# MECHANICAL AND STRUCTURE PROPERTIES OF STOICHIOMETRIC DIRECTIONALLY SOLIDIFIED $Ni_3Al$

*Losertová M., Hyspecká L.*

*Faculty of Metallurgy and Material Engineering, Technical University (VŠB-TU),*

*av. 17.listopadu 15, 708 33 Ostrava 8, Czech Republic*

# MECHANICKÉ A STRUKTURNÍ VLASTNOSTI $Ni_3Al$ PO SMĚROVÉ KRYSTALIZACI

*Losertová M., Hyspecká L.*

*Fakulta metalurgie a materiálového inženýrství, Vysoká škola báňská-Technická Univerzita Ostrava, tř.17.listopadu 15, 708 33 Ostrava 8, Česká republika*

**Abstrakt**

Morfologie struktury stechiometrického $Ni_3Al$ po směrové krystalizaci a po tahové zkoušce za pokojové teploty byla studována pomocí optické a elektronové (SEM) mikroskopie. Při fraktografickém studiu byl na lomových plochách pozorován převážně interkrystalický lom, byly však nalezeny rovněž oblasti s vyšší lokalizovanou houževnatostí s transkrystalickým tvárným, příp. smíšeným lomem. Ze srovnání šířky a vzdálenosti jednotlivých pásů a stupňů na metalografickém výbrusu a na štěpném lomu lze podpořit možnost plastické deformace mechanickým dvojčatěním uplatňujícím se vedle mechanismu pohybu a skluzu dislokací v intermetalických slitinách $Ni_3Al$.

**Abstract**

The morphology of the room-temperature tensile tested structure of the stoichiometric directionally solidified Ni3Al was investigated using the optical and SEM microscopy analysis. The brittle intergranular fracture was dominant on fracture surfaces, however the transgranular cleavage, as well as domains of localised higher ductility with ductile transgranular or mixed fracture were observed in fractographic analysis. Comparison of the width and the spacing of bands and steps on the metallographic samples and on the cleavage fracture supports the possibility of the plastic deformation mode by the mechanical twinning produced beside the motion and slip of dislocations in the intermetallic alloys $Ni_3Al$.

## 1. Introduction

Intermetallic alloys based on the $Ni_3Al$ have attracted significant attention for more than three decades already. Numerous studies on the mechanism of anomalous temperature dependence of the yield stress of these alloys have been carried out. However, the structure after the room temperature deformation of this intermetallic compound remains still known insufficiently. From a few recent studies [1,2], we can presume that the morphology of the deformation structure will be somewhat different from that of common F.C.C. metals and alloys. It has been suggested by Gottstein et al. [3]

that the stacking fault energy of $Ni_3Al(B)$ should not be significantly different from that of pure Ni. The deformation texture of nickel, like that of copper, is known to develop by the slip on {111} planes and even after heavy cold deformation, twins have not been reported in either Cu or Ni.

During room-temperature deformation, the {111} <101> slip develops in the $Ni_3Al$ with $L1_2$ structure. However, Chowdhury et al. [4] have found the presence of a high density of twins in the structure of the 85 % cold rolled $Ni_{76}Al_{24}$ (0,24 % B). Based on the measurements of the order parameter (S) by X-ray diffraction technique, they reported that heavy amounts of cold deformation can produce a transformation from cubic $L1_2$ to a tetragonal structure $D0_{22}$ (a = 0.356 nm a c = 0.719 nm), when deformation mode is mostly realised by twinning.

It is evident that like for the F.C.C. structure, also in the $L1_2$ there will be mechanical twinning associated with formation of stacking faults on {111} planes (twinning planes) [5]. Chu and Pope [1] presume for the $L1_2$ structure that the atoms on each layer glide along the deformation structure [11] direction with a shear, g=1.414 (where g is the proportionality constant and expresses the strength of the simple shear), without atomic shuffling as shown in Figure 1.

Fig.1 Schematic of the formation of a {111}[11] twin in the $L1_2$ structure [1]

The unit superlattice dislocation able to glide on {111} plane can dissociate into Shockley partials producing two complex stacking faults (CSF) and one antiphase boundary fault (APB) [6], all those stacking faults are appearing on {111}. Pope and Ezz [7] have described four stacking fault types on {111} in the $L1_2$ superlattice: antiphase boundaries (APB) being relatively high energy faults (from 83 to 140 $mJ/m^2$) [6,8,9], superlattice intrinsic stacking faults (SISF) with low energy (5-15 $mJ/m^2$) [6,10,11,12], complex stacking faults (CSF) produced by shear of type 1/6<112> and with the higher energy than that of the APB (235 ± 40 $mJ/m^2$) [6] and superlattice extrinsic stacking faults (SESF) with energy similar to that of the SISF [8].

