STRUCTURAL AND MICRO-STRUCTURAL ANALYSIS OF DIRECTIONALLY CRYSTALLISED INTER-METALLIC COMPOUND Ni_3Al REALISED BY TEM

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STRUKTURNÍ A MIKROSTRUKTURNÍ ANALÝZA SMĚROVĚ KRYSTALIZOVANÉ INTERMETALICKÉ SLOUČENINY Ni_3Al PROVEDENÁ POMOCÍ TEM

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Abstrakt

Pro provedení strukturní a mikrostrukturní analýzy byly použity vzorky Ni3Al s obsahem hliníku 25 at.%. Pro upravení jejich lité struktury byla použita metoda směrové krystalizace, kterou lze ovlivnit strukturu a tím i vlastnosti výsledného materiálu. Použité vzorky byly připraveny odlitím ve vakuové indukční peci. Tyto odlitky ve formě válečků o průměru 10 mm a délce 95 mm byly dále směrově krystalizovány Bridgmanovou metodou s vertikálním uspořádáním. Část vzorků byla připravena na katedře neželezných kovů, rafinace a recyklace VŠB-TU Ostrava a další část vzorků byla tavena v elektrokorundových trubicích se specifikovaným vrcholovým úhlem ve specielním zařízení ve Vladimiru (Rusko). Byly použity různé rychlosti směrové krystalizace pro ověření nejvhodnějších podmínek pro získání homogenní struktury. Vhodné vzorky byly upraveny soustružením na tahové tyče a ty byly následně použity pro tahové zkoušky se současným měřením akustických emisí. Pro tyto zkoušky byly použity krátké tahové tyče s kruhovým průřezem o délce 55 mm s průměrem střední části tyče 5 mm. Při všech měřeních byla pozorována intenzivní akustická emise vyznačující se charakteristickou závislostí na čase a korelací s jednotlivými stádii deformace. Vzorky 139.2 a 139.3 vykazují podstatné zvýšení tažnosti s ohledem na běžně připravované materiály tohoto typu. Na příčných i podélných průřezech bylo provedeno detailní metalografické vyhodnocení. Některé vzniklé struktury jsou velmi homogenní. U vzorků tavených v korundových trubicích se specifikovaným úhlem vznikla struktura obsahující v matrici dislokační stěny. Struktura vzorků byla podrobně analyzována pomocí transmisní elektronové mikroskopie. Na připravených foliích byla stanovena hustota dislokací v nedeformovaném a deformovaném stavu.

Abstract

Structural and micro-structural analyses were performed on samples of Ni_3Al with aluminium content of 25 at.%. Method of directional crystallisation was used for modification of

their as-cast structure, as this method can influence structure and therefore also properties of final material. The samples were prepared by casting in vacuum induction furnace. Cast rollers with diameter 10 mm and length 95 mm were afterwards directionally crystallised by Bridgman method with vertical arrangement. Part of samples was prepared at the Department of non-ferrous metals, refining and recycling at VŠB-TU Ostrava, and remaining part of the samples was melted into electro-corundum tubes with specified apex angle on special equipment in Vladimir (Russia). Various rates of directional crystallisation were used for verification of the most suitable conditions for obtaining of homogenous structure. Suitable samples were adjusted by turning to tensile rods, which were then used for tensile tests with measurement of acoustic emissions. For these tests there were used short tensile rods of round section with length 55 mm and with diameter of central part of the rod 5 mm. During all measurements there was monitored intensive acoustic emission with characteristic dependence on time and with correlation to individual stages of deformation. The samples 139.2 and 139.3 show substantial improvement of ductility in comparison with normally prepared materials of this type. Metallographic evaluation was made on cross sections and longitudinal sections. Some of the formed structures are highly homogenous. In samples melted in electro-corundum tubes with specified angle a structure containing dislocation walls in the matrix was formed. Structure of samples was minutely analysed with use of transmission electron microscopy. Dislocation density in deformed and non-deformed state was determined on prepared foils.

Keywords: Ni-Al based intermetallic compounds, tensile tests, transmission electron microscopy, dislocation density

1. Introduction

Directional crystallisation by Bridgman method is one of the simplest and most frequently used methods. The tube with charge in its bottom part is situated into thermal field of furnace. During slow passage of the tube with melted metal through steep temperature gradient there occurs progressive directional crystallisation of molten metal and thus growth of crystal [1].

