the single crystals specimens are shown in the figure 1 and the table 1 respectively. The compression test was performed at room temperature and the initial strain rate of $10^{-4}s^{-1}$ by using Instron testing machine. In order to identify the main plane of crystal deformation the slip lines method was applied, where the two lateral surfaces of the sample were thorougly polished before the compression test. Further crys-

TABLE
The chemical composition of the single crystal specimens used in the experiment

	Ni [%]	Mn [%]	Ga [%]
Specimen 1	48,43	26,7	24,86
Specimen 2	50,4	30,24	19,37
Specimen 3	49,74	25,52	24,74
Specimen 4	50,3	25,87	23,82

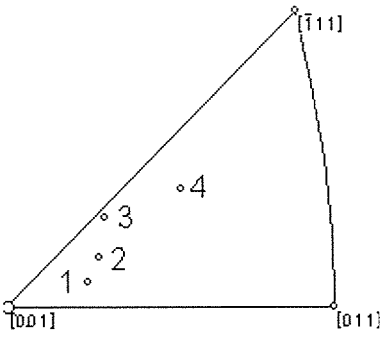

Fig. 1. Initial orientations of the compression axis of the Ni-Mn-Ga single crystals showed in the basic stereographic triangle

-tallographic features of the deformed single crystals were idintified by EBSD method using Hitachi S3400N SEM. For more details of the experimental procedure see further comment in the text.

3. Results and discussion

There are two fundamental observations drawn upon mechanical characteristics of the compression test of Ni-Mn-Ga single crystals: (i) the true stress – true strain characteristics is very sensitive to the initial crystal orientation (Fig. 2 and 3) and the chemical composition (Fig. 4); (ii) – the entry of mechanical instability is independent of crystal orientation and chemical composition and the total critical strain needed to activate the instability is of the order of 10^{-2} (see inserts of figures 2 and 3).

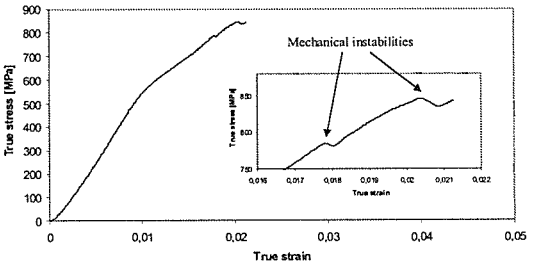

Fig. 2. True stress-true strain characteristics of specimen 1 tested by compression at room temperature (see insert showing the appearance of mechanical instabilities)

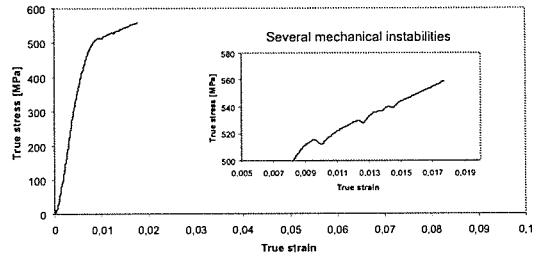

Fig. 3. True stress-true strain characteristic of specimen 4 subjected to the room temperature compression (see insert showing the appearance of several mechanical instabilities)

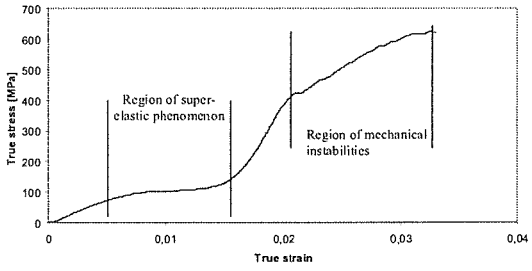

Fig. 4. True stress-true strain characteristic of specimen 2 showing the super-elastic effect generated at the compression stress of about 100 MPa and the higher stress region of mechanical instabilities

When the orientation of the compression axis is near the [001] crystallographic zone (specimen 1), two distinct stages of a linear relationship between the stress and strain is well seen prior to the activation of mechanical instabilities. The compression stress needed to nucleate the first instability is in this case more than 750 MPa, whereas in the specimen 4 the second part of the linear stress-strain characteristics is totally reduced and the mechanical instability appears at the compression stress of about 500 MPa instead. On the other hand, a change of chemical composition according to the table 1 causes the appearance of super-elastic phenomenon in the specimen 2 at the compression stress level of about 100 MPa (Fig. 4). It is already known from the literature that the increase of manganese content in the Ni-Mn-Ga alloys results in the significant increase of the critical temperature of martensitic – cubic to tetragonal phase – transformation [4], which in case of the specimen 2 is near the region of room temperatures. That is why the phenomenon can be relatively easy driven mechanically. However, the compression stresses of more than 400 MPa are still required to nucleate mechanical instabilities in this alloy.

