Rev.Adv.Mater.Sci. 25 (2010) 148-154

EVOLUTION OF MICROSTRUCTURE DURING LOW TEMPERATURE SUPERPLASTIC DEFORMATION OF BULK NANOSTRUCTURED Ni-Cr-Fe-Nb SUPERALLOY

V.A. Valitov[1], S.K. Mukhtarov[1], A.P. Zhilyaev[1,2] and O.A. Ruano[2]

[1]Institute for Metals Superplasticity Problems RAS, 39 Khalturin str., Ufa, 450001, Russia
[2]Centro National de Investigaciones, Metallurgicas (CENIM), CSIC, Avda. Gregorio del Amo, 8, 28040 Madrid, Spain

Received: December 18, 2009

Abstract. Ultrafine-grained and nanocrystalline structure have been obtained in Ni-Cr-Fe-Nb based alloy (INCONEL® 718) by multiple isothermal forging. The ultrafine-grained and nanostructured samples demonstrated low temperature (T=700 °C) superplastic behavior. Nanostructured alloy showed larger uniform elongation and reduced cavitation than those of Inconel 718 with microcrystalline structure. Electron backscatter diffraction was employed to reveal characteristics of structure, second particles distribution and grain boundary statistics.

1. INTRODUCTION

The most widely used superalloy is INCONEL® alloy 718. This alloy is utilized in high-temperature applications including aircraft engines and power generation. The heat-resistant properties of this alloy are due to a presence of coherent precipitates such as γ'' (Ni_3Nb, body-centered tetragonal) and gў ($Ni_3Al(Ti)$, face-centered cubic). The equilibrium structure corresponding to the γ'' phase is the high temperature modification of Ni_3Nb, orthorhombic incoherent δ-phase [1]. Some carbides are also present in the microstructure.

Various forming methods are used for the fabrication of parts from this hard-to-deform alloy. Superplastic forging and gas-pressure forming are widely used for alloy 718 with grain sizes of about 5-10 μm. Accordingly, much attention has been paid to the resulting microstructures and its relation to the superplastic properties [1-3]. It is well known that grain refinement to the nanoscale range enhances superplastic properties [4]. In the case of the NS material the (sub) grain size can be less than 100 nm [5]. Nanostructuring methods, as multiple isothermal forging (MIF) [4,6], can be used efficiently for hard-to-deform nickel based alloys. In particular, MIF [4,7] with step by step decreasing deformation temperature from 0.8 T_m to 0.54 T_m allows refining the coarse-grained structure into a NS one. For example, an uniform NS structure with grain sizes of about 80-100 nm was formed in the alloy 718 studied in present paper. Earlier investigations showed that this alloy reveals low temperature su-

Corresponding author: S.K. Mukhtarov, e-mail: shamil@anrb.ru

Table 1. Composition of Inconel 718 (wt.%).

Cr	Al	Ti	Fe	Nb	Mo	Co	B	C	Ni
19	0.5	0.9	18.5	5.1	3.0	0.1	0.025	0.04	Bal.

perplasticity (at 0.56 T_m) [4,8,9]. However, there are no extensive investigations of the influence superplastic deformation conditions (temperature and strain rate) on the microstructure evolution and phase composition. For this reason, the aim of the present work is to investigate the microstructure and properties during low temperature superplasticity of NS Inconel 718 alloy.

2. MATERIAL AND EXPERIMENTAL METHODS

The material selected for this study was hot-deformed nickel based alloy Inconel 718. The chemical composition of the alloy that was used in this work is shown in Table 1. Samples with sizes 40 mm in diameter and 60 mm in length of the alloy were machined from the 200 mm diameter.

Bulk samples (40-80 mm in diameter and 10-15 mm long), were processed on 100 and 630 ton presses using severe plastic deformation by MIF on flat dies at temperatures in the range 950 to 575 °C [4,8,9]. MIF is a process that provides accumulated plastic strains with little change in billet shape and size. The tests are carried out using hydraulic presses equipped with isothermal die-stack units with flat dies.

