

Microstructure and Properties of Nanostructured Alloy 718

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Abstract. Nickel-iron Alloy 718 is widely used for fabricating parts by superplastic deformation. Refinement of grains down to a nanostructure (NS) size improves the alloy's processing properties. Thermomechanical treatment has been carried out to form a NS state in bulk alloy by multiple isothermal forging (MIF) at gradually decreasing temperatures. Investigation of superplastic properties and processing behavior of Alloy 718 has been performed. The alloy with a grain size of 80 nm displays superplasticity (SP) at a temperature which is lower than for a conventional fine grained alloy by about 350°C. The values of the relative elongation δ and the strain rate sensitivity coefficient m are 350% and 0.37, respectively. The experimental data on the influence of grain size on solid-state weldability in the range of SP have been obtained. The application of the effect of low temperature SP yields lower temperatures of superplastic forming (SPF) and pressure welding (PW) as compared with conventional SP of fine-grained material. The experiment of the combined process of SPF and PW by counter-forming of two polished sheets, demonstrates its low temperature processing feasibility using NS specimens. The SPF processing of NS sheets in a cylindrical die has been investigated. It has revealed that macro-deformation is uniform in cross and longitudinal sections. Mechanical properties of Alloy 718 in NS condition and after strengthening heat treatment have been discussed.

Introduction

Realization of the highly efficient technology of complex geometry parts by low temperature SPF becomes possible due to ultrafine grained and NS material which provides high plasticity and low flow stress at the processing temperature [1]. NS condition provides widening of temperature-strain interval of SP [1] and decrease of flow stress which is important for hard-to-deform alloys. It is well known [1,2] that SP, particularly for NS material, creates unique potentialities for realizing SPF or/and PW with less power. Therefore the investigations for using low temperature SP for production of complex geometry parts from nickel based alloys are urgent.

The aim of the present work is to study the superplastic properties of Alloy 718 in NS condition and their influence on its formability, pressure weldability and mechanical properties.

Experimental Procedures

The chemical composition of the Alloy 718 used in this study is (in wt%): 19.0Cr; 18.5Fe; 5.1Nb; 0.5Al; 0.9Ti; 3.0Mo; 0.1Co; 0.04C; 0.025B; Ni balance. Samples were prepared from hot-worked rods 200 mm in diameter. For formation of NS condition the alloy was submitted to severe plastic deformation by the scheme of MIF as described in reference 3. Tensile tests of flat samples with 2 mm × 5 mm × 12 mm gauge dimensions were performed over the temperature range 650 – 950°C at the strain rates $1.5 \cdot 10^{-4} - 5.5 \cdot 10^{-3} \text{ s}^{-1}$ on a INSTRON-1185 testing device [4]. A NS forged billet was solution annealed at 980°C / 1 hour, aged at 720°C / 8 hours, furnace cooled to 620°C and hold at 620°C for a total aging time of 18 hours [5].

Characteristics of formability were studied using the SPF technique [6,7]. SPF of the polished NS sheet samples was performed at 900°C at the strain rate of 10^{-3} s^{-1} in a cylindrical die 30 mm in diameter. The depth of forming was 5, 10 and 15 mm. After forming, the samples were examined with the purpose to study the thickness and strain value distribution in longitudinal and transverse sections as well as features of deformation relief formation and microstructural changes.

An IMAS 20–78 equipment was used to conduct pressure welding [8]. The two rectangular samples with dimensions 5 mm × 5 mm × 10 mm placed one over the other, were submitted to deformation from 20 to 40% in vacuum in the temperature interval 750 – 950°C. Pressure welding was conducted at the initial strain rate of about $3 \cdot 10^{-4} \text{ s}^{-1}$.

SPF/PW was carried out on an experimental device in vacuum by the scheme of counter-forming two polished sheets with dimensions 40 mm × 40 mm and thickness 0.6 mm at 900°C for determining the possibility of this combined process.

Microstructure studies were performed with the use of optical microscope (OM) Axiovert 100A, scanning electron microscope (SEM) JSM–840 and transmission electron microscope (TEM) JEM–2000EX.

Results

Microstructure. The initial microstructure of the hot-worked Alloy 718 consists of a recrystallized, uniform grain structure with an average 40 μm grain size. TEM studies showed that, within the γ phase grains, there were disk-like γ'' phase precipitates which were uniformly distributed. The diameter of γ'' phase disks was about 60 nm, and their thickness was 20 nm. The γ'' phase precipitates were also observed on grain boundaries, as shown in Fig. 1a.