The purpose of this study was to obtain further information on the mode of the plastic deformation and to verify, whether the mechanical twinning in the $L1_2$ is possible. We utilised the optical and SEM microscopy analysis as the simplest method for investigation of the room-temperature tensile tested samples of the polycrystalline and single phased structure ($\gamma'$) and of the stoichiometric composition ($Ni_{75}Al_{25}$).

## 2. Experimental procedure

The raw material rod of stoichiometric $Ni_3Al$ was prepared by vacuum melting of pure electrolytic nickel (> 99.95 mass %) and aluminium (99.99 mass %). The impurity contents in Ni and Al was approx. 0.001 mass % in order. The melting and the casting into the graphite mould were carried out in the induction vacuum furnace installed in the laboratory of VŠB-TU Ostrava. As-cast rods were remelted and unidirectionally solidified in flowing argon atmosphere by a modified Bridgman technique with vertical arrangement (also installed in laboratory of VŠB-TU Ostrava). The growth rate was 11.4 mm per minute.

The homogenisation annealing at 1373 K for 8 hours in resistance furnace with a flowing argon was performed to assure single phased structure and to eliminate chemical inhomogeneity. Tensile samples having a gauge section 90 x 30 x 6 mm were cut from the annealed polycrystalline rods of 12 mm in diameter and 120 mm in length.

Tensile tests were carried out using an INSTRON testing machine at room temperature with low strain rate of $5.8 \times 10^{-5}$ $s^{-1}$. The yield and ultimate strength were determined from the stress-strain curves. The elongation was determined from fractured samples.

Observation of the deformation structure on the polished and etched samples (perpendicular and parallel to the tensile direction) and on the fracture surfaces was performed by an optical microscope Neophot 32 and a scanning electron microscope (SEM) PHILIPS XL 30 respectively.

## 3. Results and Discussion

The microstructure of the unidirectionally solidified and annealed samples contained great columnar grains, the grain size of which was sometimes greater than 1000μm in transverse and longitudinal section to growth direction and was elongated in the growth direction [13]. However, the grains were sectioned by sub-boundary network (average subgrain size in the transverse section to the growth direction was of 57 μm; as measured using the method of linear intercepts), and at the same time the structure contained smaller grains of approx. 50 μm. Annealing twins were observed in the homogenised columnar grains.

The obtained mechanical characteristics of this structure type of the samples are summarised in the Table 1. The ultimate strength values exhibited great scattering and did not exceed 300 MPa. Unfortunately, several samples failed by premature fracture, which reduced the number of results suitable for statistical evaluating of the mechanical properties.

From the stress-strain curve shown in the Figure 2, an abnormal course of deformation behaviour during the tensile loading is evident, which reflects the influence of the crystal morphology in the sample microstructure. During the tensile testing, "pulling-out" and elongation of long crystals were observed (Figure 3) [13]. Therefore, the determined elongation seems to be rather of orientation character [13]. In addition, the cracking was seen on the side surface of samples. Based on the study of metallographic sections, we can say that the grain boundary decohesion was produced due to casting defects (pores) in the rod structure. However, several samples remained non-fractured after the tensile testing.

Generally, our unidirectionally solidified samples exhibited smaller tensile elongation than Hirano et al. [14-16] have reported, but from the above mentioned results we can confirm that the crystals behave like single crystals with good room-temperature ductility. There remains, however, one question, particularly how much the intergranular fracture was influenced by unexpected presence of internal casting defects on the grain boundaries (pores and boundary decohesion) and how much this fracture mode is an intrinsic property of the ordered structure.

In spite of predominant presence of the brittle intergranular fracture on fracture surfaces, the transgranular cleavage was observed in fractographic analysis, as well as domains of localised higher ductility (Fig.4 and 5). The surface effects of deformation bands appearing on the surface were observed in certain grains (Fig.4).

Table 1 The mean values of the mechanical properties obtained during the room-temperature tests of unidirectionally grown and annealed samples (strain rate of $5.8 \times 10^{-5}$ $s^{-1}$ ).

Remarks : the *results for samples failed by premature fracture are not considered in the mean values

Fig.2 Stress-strain curve of tensile-tested samples 1 (fractured during testing)