Superplastic properties were determined on flat samples, $12\times5\times2$ mm^3 in gage size. Tests were conducted over a wide range of temperatures (600-950 °C) and initial strain rates (1.5×10^{-4}-5.5×10^{-4} s^{-1}) using an Instron testing machine. The strain rate sensitivity, *m*, was determined by the method of strain-rate jump test and calculated from the formula [10]:

$$m = \frac{d \ln \sigma}{d \ln \dot{\varepsilon}}, \quad (1)$$

where σ is the flow stress, $\dot{\varepsilon}$ is the strain rate.

The investigation of surface relief has performed on polish specimens after superplastic deformation. SEM and OM images were taken from specimens etched with Marble's reagent (4 g $CuSO_4$, 20 ml water, 20 ml HCl).

Microstructural characterization was carried out using optical microscopy, scanning electron microscopy (SEM), JXA-6400, and transmission electron microscopy (TEM), JEM-2000EX. The dislocation density was measured as the amount of exit points on the foil surface [11]. Electron backscatter diffraction (EBSD) was carried out on a JXA-6400 equipped with HKL software.

3. EXPERIMENTAL RESULTS AND DISCUSSION

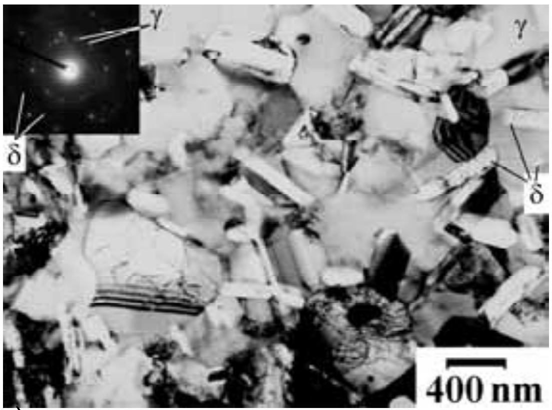
3.1. Initial microstructure

After MIF of bulk samples of Inconel 718 alloy with decreasing deformation temperature to 800 °C, a uniform microcrystalline structure with grain size about 1 μm was formed. Additional forging at decreasing temperatures of 700 and 575 °C resulted in a nanocrystalline grain size of 300 and 80 nm, respectively, as shown in Fig. 1. Electron diffraction patterns in the TEM showed that the duplex structure consists of γ+δ-phases (Fig. 1a). Diffraction spots from plane (221) of δ- phase and plane (111) of γ- phase were revealed. Grains of δ-phase with non-coherent boundaries were observed by morphological character on TEM image and distributed uniformly. The NS structure is characterized by high dislocation density and non-equilibrium boundaries, which are characterized by a diffuse contrast typical of nanocrystalline materials (Figs. 1b and 1c) [8,12].

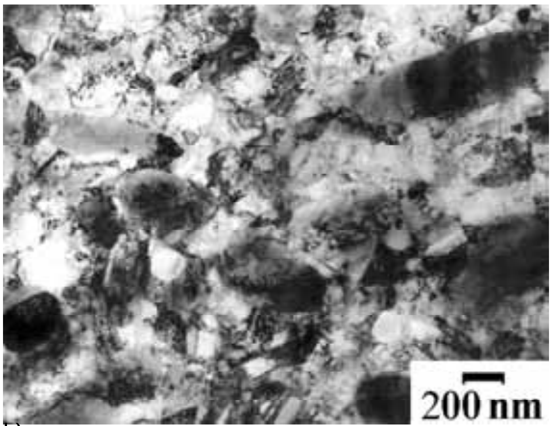
3.2. Superplastic properties

Results of superplastic characteristics are summarized in Table 2. In general, the range of temperatures where superplastisity is observed diminishes with decreasing grain size. The alloy with 80 nm displays superplastic behavior at 600 °C and strain rate of $1.5\text{x}10^{-4}$ s^{-1}. Under these conditions the elongation to failure was 350% and the strain rate sensitivity, *m*, was 0.37. Grain growth occured during the tensile testing.