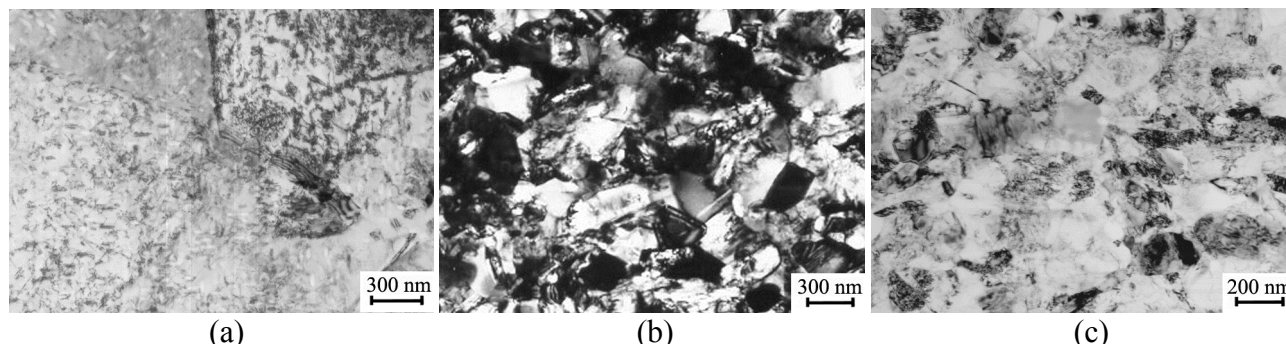


Fig. 1. Microstructure of Alloy 718: (a) - initial state; (b) - after MIF down to 700°C and (c) - 575°C; TEM.

After MIF of bulk samples of Alloy 718 with decreasing deformation temperature down to 800°C, a uniform $\gamma+\delta$ duplex fine-grained structure with a grain size of about 1 μm was formed. Additional forging at decreasing temperature down to 700°C and 575°C resulted in a NS grain size of about 300 and 80 nm, respectively (Fig. 1b, c) [3,4]. TEM and SEM showed that during MIF the metastable γ'' -Ni₃Nb phase transformed to the stable structure δ -Ni₃Nb.

Superplastic properties. The analysis of the test results on samples with fine-grained and NS structures has shown (see Table 1) [3,4,8] that the initial grain size exerts an essential influence on superplastic behavior of Alloy 718. It is seen that the alloy with a fine-grained structure ($d = 1 \mu\text{m}$) demonstrated superplastic behavior at 800°C. Additional structure refinement to a NS range allows the low temperature SP: NS alloy displays superplastic behavior at 650°C.

The forming was carried out at the temperature and the strain rate in accordance with superplastic properties (Table 1). All the processed samples are accurate in their shape and don't have any failure defects (Fig. 2). These samples were examined to study their thickness and strain distribution in longitudinal and transverse sections (Fig. 3). It has been established that, in NS samples, the distribution of thickness and strain is almost similar in longitudinal and transverse sections. Consequently one can conclude that, for all forming depth, the macro-deformation in

longitudinal and transverse sections occurs homogeneously and uniformly. Maximum deformation is observed in the angle zones of the samples, and is about 400% in the samples formed to the depth of 15 mm. According to the literature data the upper limit of ultimate strain at superplastic forming does not exceed 300% [6] in most cases. When analyzing the obtained data, one can certify that the processed NS Alloy 718 sheets exhibit sufficient formability for processing various complex shaped hollow parts by SPF. Moreover, the NS provides a deformation temperature decreased by 50 – 100°C as compared to the fine-grained material.

Table 1. Superplastic properties of Alloy 718.

σ_{40} : stress at 40% plastic strain; δ : elongation to failure; m : strain rate sensitivity coefficient.

Grain size [μm]		T [°C]	$\dot{\epsilon}$ [s ⁻¹]	σ_{40} , [MPa]	δ , [%]	m
γ phase	δ phase					
6	-	980	$5 \cdot 10^{-4}$	70	514	0.5
1-2	0.15-0.6	950	$5.5 \cdot 10^{-4}$	63	660	0.6
		900	$5.5 \cdot 10^{-3}$	226	215	0.31
		800	$1.5 \cdot 10^{-4}$	134	390	0.33
0.3	0.1-0.6	900	$5.5 \cdot 10^{-4}$	65	790	0.6
		800	$5.5 \cdot 10^{-3}$	292	270	0.3
		800	$1.5 \cdot 10^{-4}$	87	1095	0.54
		700	$3 \cdot 10^{-4}$	224	700	0.41
		650	$5.5 \cdot 10^{-4}$	514	370	0.3
0.08-0.1		700	$3 \cdot 10^{-4}$	230	580	0.4
		650	$3 \cdot 10^{-4}$	310	342	0.34
		600	$3 \cdot 10^{-4}$	752	253	0.35

