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INFLUENCE OF SEVERE PLASTIC DEFORMATION AND HEAT TREATMENT ON MICROSTRUCTURE AND MECHANICAL PROPERTIES OF A NICKEL-IRON BASED SUPERALLOY

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Abstract. The present paper is a continuation of earlier studies devoted to the Alloy 718 processed by severe plastic deformation techniques such as high pressure torsion and multi-axial forging. Microhardness of the alloy in a nanostructured condition was found to be higher on 20-30% than that of the conventional coarse-grained counterpart. Tensile tests of the nanostructured alloy after multi-axial forging have shown very high strength and lower ductility. Fatigue tests at room temperature were performed for the nanostructured condition subjected to heat treatment and for the conventional coarse-grained condition. The tests revealed appreciably higher fatigue properties of the nanostructured alloy subjected to heat treatment in contrast to the conventional coarse-grained alloy. The obtained results have shown that the mechanical properties of the Alloy 718 in the nanostructured condition can be significantly improved using appropriate heat treatment.

1. INTRODUCTION

It is well known that nickel-iron based INCONEL® alloy 718 is widely used in aircraft engine building [1-2]. A specifically "tailored" version of INCONEL alloy 718 is INCONEL® alloy 718SPF with a grain size of about 10 μm, designed for the superplastic forming process and commercially produced in sheets and strips. As has been recently revealed that this alloy demonstrated superplastic behavior at 950-1000 °C and low strain rates. Particularly, this alloy showed elongation to rupture δ = 194% at temperature of 954 °C and an initial strain rate of 1.3×10^{-3} s^{-1} [3]. The elongation increased with a decrease in the strain rate exceeding δ = 450% as the strain rate was as low as 1.3×10^{-4} s^{-1}. In this case the strain rate sensitivity coefficient μ was measured to be 0.45 and low flow stresses typical of superplastic behavior (<28 MPa) were obtained. Superplastic properties of the Alloy 718 with the grain size of d = 6-8 μm were studied at 982 °C and a strain rate of 5×10^{-4} s^{-1} [4]. According to paper [4] the strain rate sensitivity coefficient was m = 0.5 and superplastic elongations as high as δ = 500% were attained. A study of the alloy with a smaller grain size, d = 4-5.6 μm (12-13 ASTM), carried out in [5] also confirmed superplastic properties of the alloy. A decrease in the alloy grain size leads to a decrease in the superplastic temperature. Particularly a nanostructured (NS) Alloy 718 produced by multi-axial forging (MF) and having a grain size of 0.08 μm showed superplastic properties at 700 °C [6]. At this temperature the strain rate sensitivity coefficient at the strain rate of 3×10^{-4} s^{-1} was m = 0.4 and elongation to rupture as high as δ = 580% was obtained. No doubt that superplastic forming is a good opportunity in respect of the hard-to-deform

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and difficult-to-machine superalloys like nickel-iron based superalloys. At the same time superplastic forming is only an intermediate processing step taking into account that final heat treatment should be performed to provide desirable mechanical properties including acceptable high-temperature capability. In other words, mechanical properties of the NS alloy subjected to final heat treatment are of importance. Note that the Alloy 718 is used after conventional heat treatment including solution annealing and precipitation hardening. The Alloy 718 is hardened by precipitation of the secondary phases γ"+γ' in the metal matrix [7]. The second phase's solution and precipitating can occur in the alloy during hot forging or severe plastic deformation [8,9]. The major strengthening γ" phase is dissolved at 650 °C and higher temperatures; the precipitation of the δ phase occurs within the temperature range of 750-1020 °C. At temperatures between 840 °C and 950 °C, the plates of δ form rapidly in times less than 24 h. The δ solvus temperature is about 1000 °C. It is known that the equilibrium δ-phase has an important influence on the mechanical properties of the alloy. Particularly it has been reported [8] that the δ-phase precipitates affected the stress-rupture, fatigue properties of the fine-grained alloy. In references [10-12] the comparison of mechanical properties of a duplex (γ+δ) Alloy 718 with different grain sizes obtained by MF was carried out at room temperature. The NS alloy with a grain size of d = 80 nm without any heat treatment had ultimate tensile strength about 50% higher (σ_{UTS}=1920 MPa) than that of the coarse-grained alloy (σ_{UTS}=1276 MPa). In addition, a reduction of ductility in the NS alloy by about 50% was detected. It should be noted that there are not data concerning the influence of the nanostructuring followed by heat treatment on the mechanical properties of the Alloy 718. Reasoning from this the aim of the present work is to evaluate the influence of severe plastic deformation followed by heat treatment on the microstructure and mechanical properties of the Alloy 718.