Crystal defects influence substantially properties of materials. Dislocations are line defects, which enable plastic deformation of crystals. TEM is used for determination of their density, arrangement, Burgers vector and slip system, in which they move. It is also possible to observe interaction of dislocations with various obstacles [2]. Dislocation density influences strength of metal. Number of dislocations increases due to mechanical load, and resistance to deformation is thus also increased, as there occurs strengthening and increase of strength. Unequivocal influence of dislocation strengthening on increase of yield strength was proven. Determination of dislocation density can be made with use of technique of thin metallic foils prepared from investigated material. This technique enables observation of very fine semi-coherent, or coherent precipitates in initial stage of their formation and obtain thus also an idea about state of sub-structure of individual phases. It is thus possible to obtain information about kind, density and distribution of crystal-lattice defects (dislocations).

2. Preparation of samples

Cast pieces were used for experimental purposes. The cast pieces were prepared in vacuum induction furnace LEYBOLD - type IS3/1. Prior to the melting there were performed 2 vacuum treatments, namely below the value of 0.04 mbar with use of two-stage pumping by

rotary and Roots pumps. Several basic samples were cast with composition of 25 at. % Al in the form of rollers with length of 95 mm and diameter of 10 mm. Melting was realised in a corundum crucible and casting was made into graphite ingot moulds.

The samples 128.1 and 128.2 were directionally crystallised by Bridgman method at the Department of non-ferrous metals, refining and recycling at VŠB-TU Ostrava. Samples 139.1, 139.2 a 139.3 were used for directional crystallisation by Bridgman method on the equipment in Vladimir in Russia. Special corundum crucibles with an apex angle 60-70° were used for these samples.

3. Mechanical properties and structural characteristics of directionally crystallised Ni_3Al

The samples prepared in this manner were adjusted by turning to tensile rods, which were then used for tensile tests with measurement of acoustic emissions. For these tests there were used short tensile rods of round section with length 55 mm and with diameter of central part of the rod 5 mm. During all measurements there was monitored intensive acoustic emission with characteristic dependence on time and with correlation to individual stages of deformation. The samples 139.2 and 139.3 show substantial improvement of ductility in comparison with normally prepared materials of this type [3]. Tables 1 and 2 give speed of directional crystallisation (DC), values of micro-hardness, porosity and mechanical properties of the used samples.

Table 1 Speed of directional crystallisation, values of micro-hardness, porosity and mechanical characteristics of the samples 128

Sample No.	Porosity [%]	Micro-hardness HV 0.05	Speed DC [mm/h]	R_m [MPa]	R_p [MPa]	A [%]
128.1Z	0.0430	523	100	318	295	0.51
128.1 K	0.0388	572	100			
128.1H	0.0462	374	100			
128.2Z	0.0835	555	50	330	255	2.83
128.2 K	0.0180	549	50			
128.2H	0.0511	312	50			

Z – Beginning of the sample, cross section
K – End of the sample, cross section
H – Head of tensile bar, longitudinal section

Table 2 Speed of directional crystallisation, values of micro-hardness, porosity and mechanical characteristics of the samples 139

Sample No.	Porosity [%]	Micro-hardness HV 0.05	Speed DC [mm/h]	R_m [MPa]	R_p [MPa]	A [%]
139.1Z	0.0895	256	108	392	267	13.9
139.1S	0.0703	268	108			
139.1K	0.0242	485	108			
139.2Z	0.0735	286	60	707	343	20.5
139.2S	0.0503	269	60			
139.2K	0.0288	394	60			
139.3Z	0.0606	344	18	505	245	53.4
139.3S	0.0370	262	18			
139.3K	0.0354	458	18			

Z – Beginning of the sample, longitudinal section
S – Centre of the sample, cross section
K – End of the sample, cross section

Metallographic evaluation was made on cross sections and longitudinal sections. Some of the formed structures are highly homogenous. In samples melted in electro-corundum tubes with specified angle a structure containing dislocation walls in the matrix was formed. Structure of samples was minutely analysed with use of optical and transmission electron microscopy. Micro-hardness and porosity were determined in cross-section and longitudinal sections.