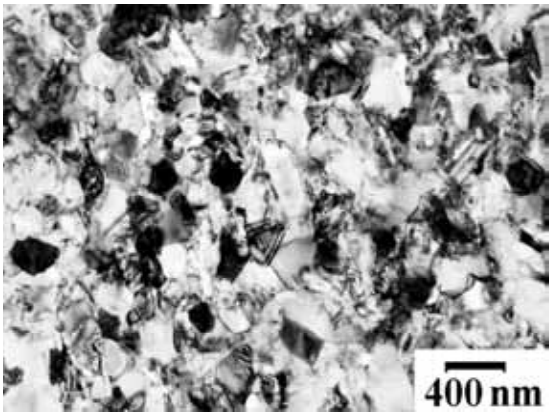
The superplastic properties of the alloys with average grain sizes 1 μm and 300 nm are summarized on Fig. 2 at the temperature range of 600-980 °C [9]. The figure shows the dependences of flow



a)



b)



c)

Fig. 1. TEM micrographs of the starting microstructure of Inconel 718 alloy with mean grain sizes: a) 1 μm; b) 300 nm; c) 80 nm.

strain and average grain size, *d* (Fig. 2a), elongation to failure, δ, and coefficient *m* (Fig. 2b) as a function of temperature. The strain of 40% has been taken as steady-state as a comparison for all temperatures [8]. It is evident that the NS alloy (*d*=300 nm) is less creep resistant and has higher elongation to failure and strain rate sensitivity than the MC alloy. The stresses at 950 °C of both alloys were similar that concerned with structure grain grows up to the same size (Fig. 2a). The NS alloy was achieved a higher ductility than MC alloy that depends on decreasing the grain size of alloy and, accordingly, increasing the grain boundary extension which raises the grain boundary diffusion during. The process of grain boundary sliding becomes easier that confirmed with higher strain rate sensitivity.

Table 2. Superplastic properties of bulk Inconel 718. The stress σ_{40} is the stress at plastic strain of 40%.

Grain (particle) size, μm		*Temperature*,°C	*Strain rate*, s^{-1}	σ_{40},MPa	δ,%	*m*
γ-phase	δ-phase					
1-2	0.15-0.6	950	$5.5x10^{-4}$	63	660	0.6
		800		193	350	0.3
0.3	0.1-0.3	900	$5.5x10^{-4}$	65	790	0.69
		800		123	580	0.5
		700		250	440	0.35
0.08		800	$5.5x10^{-4}$	128	323	0.4
		700	$3x10^{-4}$	230	580	0.4
		650	$3x10^{-4}$	310	342	0.34
		600	$1.5x10^{-4}$	414	350	0.37