2. MATERIAL AND EXPERIMENTAL METHODS

The material selected for this study is hot-deformed nickel based Alloy 718. The chemical composition of the alloy is (in wt.%): 19.0Cr; 18.5Fe; 5.1Nb; 0.5Al; 0.9Ti; 3.0Mo; 0.1Co; 0.04C; 0.025B; Ni balance. The samples were machined from 200 mm diameter billet. Bulk NS samples were generated using severe plastic deformation techniques such as high pressure torsion (HPT) and MF. HPT was carried out through 5 turns at room temperature. The initial sample size was 4 mm in diameter and 0.7 mm thick. MF was performed on a hydraulic press equipped by isothermal die unit in the temperature interval of 950-575 °C using flat die tool. The forged workpieces had dimensions 60-80 mm in diameter and the thickness is 20-30 mm. The standard heat treatment of the forged material was carried out in accordance with the Aerospace Material Specification (AMS) 5662 [7]: solution annealing at 941-1010 °C±14 °C/1 hour, aging at 720 °C/8 hours, furnace cooling to 620 °C, holding at 620 °C for a total aging time of 18 hours. Microhardness measurements were carried out at room temperature. To do it, the loading of 50 g was used. The tensile tests were performed at room temperature on flat samples with a gauge section of 12 mm × 5 mm × 2 mm using the Instron testing machine. The tension-tension fatigue tests were determined on samples with a gauge section of 4 mm in diameter and 25 mm long using the Schenck HYDROPULS PSA 10 testing machine at room temperature. The axial fatigue tests were carried out at a frequency of 10 Hz and a strength 910 and 696 MPa for comparison with the results of the conventionally produced alloy. Other mechanical properties were tested on standard samples at room and elevated temperatures. The microstructure characterization was performed by means of JEOL JEM-2000EX transmission electron microscope (TEM), JXA-6400 scanning electron microscope (SEM) and Axiovert-100À optical microscope (OM).

3. RESULTS AND DISCUSSION

3.1. Microstructure of the as-received alloy

The microstructure was fully recrystallized, coarse-grained with a mean grain size of 40 μm. TEM studies have shown that coherent disk-type γ"-precipitates are uniformly distributed within γ matrix (Fig. 1). The diameter of γ"-phase disks is about 60 nm, and their thickness is 20 nm. The δ phase is not observed. Carbides with a mean size of about 5 μm are presented.

3.2. Microstructure of the alloy after severe plastic deformation

The study of the microstructure after shear deformation under HPT showed that the mean grain size is about 30 nm (Fig. 2.). This structure is characterized by the significant elastic stresses and a very

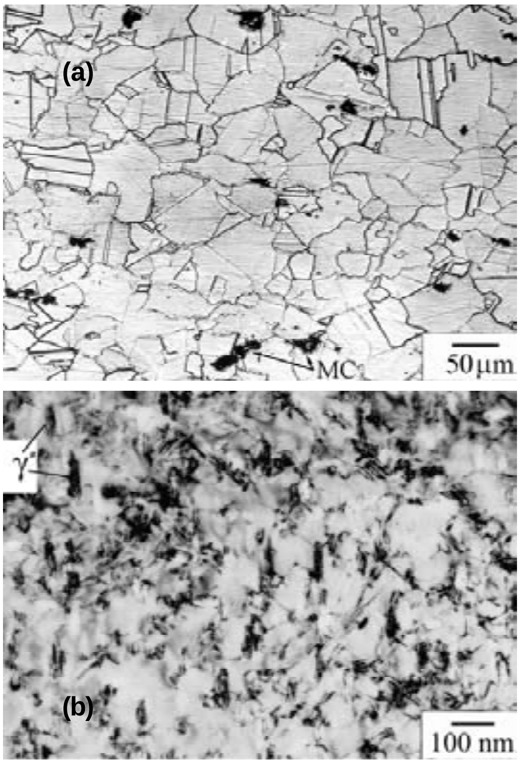

Fig. 1. Microstructure of as-received Alloy 718: a - OM image; b - TEM image of γ”-precipitates uniformly distributed within γ grain.