The Figures 1 and 2 show micro-structures of on cross sections of the samples Nos. 128.1 and 1128.2 in central part of the sample. The Figures 3, 4 and 5 show micro-structures of on cross sections of the samples Nos. 139.1, 139.2 and 139.3 in central part of the sample.

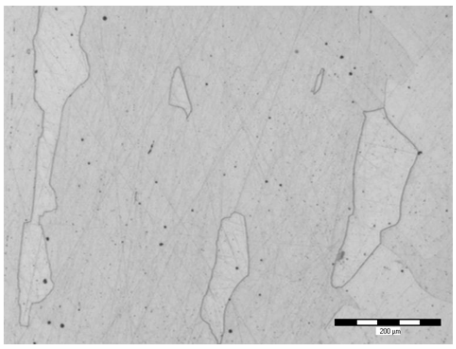

Fig.1 Sample No. 128.1, cross section, Z=100x

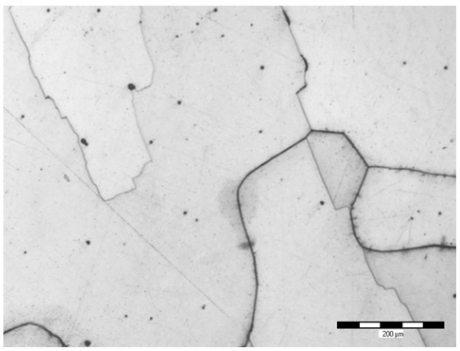

Fig.2 Sample No. 128.2, cross section, Z=100x

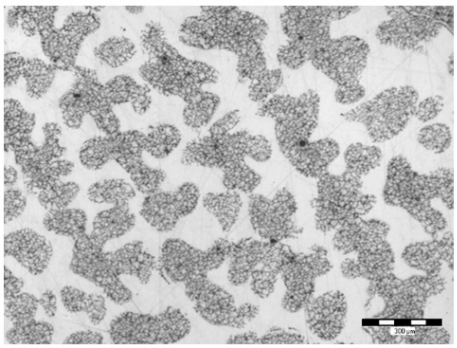

Fig.3 Sample No. 139.1, cross section, Z=50x

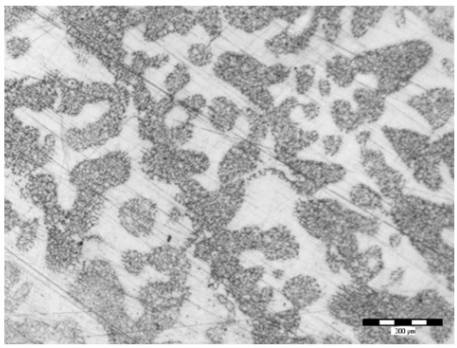

Fig.4 Sample No. 139.2, cross section, Z=50x

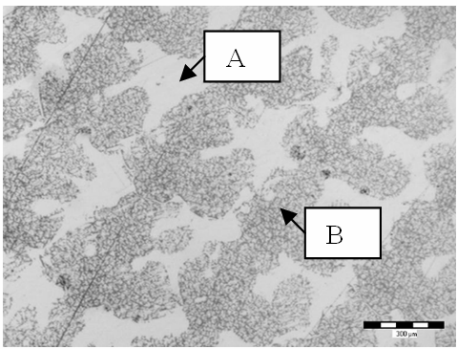

Fig.5 Sample No 139.3, cross section, Z=50x

A - matrix
B - structure formed by cellular structure of dislocation walls in basic matrix Ni_3Al

4. Determination of dislocation density of material in non-deformed and in deformed state

Dislocation density was determined in initial non-deformed state and then in deformed state of the given material. Deformation occurred at tensile tests when applied strain rates were within the interval from 1.15-1.20*10^{-4} s^{-1}.

Determination of dislocation density was performed on 5 samples marked as 128.1, 128.1, 138.1, 138.2 and 138.3 (see Tables 3 and 4). Dislocation density was determined from each sample from its deformed part (D), and also from its non-deformed part (N). Targets were prepared from parts of bars after tensile test.