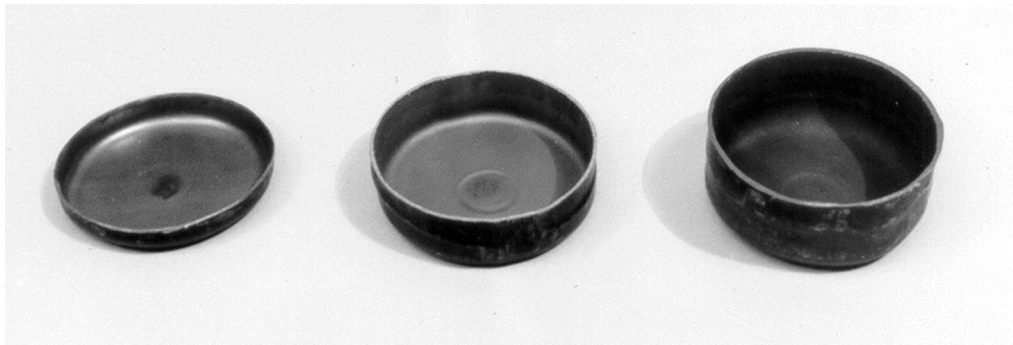


Fig. 2. Samples after SPF at 900°C at a strain rate of 10^{-3} s^{-1} to the depths 5, 10 and 15 mm.

The investigations of the samples surface after SPF allowed establishing the regularity of deformation relief formation. Fig. 4 shows a homogeneous micro-relief of the surface after SPF. The microstructure analysis of samples after SPF to the depth 5, 10 (Fig. 5) and 15 mm has shown that the microstructure is duplex ($\gamma+\delta$), homogeneous with equilibrium grain boundaries and twins, and corresponds to the structure in annealed condition. The density of dislocations inside a grain body is low. Grain boundaries have a clear fringed contrast.

The results of microstructure studies on NS samples submitted to SPF allowed to establish the influence of superplastic strain and hold time of SPF on the grain size. When increasing the equivalent strain at the forming temperature, a coarsening of grains occurs, with some dissolution of δ phase. After SPF to the depth 5 mm, 10 mm and 15 mm, the mean grain size of γ phase increases to 1 μm , 1.1 μm and 1.2 μm , respectively.

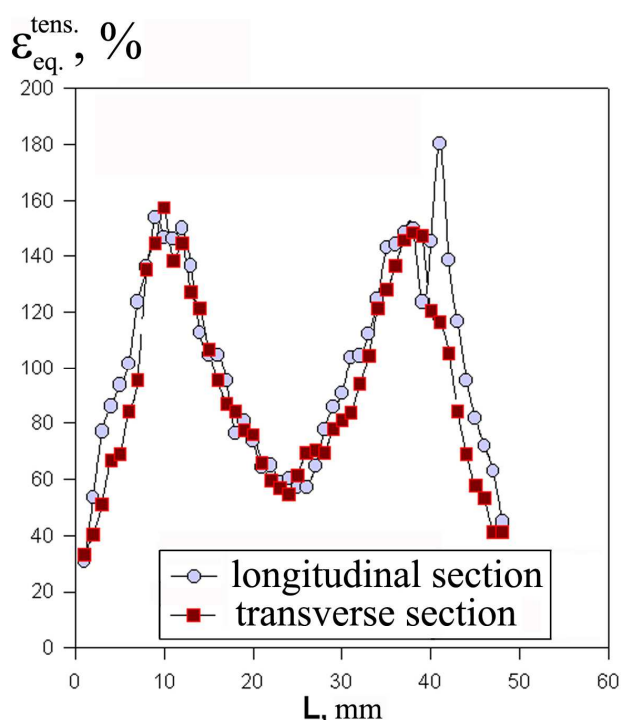


Fig. 3. Equivalent tensile strain distribution in two normal sections versus the profile length (L) for the sample formed to the 10 mm depth at 900°C and 10^{-3} s^{-1} strain rate.