high density of grain boundaries [13]. During that deformation the metastable γ” phase can be dissolved [9]. After MF with reducing temperature of deformation down to 800 °C the fine-grained structure was formed. The uniform NS with a mean grain size of about 80 nm was formed in bulk samples subjected to MF with deformation temperature decreasing to 575 °C (Fig. 3). γ” phase was dissolved during MF and δ-phase plates with non-coherent boundaries were precipitated uniformly (Fig. 3a). SEM investigations have shown that the $\gamma+\delta$ duplex structure was formed. Mainly high-angle grain boundaries were presented in the structure. Many grains have high dislocation density (Fig. 3b).

3.3. Fracture surface of the tensile samples at room temperature

The tensile tests [10-12] of Alloy 718 with different structure obtained by MF showed that strength and ductile properties depend on a grain size when reducing the average grain size of alloy, the strength increases and ductility goes down. The fracture surface investigations of these samples at high magnification have demonstrated the picture typical of NS material with submicrometer-scale dimpled features (Fig. 4a) [14]. At low magnification of the fracture surface in Fig. 4b one can see the shear regions described in the overview [15]. The same result was observed in paper [16]. Rabinovich and Markushev show that the length of a crack path is less in fine-grained aluminium alloys. This path reduction proves that a smaller area of fracture surface and consequently less work is needed for crack growth. The effect of grain size on crack resistance is in the crack nucleation resistance increase and crack growth resistance decrease with grain refinement.

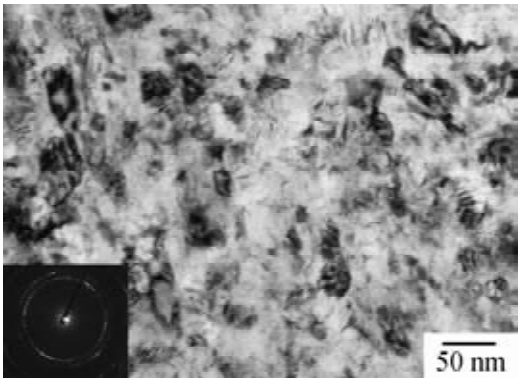

Fig. 2. Bright-field TEM micrograph of Alloy 718, processed by HPT. Inset shows the electron diffraction pattern.

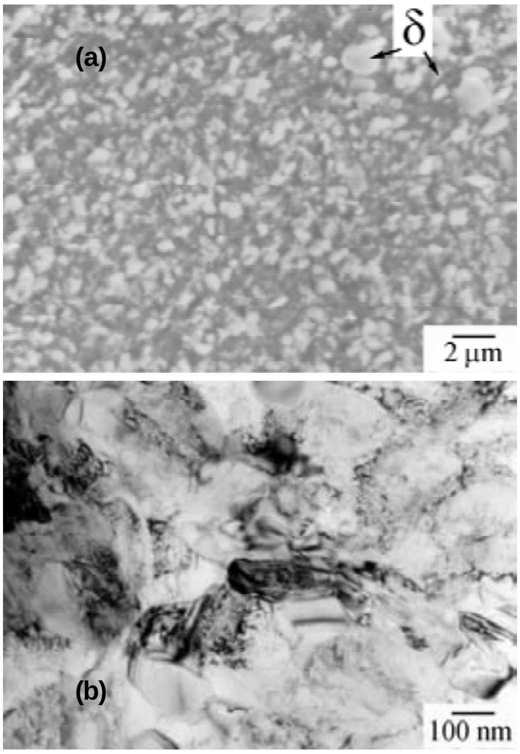

Fig. 3. Microstructure of NS Alloy 718 produced by MF: a - SEM image; b - TEM image.