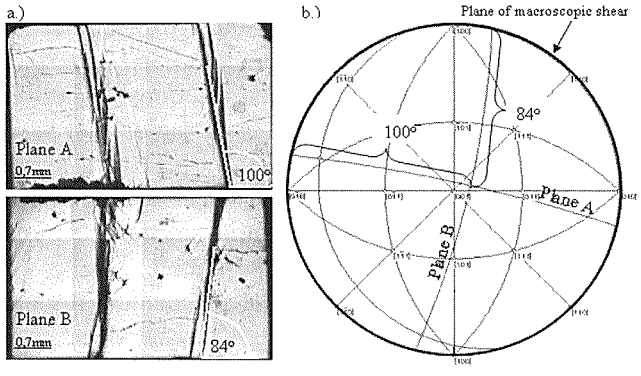

Fig. 5. The optical micrograph of the specimen 1 (a) showing two bands of localized shear deformation associated with two mechanical instabilities (see insert of Fig.2), and the crystallographic analysis of the plane of macroscopic shear performed on the lateral surfaces A and B of the crystal by means of the [001] stereographic projection (b)

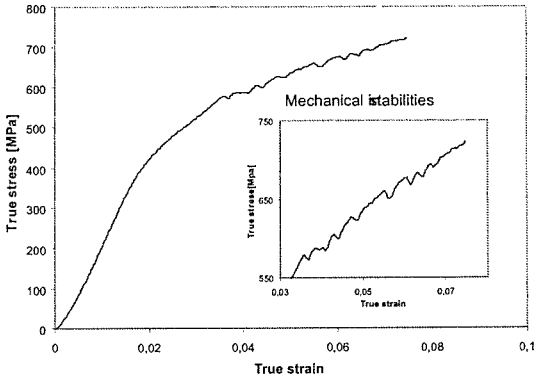

Fig. 6. True stress-true strain characteristics of the specimen 3 showing several mechanical instabilities generated during room temperature compression test

It is to emphasize that before the sample failure a real change of the height of deformed single crystals was of the order of 10^{-2} (Figs. 2-4) and it was generated by the crystal deformation regions where the mechanical instabilities have appeared. Moreover, the number of mechanical instabilities observed on the stress-strain curves corresponds to the number of local deformation bands, which can be easy observed on the lateral surfaces of the deformed crystals by optical microscope. For example, the two load drops observed on the mechanical characteristics of the specimen 1 (Fig. 2) is caused by the activation of the corresponding two deformation bands localized in two different crystal regions (Fig. 5a). Combining the optical microscopy observations and x-ray measurements the trace analysis was performed (Fig. 5b), which shows undoubtedly that crystal deformation within the bands is organized on the {001} type crystallographic planes. The specimen 3 was able to survive several activations of mechanical instabilities before it had broken (Fig. 6), and of course, several deformation bands are well seen on the lateral surfaces of the deformed crystal (Fig. 7a). Again the trace analysis shows that the plane of the macroscopic shear of specimen 3 is parallel to the cube plane of the crystal lattice (Fig. 7b). Figure 7 shows another important feature of the deformation bands. One can see that the shear within the bands is very homogeneous and similar to that caused by twin or martensitic transformations. One can also suggest that many parallel crystal planes of {100} type should be cooperatively involved in order to produce such homogeneous shear within the deformation bands. Such mechanism of crystal deformation should give rise to a sudden change in the lattice orientation across the interface between the matrix and the deformation band. The discontinuous change of crystal orientation across the matrix/deformation band interface was revealed by EBSD measurements in each of the specimen used in the experiment. For instance, in the case of specimen 1 the orientation relationship between the matrix region

and the deformation bands (Fig. 8a) can be described by the misorientation angle of about 12 degrees (Fig. 8b) with the axis of rotation of <110> type (Fig. 8c). It to note that the axis of rotation is always as close as possible perpendicular to the axis of compression. As it was observed in the case of specimen 2, the increase of total crystal deformation may result in larger angle of misorientation and in the activation of secondary mechanical instability phenomenon (Fig. 9a,b). It is worth to emphasize that the activation of secondary instability causes another rotation of crystal lattice within the primary deformation bands and the rotation axis remains unchanged with respect to that of primary mechanical instability. Moreover, the crystal structure of both primary and secondary deformation bands is still of regular symmetry (Fig. 9c) and the cumulative angle of misorientation was about 30 degrees. It is important to note that