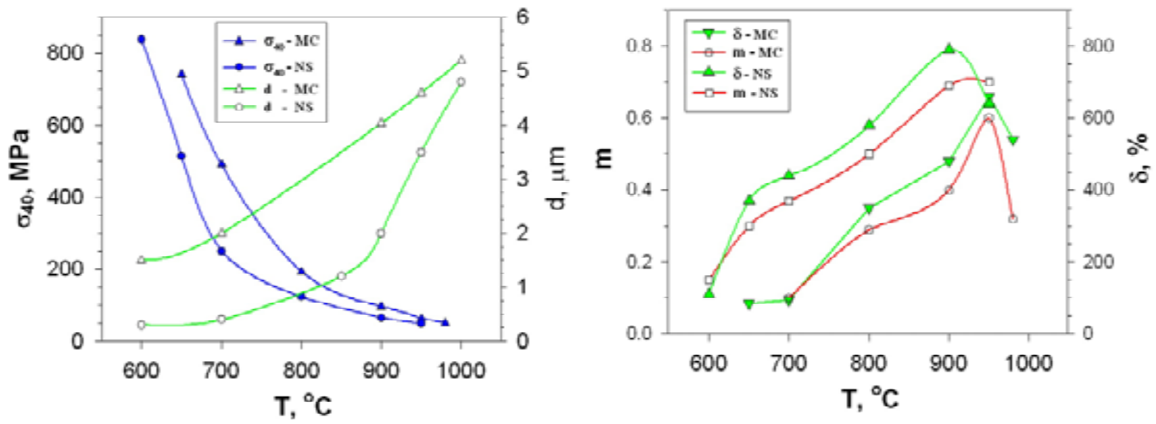


Fig. 2. Superplastic properties of Inconel 718 in the MC (d_{av}=1 µm) and NS (d_{av}=300 nm) conditions at a strain rate of 5.5x10^{-4} s^{-1}. The *m* value was calculated varying the strain rate by factor of 1.5.

3.3. Microstructure of samples subjected to superplastic deformation

Fig. 3 show TEM micrographs and electron-diffraction patterns of NS alloy (d_{av}=300 nm) after superplastic deformation in the temperature range 600 to 950 °C and strain rate 5.5x10^{-4} s^{-1}. The average grain size of γ-phase has not been changed (Fig. 3a) at temperatures in the range 600 to 700 °C (Fig. 3a)

Grain growth of γ-phase up to 0.6, 2 µm and some dissolution of δ-precipitates were observed with increasing deformation temperature from 800 °C to 950 °C (Fig. 3b). Thus after deformation at 950 °C the alloy becomes fine-grained. The increasing of the deformation temperature results in reduction of dislocation density in the grains interior and at the grain boundaries.

TEM study of microstructure evolution after superplastic deformation of NS Inconel 718 (d_{av}=80 nm) within the range 550-700 °C and strain rates 0.9x10^{-4} - 5.5x10^{-4} s^{-1} has been performed earlier [8]. It was shown that at 550 °C the average grain size has not changed. The microstructure consists of areas of recrystallized grains and areas with high dislocation density in grains interior and high fraction of δ-phase. With increasing deformation temperature from 600 to 700 °C, at strain rate 3x10^{-4} s^{-1}, the average grain size of γ-phase increases from 0.15 up to 0.3 µm, and the dislocation density reduces. More equilibrium grain boundaries were observed after superplastic deformation of the NS alloy.

Fig. 4a shows a SEM image of NS alloy subjected to superplastic deformation at 700 °C with strain rate 5.5x10^{-4} s^{-1}. The γ-phase is dark and Nb

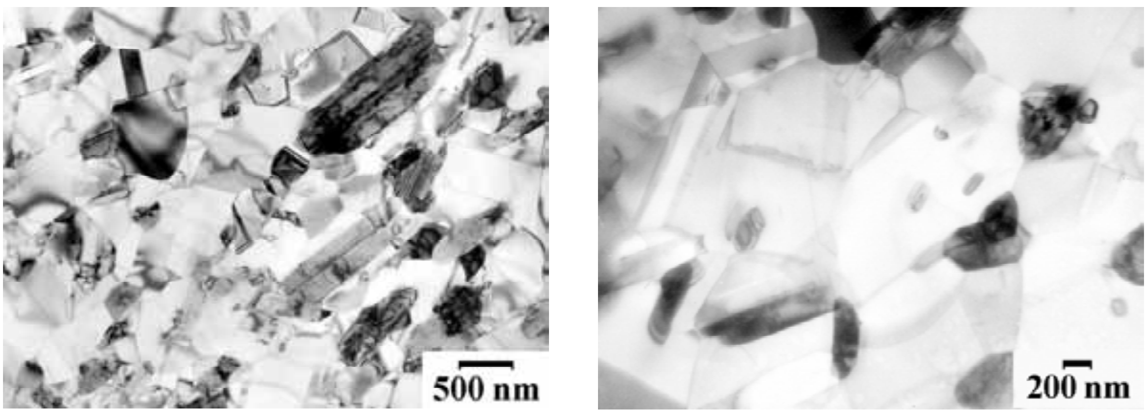
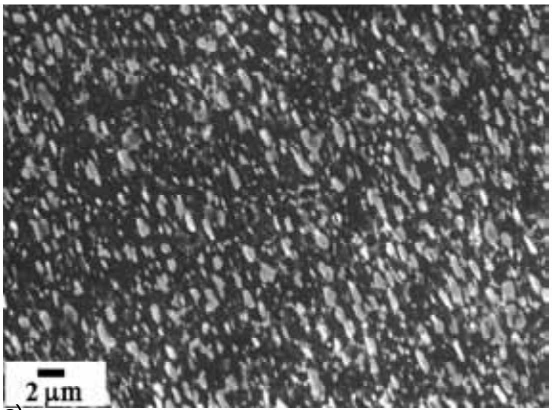
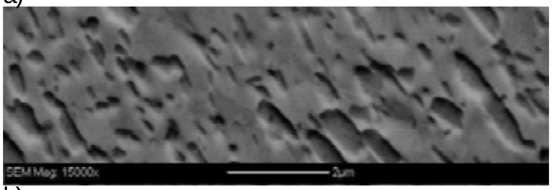