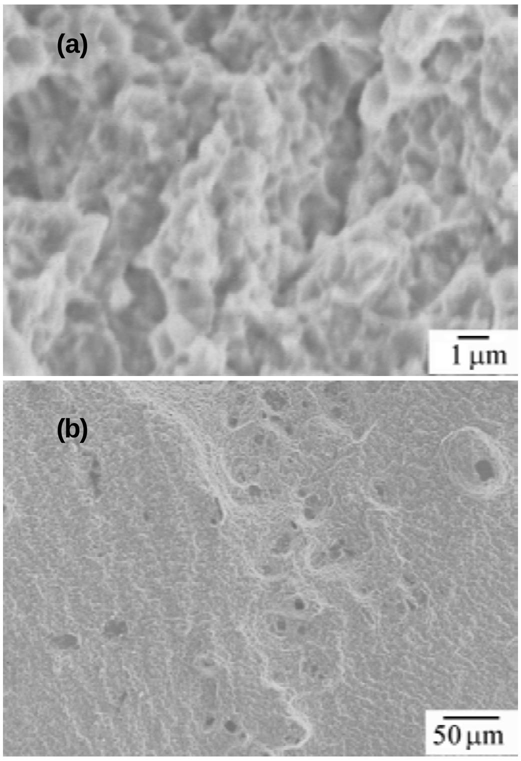

Fig. 4. Tensile fracture surface of NS Alloy 718 at different magnifications (SEM): a - ×3000; b - ×100.

3.4. Effect of severe plastic deformation on microhardness

The microhardness of the alloy in the coarse-grained state and nanostructured by HPT and MF is presented in Fig. 5. The obtained results show that severe plastic deformation increase alloy hardness. The sample subjected to MF with a grain size of about 80 nm has the highest value (6.7 GPa). Microhardness of the alloy subjected to HPT with a grain size of about 30 nm is lower (6.2 GPa) than that after MF. Therefore, the alloy microhardness after severe plastic deformation could be depends on not only on a grain size, but also a phase composition.

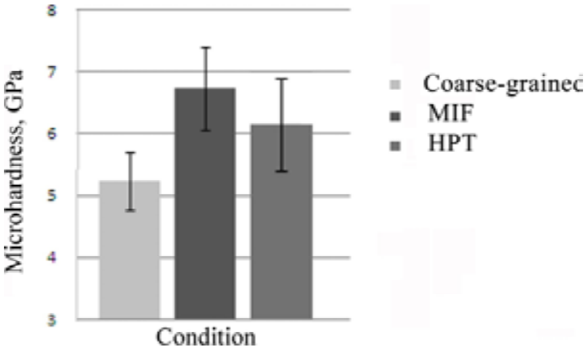

Fig. 5. Microhardness of Alloy 718 after different processes.

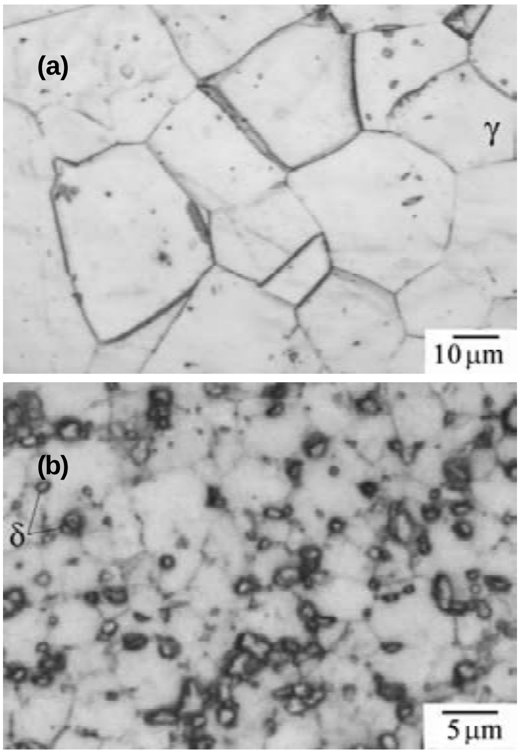

Fig. 6. Microstructure of NS Alloy 718 subjected conventional heat treatment (OM) with solution annealing at: a - 1000-1010 °C /1 hour; b - 980-990 °C /1 hour.