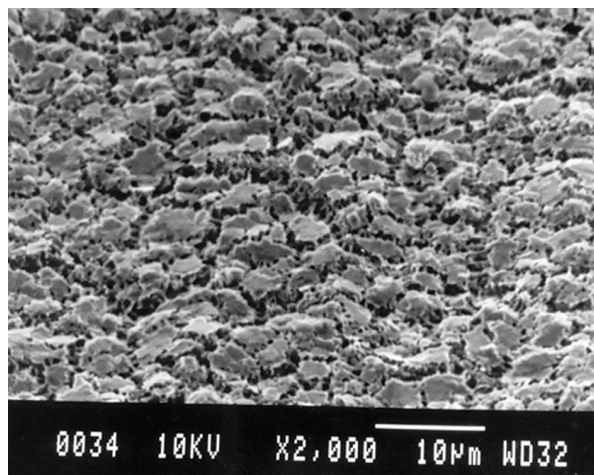


Fig. 4. Micro-relief of the Alloy 718 sheet surface after SPF at 900°C and 10^{-3} s^{-1} strain rate to the depth 10 mm, $\epsilon_{eq} = 150\%$.

Pressure welding. It has been shown that a sound solid-state joint (SSJ) free of pores can be processed in the coarse-grained alloy only at temperatures and strain rate higher than respectively 950°C and $3 \cdot 10^{-4} \text{ s}^{-1}$. A visual joint line is seen over the metallographic section. Pressure welding of the fine-grained alloy was successful at 800°C. The joint line is not visible (Fig. 6a). The SSJ processed by PW at 750–900°C and 20–40% strain has no visible interfaces even after etching of the cross-sectioned sample (Fig. 6b).

It is rather difficult to process homogeneous NS or fine-grained structure within the whole volume of real half-finished products. Therefore it is interesting to analyze the process of SSJ formation during the deformation of alloys with different structures. Sound joint between samples with coarse- and fine-grained structures can be processed at 850°C and 20% strain (Fig. 6c). Interfaces are revealed by the difference of etching between samples with different structures. The mean γ phase grain size in the coarse-grained portion of the sample is about 40 μm , and in the fine-grained portion the mean γ phase grain size is about 1.5 μm , and the δ phase about 1 μm .

Experimental testing of integrated SPF/PW. Processing behavior of sheet NS samples has shown that they can be used successfully in integrated SPF/PW technology. The possible temperatures of these processes are 850–900°C for SPF and 800–900°C for PW. Fig. 7 shows the feasibility of the SPF/PW technology using NS Alloy 718 sheet.

Mechanical properties of NS alloy and after heat treatment. Tensile properties of Alloy 718 after MIF with various grain sizes are shown in table 2 [9,10]. The NS ($\gamma+\delta$) alloy with a grain size of about 80 nm exhibits a very high room-temperature strength, which is much higher than that of the ($\gamma+\gamma''$) alloy submitted to the strengthening thermal treatment. It is obvious that the NS alloy

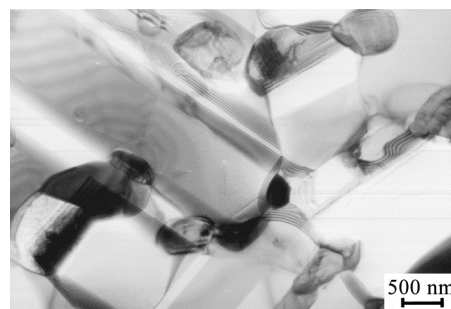


Fig. 5. TEM image of sheet NS Alloy 718 after SPF to the depth 10 mm.

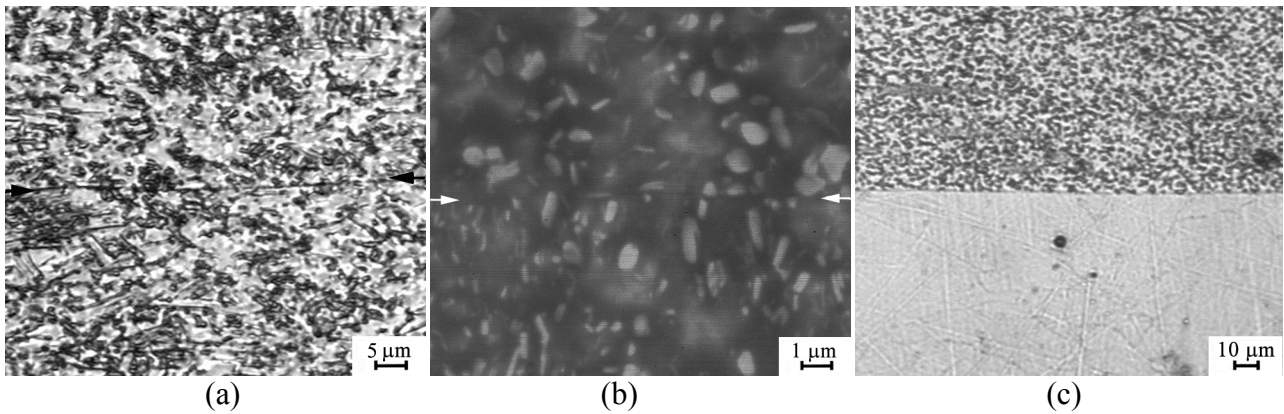


Fig. 6. SSJ of Alloy 718: at 800°C - FG/FG (a), OM; at 750°C - NS/NS (b), SEM; at 850°C - FG/CG (c), OM.

results in maximum strength at room temperature. However, the strength increase is accompanied by some reduction of ductility. The data of heat treatment for the NS alloy correspond to the Aerospace Material Specification (AMS) requirements.