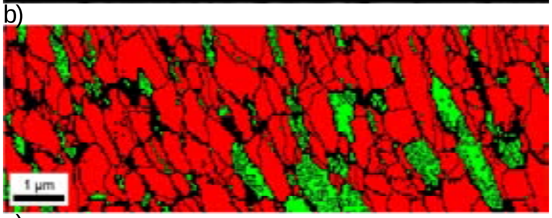
Fig. 3. Microstructure and electron-diffraction pattern of NS Inconel 718 (d_{av}=300 nm) subjected to tension at 5.5x10^{-4} s^{-1}: a) 700 °C; b) 800 °C; TEM.



a)

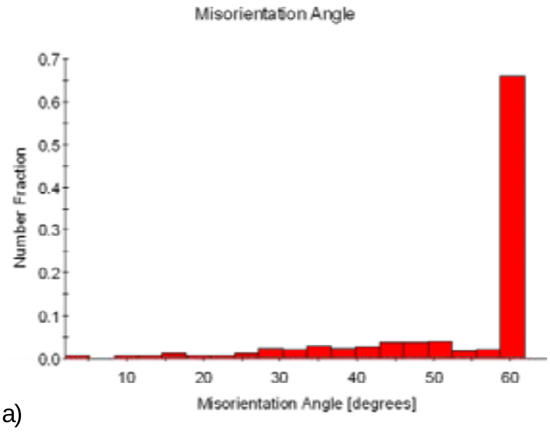


b)

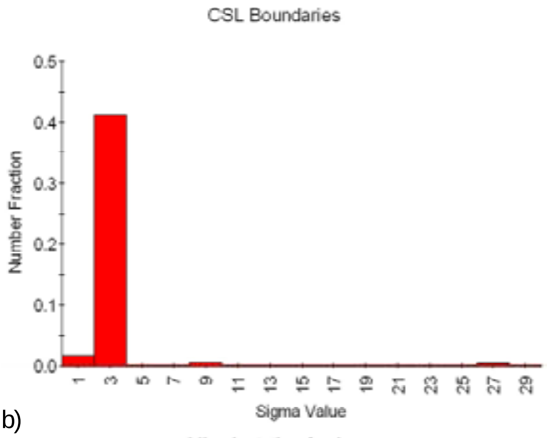


c)

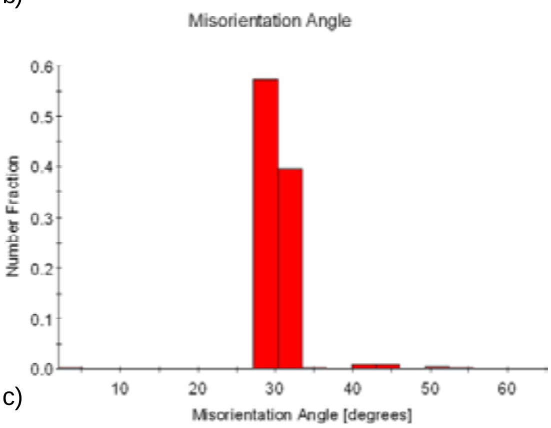
Fig. 4. Secondary electron image (a) and EBSD analyze results of NS Inconel 718 (d_{av}=300 nm) alloy subjected to tension at 700 °C at a strain rate of 5.5x10^{-4} s^{-1}: b) – map image; c) – EBSD map.