Targets with thickness of 0.5mm were cut by diamond saw from the relevant part of the samples. They were used for preparation of targets with diameter of 2.8mm that were cut by electro-spark cutter. These targets were afterwards further thinned by grinding on metallographic sand papers to the thickness of 0.15mm. They were then used of electrolytical preparation of foils for observation in transmission electron microscope Jeol 2000FX. Electrolyte consisted of solution 5% $HClO_4$ + 95% CH_3OH, voltage 20V, temperature minus 10°C.

Dislocation density ρ was determined according to the relation:

$$\rho = \frac{1}{t}\left\{\frac{\sum n_1}{\sum L_1} + \frac{\sum n_2}{\sum L_2}\right\} \tag{1}$$

where t is thickness of foil at the observed point, n_1 is number of intersections of dislocation lines with vertical lines of regular grid, L_1 is vertical dimension of the grid, n_2 is number of intersections of dislocation lines with horizontal lines of regular grid, L_1 is horizontal dimension of the grid. Sums in the formula mean summing across all the vertical or all the horizontal lines. Initial objective consisted in determination of the foil thickness with use of diffraction of electrons in a convergent beam (CBED). However, it was found that points, at which it is possible to make evaluation of dislocation density are so thick, that this method is unusable. That's why for determination of the value t we used the fact that majority of dislocation lines lies in directions of the type [110] [4]. It means that if crystallographic and geometric orientation of the foil in a microscope at taking of the photo is known, it is possible to count from the length of projections of dislocation lines calculate the foil thickness. Crystallographic orientation was determined with use of Kikuchi lines in diffraction of electrons and geometric orientation of the foil was determined by reading of angles of inclination on goniometer.

Results are summarised in the following tables:

Table 3 Dislocation density in non-deformed parts of material

Sample	128.1-N	128.2-N	139.1-N	139.2-N	139.3-N
A - ρ [10^{13} m^{-2}]	0.7	0.6	1.2	0.8	0.3
B - ρ [10^{13} m^{-2}]	-	-	-	--	-

Table 4 Dislocation density in deformed parts of material

Sample	128.1-D	128.2-D	139.1-D	139.2-D	139.3-D
A - ρ [10^{13} m^{-2}]	2.2	8.7	6.1	13.7	10.8
B - ρ [10^{13} m^{-2}]	-	-	5.0	7.8	3.6

The values marked as A are dislocation densities from the places where there is no cell structure of dislocation walls. The values marked as B are dislocation densities from the places where there is cell structure of dislocation walls.

Dislocations usually occur in material in pairs (Fig. 6) and direction of dislocation line is identical with some type of crystallographic direction [110].

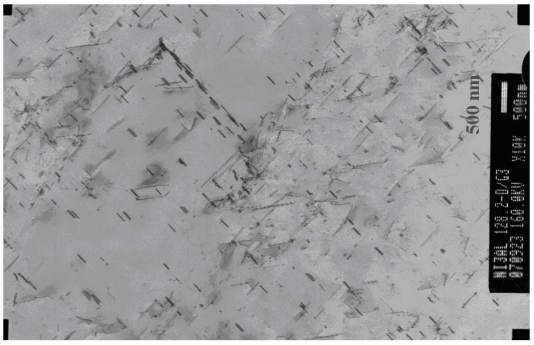

Fig.6 TEM picture – deformed part of the sample 128.2D

It is obvious from comparison of dislocation structure observed in the samples of the type 128.x with the structure observed in the samples of the type 139.x, that these structures differ. In the second type of samples cellular structure of dislocation walls was observed both in non-deformed parts (Fig. 7, 9 and 11) and in deformed parts of the sample (Fig. 8, 10 and 12). Comparison of diffraction from the area of dislocation wall and matrix does not show presence of another phase, e.g in the form of particles. Origin of this cellular structure of dislocation walls is connected with technology of preparation of samples (directional solidification).