The anomalous yield behaviour is explained as being due to the thermally activated cross-slip of screw superlattice dislocations and due to the energy anisotropy and mobility of the screw superlattice dislocations [6,10,17,18]. As it was described above, the screw dislocations can dissociate on the octahedral {111} planes and namely on the cube {010} planes [17]. Considering the fact that the twinning dislocations are associated with stacking faults and that four types of stacking faults were found in the $L1_2$ structure relative to the dissociation of superlattice dislocations, we can assume that the observed structure phenomena on the transgranular cleavage fracture surfaces and on the metallographically polished samples will be mechanical twins. After comparing the width and the spacing of bands and steps on the metallographic samples and on the cleavage fracture it is possible to state, that they are corresponding in size. The width of mechanical twins is ranging from 1 to 15 μm and their spacing ranges from 5 to 160 μm. It is evident from figure 6 that parallel twin pairs occur in the microstructure, presumably with semicoherent interfaces and they can standstill on subgrain boundaries or traverse them. At higher magnification (800x) we observed that at traversing grain or subgrain boundaries the bands are bent although under very low angle [13]. In addition, the transgranular cleavage was observed as being related to appearance of twins, it therefore remains unclear, how we can compare our results with the assumption concerning the role of the low angle boundaries (Σ3) in ductility enhancing of the structure [20,21].

Figure 7 shows schematically the fracture mode of slip band/twin band as it was proposed by Stoltz and West [22] based on experimental observation of transgranular cleavage fracture in Nitronic 40 alloy (with stacking fault energy of 32 $mJ/m^2$). Using this schematic we can explain the transgranular cleavage fracture in relation to deformation twinning in our alloys.

Fig.7 Schematic of slip band/twin band cracking process in low strength F.C.C. alloys [22]

a) slip band/twin band; b) c) cracking process

The observed deformation structure of $Ni_3Al$ contained naturally dislocation pile-ups (width below 1 μm), which were well revealed by the selective etching [13]. It means, that besides the plastic deformation mode by mechanical twinning, the mode of slip of dislocations also appears.

More detailed crystallographic analysis of the deformation structure is needed to verify the view discussed above, e.g. by method of diffraction analysis.

## 4. Conclusion

The presented results support the possibility of the plastic deformation mode by the mechanical twinning produced beside the slip of dislocations in the intermetallic alloys $Ni_3Al$. It was experimentally confirmed by observation of the morphology of the tensile-tested microstructure on the metallographically polished and etched samples by means of optical microscopy and on the fracture surfaces by means of SEM microscopy.

## Literature


[1] Chu, F., Pope, D.P.: Mat. Sci. Eng., A170, 1993, p. 39

[2] Chowdhury, S.G., Ray, R.K., Jena, A.K.: Scr. Metall. Mater., 32, 2, 1995, p. 213

[3] Gottstein, G., Nagpal, P., Kim, W.: Mater. Sci. Eng., A108, 1989, p. 165

[4] Chowdhury, S.G., Ray, R.K., Jena, A.K.: Scr. Metall. Mater., 32, 9, 1995, p. 1501

[5] Christian, J.W.: The Theory of Transformation in Metals and Alloys, Pergamon, London, 1975

[6] Karnthaler, H.P. et al.: Acta Metall. Mater., 44, 2, 1996, p. 547

[7] Pope, D.P.-Ezz, S.S.: Inst. Met. Rev., 1984, 29, p. 136

[8] Stoloff, N.S.: Physical and mechanical metallurgy of $Ni_3Al$. Int. Mat. Rev., 1989, 34, 4, p. 153

[9] Kear, B.H.-Hornbecker, M.F.: Trans. Am. Soc. Metals, 59, 1966, p.155

[10] Veyssiere, P.: ISIJ International, 31, 10, 1991, p.1028

[11] Douin, J., Veyssiere, P., Beauchamp, P.: Philos. Mag. A, 54, 1986, p. 375

[12] Chen, S.P., Voter, A.F., Srolovitz, D.J.: Scr. Metall., 20, 1986, p. 1389

[13] Losertová, M.: Material and technical aspects of intermetallic alloys based on $Ni_3Al$. Thesis, VŠB TUO (CZ) - ECP (F), August 1998, 178 p.

[14] George, E.P., Imai, M., Hirano, T.: Intermetallics, 5, 1997, p. 425

[15] Hirano, T., Kainuma, T.: ISIJ Intern., 31, 10, 1991, p. 1134

[16] Mawari, T., Hirano, T.: Intermetallics, 3, 1995, p. 23

[17] Kear, B.H., Wilsdorf, H.G.F.: Trans. MS AIME, 224, 1962, p. 382

[18] Paidar, V., Pope, D.P., Vitek, V.:Acta Metall., 1984, 32, p. 435

[19] Korner, A.: Acta Metall. Mater., 42, 5, 1994, p. 1695

[20] Hui Lin, Pope,D.P.: Acta Metall. Mater., 41, 2, 1993, p. 553

[21] Chiba, A. et al.: Acta Metall. Mater., 42, 5, 1994, p. 1733

[22] Stoltz, R.E., West, A.J.: in: Hydrogen Effects in Metals, ed. I.M.Bernstein and A.W.Thompson, Proc. of the 3rd Intern. Conf. on Effect of Hydrogen on Behavior of Materials, Metall. Soc. of AIME, 1980, p. 541