3.5. Microstructure and mechanical properties of the alloy after MF and conventional heat treatment

The study on microstructure after the standard heat treatment of NS alloy showed that the formation of a homogeneous structure and an average grain size of γ-phase depends on solution annealing temperature. After solution annealing for an hour at 1000-1010 °C, microstructure has average grain size of about 15 μm (Fig. 6a). The most of δ phase plates have dissolved because the annealing temperature above the δ solvus. If the solution annealing of NS alloy was carried out for an hour at 980-990 °C the γ phase average grain size would be of about 4 μm. Not all δ phase plates have dissolved during the annealing process at the temperature above (Fig. 6b). Volume fraction of δ-phase is 3.3% for NS alloy. The fine-grained and NS alloys after MF were subjected by conventional heat treatment with last annealing temperature. In this case the fine-grained structure was formed at both alloys. The microstructure after carrying out the heat treatment of fine-grained alloy showed that it resulted in the genera-

Table 1. Mechanical properties of Alloy 718 after heat treatment.

Alloy condition	Ultimate strength, [MPa]	Yield strength, [MPa]	δ,[%]	ψ,[%]	Grain sizes, [μm]	Fatigue strength	
						[MPa]	[Cycles]
AMS 5662 [7]	≥1276/ 1000*	≥1034/862	≥12/12	≥15/15	-	-	-
Coarse-grained + heat treatment [7,17]	1428/1176	1180/976	19/18	20/32	11-22	910 696	10^5 10^6
Fine-grained** + heat treatment***	1488/1169	1234/995	17/23	23/45	4.6	910 696	$>1.7\times10^5$ $>10^6$
NS** + heat treatment***	1520/1164	1252/993	19/21	35/42	4	910	$>1.7\times10^5$

* At room temperature/at 650 °C.

** After MF.

*** Solution annealing at 980-990 °C/1 hour, aging at 720 °C/8 hours, furnace cooling to 620 °C, holding at 620 °C for a total aging time of 18 hours.

Table 2. Stress rupture data at 650 °C of Alloy 718 after heat treatment.

Alloy condition	Stress, [MPa]	Time, [hrs]	δ, [%]	ψ, [%]
AMS 5662	689	≥23	≥4	-
NS+ heat treatment*	710	27.0	25.3	73.5

* Solution annealing at 980-990 °C/1 hour, aging at 720 °C/8 hours, furnace cooling to 620 °C, holding at 620 °C for a total aging time of 18 hours.

tion of uniform structure, the average grain size of γ-phase being 4.6 μm.

Table 1 presents the data on mechanical properties of the heat treated alloy. These data obviously meet the material specification requirements [7]. AMS 5662 data shows minimal required properties of alloy after conventional heat treatment. Tensile properties of the fine-grained and NS alloys after heat treatment are compared with properties of heat treated coarse-grained alloy that presented in work [17]. The data from Table 1 shows that the refinement of γ-phase grain size leads to some increase of strength at room temperature but some decrease of strength at 650 °C, the plasticity at these tests are about the same. It is clear that the original structure during the same treatment influence the structure being formed as well as its strength properties at room temperature. The alloy with the initial nanostructure after heat treatment has maximum strength at room temperature.

Fatigue strength tests of the samples at room temperature have shown that the properties of NS and fine-grained alloys after heat treatment on the base of 10^5 cycles are higher by the factor of 1.7 comparing to the data predetermined in the work [7]. The same tests have shown that fine-grained alloy properties on the base of 10^6 cycles are also higher.

Stress rupture data of NS alloy in comparison with the coarse-grain alloy are given in Table 2. All properties meet the material specification requirements. It is significant that NS alloy has increased ductility. All presented mechanical properties show that the presence of δ phase due to reduction of γ" phase and finer γ grains of structure lead to increase strength at room temperature. Smaller both grain size and concentration of γ" phase at this structure conduct to raise plasticity at 650 °C long term testing.

The obtained results show that the mechanical properties of the Alloy 718 in the nanostructured condition can be significantly improved using suitable heat treatment.

4. CONCLUSIONS

1. The γ+δ alloy subjected to MF with a grain size of about 80 nm has the maximum microhardness (6.7 GPa) at room temperature.
2. The solution annealing of NS alloy (d = 80 nm) and fine-grained alloy (d = 1 μm) for an hour at 980-990 °C leads to the γ phase grain size increase up to 4 and 4.6 μm, accordingly.
3. The NS alloy after heat treatment has maximum strength (σ_{UTS}=1520 MPa, σ_{02}=1252 MPa) and good plasticity at room temperature.
4. Fatigue strength tests of the samples at room temperature have shown that the properties of NS Alloy 718 subjected to heat treatment on the base of 10^5 cycles are higher by the factor of 1.7 than in conventional coarse-grained alloy.

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