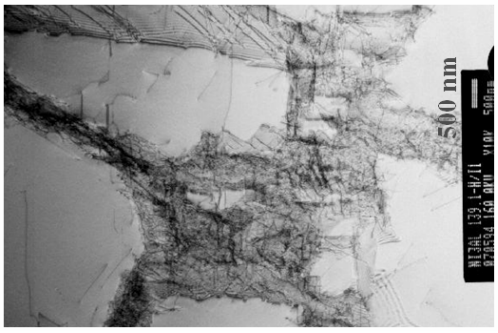

Fig.7 TEM picture – non-deformed part of the sample 139.1N

Fig.8 TEM picture – deformed part of the sample 139.1D

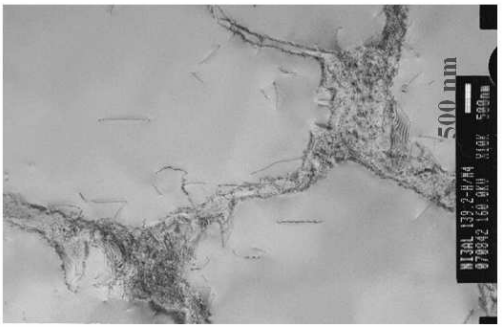

Fig.9 TEM picture – non-deformed part of the sample 139.2N

Fig.10 TEM picture – deformed part of the sample 139.2D

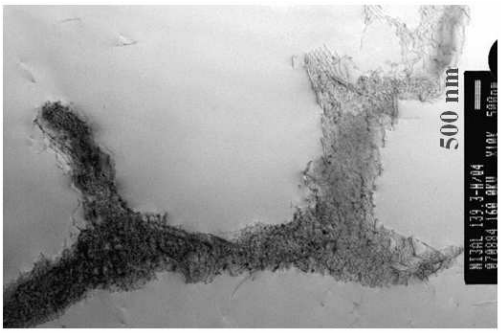

Fig.11 TEM picture – non-deformed part of the sample 139.3N

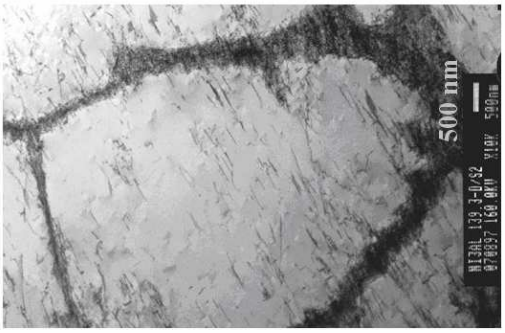

Fig.12 TEM picture – deformed part of the sample 139.3D

Character of dislocation structure

Dislocation density is homogenous both in non-deformed part of the samples 128, and in the deformed part of the samples (Fig. 6).

Presence of cellular structure of dislocation walls decreases dislocation density in the samples 139. Dislocation density in non-deformed part of these samples was not evaluated, however, it was obvious at analyses of the pictures (Fig. 7, 9 and 11) that dislocation density inside the cells is very low (several dislocations per one cell). The same phenomenon, i.e. lower dislocation density inside cells, was observed also in deformed parts of the sample, where the densities were evaluated – see the Table 4. Possible explanation of this phenomenon lies in presence of dislocation walls, which can be a place of dynamic recovery of dislocations inside cells. Another characteristic feature of this structure is greater scatter of dislocation density in comparison with dislocation densities from the area outside the cellular structure. This is in accordance with the possible explanation expressed above of lower density due to existence of dynamic recovery.

5. Evaluation

Within the frame of this work structural characteristics of the samples, prepared by Bridgman method on various equipment for directional crystallisation, were evaluated. The samples prepared in Vladimir show highly homogenous structure approaching the targeted required structure. Ductility of the samples 139.2 and 139.3 was determined to be 20 and 53 %. Their structure is formed by Ni_3Al matrix and cellular structure of dislocation walls in basic Ni_3Al matrix.

Moreover dislocation density in material before and after mechanical load was determined with use of transmission electron microscopy. Extent of the error at determination of dislocation density is max. 20%. In the samples 139 a cellular structure of dislocation walls was observed both in non-deformed parts and in deformed parts. Presence of cellular structure of dislocation walls decreased density of dislocations in the samples. Lower density of dislocations inside the cells is observed both in non-deformed and deformed parts of the samples 139. Another characteristic feature of this structure is greater scatter of dislocation in comparison with dislocation densities from the areas outside the cellular structure.

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