a)



b)



c)

Fig. 5. EBSD results analysis of NS Inconel 718 (d_{av}=300 nm) subjected to tension at 700 °C at a strain rate of 5.5x10^{-4}s^{-1}: a) – grain boundary distribution of γ-phase; b) – special boundary or coincident site lattice (CSL) boundary distribution of γ-phase; c) – boundary distribution in δ-phase plates.

- consisted δ - phase is bright phase. The amount of δ - phase is about 30%. EBSD analysis of the alloy shows that the grain size of the γ- phase is less than 1 micron. Figs. 4b and 4c are images of the same area. The very fine particles of δ - phase between large bright ones were not counted due to limitations of the step size during EBSD scanning (Fig. 4c). The calculated data shows that the structure has a large amount of high angle grain boundaries and twins. At the same time, most grain boundaries have a misorientation angle of about 60° (Fig. 5a), which corresponds to Σ3 or twins in γ- phase (Fig. 5b). The low stacking fault energy γ-phase shown a high fraction of Σ3 twin boundaries after deformation and annealing. These twin on the {111} plane of the FCC lattice and form at 60 degree [13].

Fig. 3a showed δ- phase plates containing twin-like boundaries. EBSD analysis of this phase revealed the presence of a large amount of high angle grain boundaries with a misorientation angle of about 30° (Fig. 5c). These may be twins that were observed earlier in the orthorhombic lattice [14,15]. Lastly, twin boundaries at about-30-deg rotation about [001] were observed. The 30-deg variant in-

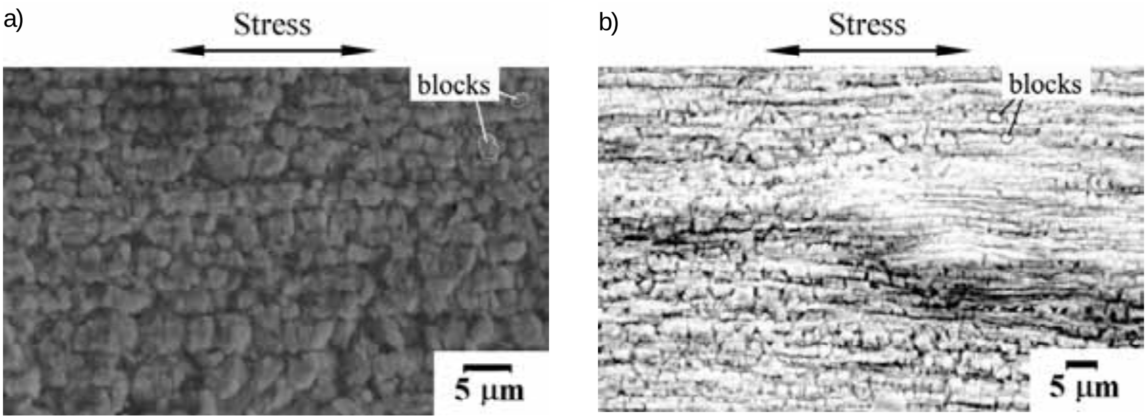


Fig. 6. Relief of Inconel 718 sample surface after superplastic deformation by tension: (a) 700 °C, strain rate – $3x10^{-4}$ s^{-1}, d_{av}=300 nm; (b) 600 °C, strain rate – $1.5x10^{-4}s^{-1}$, d_{av}=80 nm. SEM.

terfaces would be expected to be a preferred variant boundary type.