The microstructure studies on the NS alloy [10] after carrying out the standard heat treatment have shown the generation of a uniform structure with an average γ matrix grain size of 3.9 μm . After heat treatment (HT), some amount of globular δ phase is preserved at grain boundaries, which retards grain growth during annealing. The volume fraction of δ phase is about 3%.

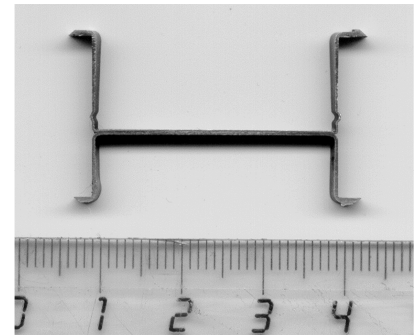


Fig. 7. The section of bonding sample after counter-forming of two NS Alloy 718 sheets.

Table 2. Mechanical properties of Alloy 718 at room temperature.
 δ : elongation to failure; ψ : reduction of area.

Alloy condition	Grain size [μm]	Phases	Ultimate tensile strength [MPa]	Yield tensile strength [MPa]	δ [%]	ψ [%]
AMS 5662 ^a	-	$\gamma+\gamma''$	≥ 1276	≥ 1034	≥ 12	≥ 15
NS	0.3	$\gamma+\delta$	1560	1300	5.1	11.0
NS	0.08	$\gamma+\delta$	1920	1845	4.8	6.1
NS+HT	3.9	$\gamma+\gamma''+\delta$	1520	1252	19	35

^a after heat treatment [5].

Discussion

Thus, according to experimental results, the additional refinement of structure down to a NS scale permits realization of low temperature SP. The use of NS semi-products allows reducing the flow stress by a factor 1.5 and temperature of forming during low temperature SPF.

The analysis of the superplastic welding results shows that the joint evenness of the fine-grained Alloy 718 is similar to the one observed in other alloys [2,7]. First of all it concerns the influence of grain boundary sliding (GBS) on enhanced solid-state weldability of the alloy. Really, two parts of a coarse-grained alloy can only be successfully joined at 950°C, while this can be done at 800°C with a fine-grained alloy. The main difference in the deformation mechanisms of alloys with different structures is evidently attributed to the significant increase of GBS contribution to the total deformation at the transition to the mechanism of SP. With decreasing temperature to 750°C the fine-grained alloy actually leaves the superplastic condition [4] that as it is known, is accompanied by reduction of the GBS contribution to the total deformation [1]. This exerts a negative influence

on the weldability of the alloy. On the other hand, the NS alloy exhibits SP at 750°C and this is correlated with its formability and solid-state weldability at this temperature.

It is known [1] that with decreasing grain size, the total length of grain boundaries increases, which actually explains the increased role of the GBS in the total deformation. The pressure welding experiments on samples with different initial structures also confirmed the possibility to decrease the pressure welding temperature by using a sample with a finer structure.

The revealed ability of Alloy 718 to solid-state welding in superplastic condition along with its known ability to SPF make it possible to use this alloy in advanced technologies combining superplastic deformation and pressure welding for processing bulk and hollow complex shaped parts, such as disks with blades, hollow blades and panels.

Summary

1. Forming of NS Alloy 718 sheets in a cylindrical die has been investigated at temperature-strain rate conditions of SP in the strain range up to 400%.
2. Microstructure investigation of samples after SPF at 900°C has revealed the grain growth from 0.3 μm up to 1.2 μm .
3. The experiments have shown that it is possible to process a sound SSJ in Alloy 718 with coarse-grained, fine-grained, NS alloys at temperatures 950°C, 800°C and 750°C, respectively.
4. The application of low temperature SP in NS Alloy 718 allows decreasing the temperature of solid-state weldability and formability by 50-150°C as compared to the conventional SP of a fine-grained alloy.
5. Grain size refinement of Alloy 718 to a NS scale results in a significant increase of its strength properties at room temperature; in particular ultimate tensile strength is increased up to 1920 MPa.
6. It is highly appropriate to employ NS Alloy 718 after heat treatment as it possesses high strength and sufficient ductility.

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