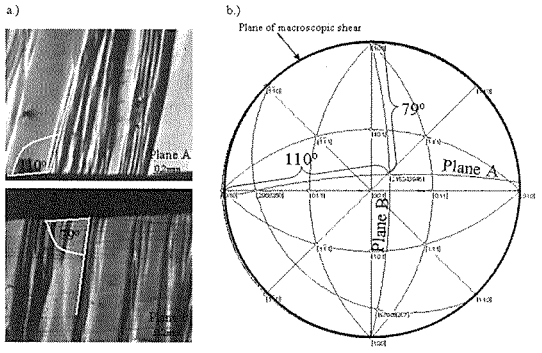

Fig. 7. The optical micrograph of the specimen 3 (a) showing several bands of localized shear deformation on the lateral surfaces A and B of the crystal and (b) and the crystallographic analysis of the plane of macroscopic shear performed on the lateral surfaces A and B of the crystal by means of the [001] stereographic projection (b)

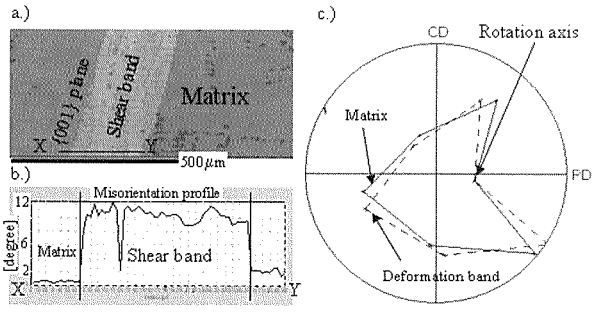

Fig. 8. The EBSD picture of the localized deformation band of the specimen 1 (a), the corresponding profile of the angle of misorientation (b) and the stereographic projection representation of the crystallographic relationship between the lattices of the matrix and the localized deformation band (c)

the appearance of the secondary deformation bands do significantly enhance the process of nucleation of cracks and their macroscopic propagation in a specimen seems strongly correlated with the position of the deformation bands what is shown in the figure 9a. The phenomenon of secondary mechanical instabilities and their role in the cracks nucleation process was also well seen in the case of specimen 3, where the angle of misorientation across the interface between the second deformation bands and the matrix region was of about 20 degrees.

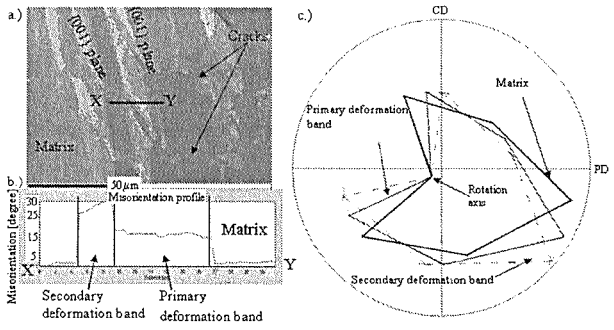

Fig. 9. The EBSD picture of the localized deformation band of the specimen 2 (a), the corresponding profile of the angles of misorientation (b) and the stereographic projection representation of the crystallographic relationship between the lattices of the matrix and the primary and secondary localized deformation bands (c)

4. Concluding remarks

The room temperature compression of Ni-Mn-Ga alloy single crystals of cubic symmetry generates at relatively high stress level (0.5-1.0 GPa) well defined mechanical instabilities which are activated irrespective of the initial crystal orientation or the chemical composition used. However, the elastic parts of the true stress-true strain compression characteristics are very sensitive to these factors.

The mechanical instabilities are always accompanied with the formation of local deformation bands and the crystal regions within the band are still of cubic symmetry, however, a discontinuous change of crystal orientation across the matrix/deformation band interface is well seen. The crystal orientation relationship between the matrix and the deformation band region can be described by a rotation around one of the <110> type crystallographic directions, and the amount of the angle of misorientation depends on the initial crystal orientation and the total value of crystal deformation.

Very important feature of the mechanical instability seems to consists of cooperative crystal shear events which are organized on the macroscopic scale parallel to one of the {001} type planes of crystal lattice. This suggests that the character of the associated process of deformation band formation is rather purely transformational.

The mechanism of room temperature fracture of the Ni-Mn-Ga alloy single crystals is strongly controlled by

the entry of primary and particularly the secondary mechanical instabilities, and the second order mechanical instabilities do generate within the primary bands another deformation bands (second order bands) which are still of cubic symmetry.

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REFERENCES

[1] O. Heczko, L. Straka, Czechoslovak Journal of Physics **54** (2004).
[2] A. A. Likhachev, K. Ullakko, Physics Letters A **275** 142-151 (2000).
[3] R. Hayashi, S. J. Murray, M. Marioni, S. M. Allen, R. C. O'Handley, Sensors and Actuators **81** 219-223 (2000).
[4] S. K. Wu, S. T. Yang, Materials Letters **57** 4291-4296 (2003).
[5] F. Albertinia, F. Canepab, S. Cira.cib, E. A. Franceschib, M. Napoletanob, Paoluzia, L. Paretia, M. Solzi, Journal of Magnetism and Magnetic Materials **272-276** 2111-2112 (2004).