In the investigation of surface relief of NS samples with grain sizes 300 nm and 80 nm after superplastic deformation it was revealed that deformation bands divided the microstructure into separated blocks. The size of the blocks is about 2 to 5 times larger than the size of individual grains (Fig. 6). The smallest size of blocks was observed in the alloy with finer grains.

Cavitation was observed in the NS alloy (d_{av}=300 nm) subjected to superplastic deformation at 900 °C at a strain rate of $5.5x10^{-4}$ s^{-1}. Cavities and cavity linkage were revealed in the gauge length of samples and were aligned along the tensile direction. Only few cavities were observed at areas close to the grip section. The size and amount of the cavities, at the same conditions, are smaller than in the fine-grained alloy [9]. Separate cavities were founded far from the ruptured zone of the sample. The width of the cavities was less than 5 μm, the length was less than 8 μm and the length of cavity linkage was less than 70 μm (Fig. 7a). Nanocrystalline structure contains larger extension of grain boundaries than ultrafine-grained structure. Accommodation and diffusion processes during superplastic deformation promote cavitation. The different methods of structure investigation give the most reliable data about influence of superplastic deformation on structure evolution of NS alloy. During low temperature superplasticity a duplex structure with grain size less

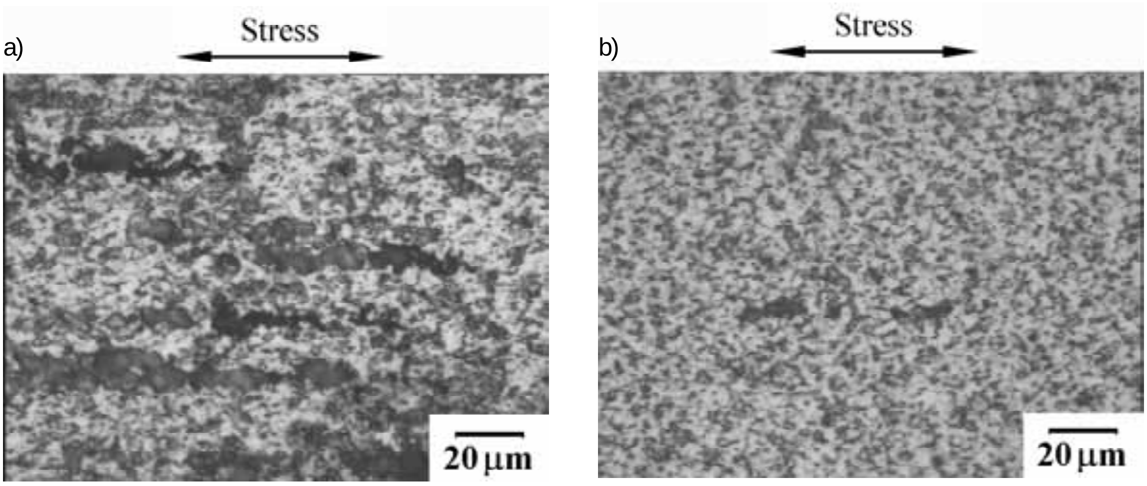


Fig. 7. Microstructure of NS Inconel 718 (d_{av}=300 nm) subjected to tension at 900 °C with strain rate $5.5x10^{-4}$ s^{-1}: (a) – sample's gauge zone; (b) – near sample's grip section. OM.

than 500 nm and high fraction of high angle and special boundaries in γ and δ -phases was observed. The investigation of surface relief and microstructure in samples after superplastic deformation shows promising applications of the NS alloy as better uniformity of deformation and reducing cavitation have been achieved. Fine and homogeneous microstructures that were present in the samples promote uniform elongation during superplastic deformation. The average size of cavity in NS alloy is smaller than in fine-grained alloy by approximately a factor of two. The investigation of the deformation behavior of the alloy with different microstructures revealed a superplastic behavior inherent at all microstructures. The processes of cooperative grain boundary sliding may have an importance in deformation [16]. Since grain growth occurred during deformation and due to the low temperatures investigated dislocation creep may play a role in the interpretation of the results. Formation of cooperative grain blocks was observed and the size of blocks in cross-section was approximately 2 to 5 times larger than the size of individual grains. It seems likely that a finer size of blocks provides more uniform deformation distribution along the cross-section.

4. CONCLUSIONS

1. Microstructure investigations of NS Inconel 718 show that microstructure remains in the NS range during low temperature (700 °C) superplastic deformation.
2. EBSD analysis of NS Inconel 718 (d_{av}=300 nm) subjected to tension at 700 °C at a strain rate of 5.5×10^{-4} s^{-1} shows that the microstructure has a large amount of high angle grain boundaries of about 60 ° and twins with special boundaries being sigma 3. The δ - phase plates contain a large amount of high angle grain boundaries of about 30°.
3. It has been established that NS microstructures formed in bulk semi-products from Inconel 718 provide a reduction in cavitation during superplastic deformation as compared with fine-grained structures.
4. The studies of the surface relief of Inconel 718 samples with NS microstructure after superplastic deformation have revealed the presence of blocks of grains. A more detailed investigation is necessary to determine the influence of these blocks on deformation.

ACKNOWLEDGEMENTS

This study was supported by Russian Foundation for Basic Research, Projects No. 07-08-00287 and No. 08-08-00387a. A.P. Zhilyaev thanks the Spanish Ministry of Science and Innovation for an R&C contract. The work was also partially support by MICINN (MAT2009-14452-C02-01) of Spain.

REFERENCES

[1] Y. Huang and T.G. Langdon // *J. Mater. Sci*. **42** (2007) 421.
[2] W.T. Chandler, A.K. Ghosh and W.M. Mahoney // *J. Spacecraft* **21** 1 (1984) 61.
[3] M.W. Mahoney, In: *Superalloy 718 - Metallurgy and Applications*, ed. by E.A. Loria (TMS, 1989), p. 391.
[4] V.A. Valitov, O.A. Kaibyshev, Sh.Kh. Mukhtarov, B.P. Bewlay and M.F.X. Gigliotti // *Mater. Sci. Forum* **357-359** (2001) 417.
[5] Y.T. Zhu, Z.L. Wang and T.G. Langdon // *J. Mater. Sci*. **42** 5 (2007) 1401.
[6] R.R. Mulyukov, A.A. Nazarov and R.M. Imayev // *Mater. Sci. Forum* **584-586** (2008) 29.
[7] F.Z. Utyashev, O.A. Kaibyshev and V.A. Valitov // *EP 0909339Â1. Method for processing billets from multiphase alloys*. (2001)
[8] V.A. Valitov, Sh.Kh. Mukhtarov and Yu.A. Raskulova // *Rev. Adv. Mater. Sci*. **11** 2 (2006) 159.
[9] Sh. Mukhtarov, V. Valitov and N. Dudova, In: *Superalloys 718, 625, 706, and Various Derivatives*, ed. by E.A. Loria (TMS, 2005), p. 507.
[10] O.A. Kaibyshev and F.Z. Utyashev, *Superplasticity: Microstructural Refinement and Superplastic Roll Forming* (Futurepast, Arlington, 2005).
[11] O.M. Gloer, *Practical Methods in Electron Microscopy* (Mashinostroenie, Leningrad, 1980).
[12] R.Z. Valiev and I.V. Aleksandrov, *Nanostructured materials obtained by severe plastic deformation* (Logos, Moscow, 2000).
[13] M. Kumar, A.J. Schwartz and W.E. King // *Acta Mater*. **50** (2002) 2599.
[14] S.P. Ge and K.H. Kuo // *Metall. Mater. Trans.* **A 30a** (1999) 697.
[15] D. Li and C.J. Boehlert // *Metall. Mater. Trans*. A **36a** (2005) 2569.
[16] O.A. Kaibyshev, A.I. Pshenichniuk and V.V. Astanin // *Acta Mater*. **46** 14 (1998) 4911.