Superplastic behaviour and solid state weldability of a polycrystalline heavily alloyed nickel-base superalloy

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Abstract. Effective thermomechanical treatment has been developed for the heavily alloyed Ni-base superalloy produced via ingot metallurgy with a high content of γ’-forming elements. It allowed us to eliminate dendritic segregation and to obtain a uniform fine-grained structure with a γ grain size of d=5-15 µm. In the fine-grained condition the superalloy exhibited superplastic properties at 1125-1175°C (δ>300%, σ=10-27 MPa, m>0.3). Pressure welding experiments were performed at 1150°C for samples with the fine-grained structure. The solid-phase joint was formed only after compression to the strain of 50%. It was shown that insertion of a nanocrystalline interlayer of the same superalloy between the welded samples facilitated formation of the solid-phase joint.

1. Introduction
The development of aircraft gas turbine engines requires the development of new advanced heat-resistant materials, such as nickel-base superalloys, capable of operating at higher loadings and temperatures [1,2]. Computer methods are used to design new superalloys. One of the designing ideas in the development of new nickel based superalloys is associated with an increase of the γ’-forming elements (Al, Ti, Nb, Ta) content and alloying with substitution elements like rhenium, which could effectively strengthen the γ matrix [3]. Rhenium in an amount of typically 2-6 wt. % is mainly used as an alloying element in single crystalline superalloys of 2nd-6th generations [4-6]. In recent years, several polycrystalline powder metallurgy superalloys containing rhenium have been developed [7,8]. Data on mechanical behavior and technological properties of the rhenium alloyed nickel base polycrystalline superalloys are not readily available in the literature.

The present work was aimed to study the microstructure and technological properties of a heavily alloyed rhenium-containing nickel based superalloy produced via ingot metallurgy and subjected to thermomechanical treatment. The superplastic properties and solid state weldability of the superalloy in the fine-grained condition obtained by thermomechanical treatment were investigated.

2. Material and methods
The heavily alloyed nickel based superalloy with the nominal chemical composition Ni-30(Cr,Co,Re)-12(Al,Ti,Ta,Nb)-7.5(W,Mo) (in wt.%) manufactured by ingot metallurgy was taken as a starting material. The superalloy composition measured by energy dispersive X-Ray analysis was found to be...
very close to its nominal composition. The $\gamma'$ solvus temperature for the superalloys was defined as $T_s=1220^\circ$C. Thermomechanical processing for this superalloy was developed earlier in our previous work [9].

Microstructure examination was performed using scanning (SEM, BSE and SE) and transmission electron microscopy (TEM). The fine-grained forgings obtained by thermomechanical treatment were used to prepare flat samples with a gauge section $10\times3\times2$ mm$^3$ for tensile tests. The tensile tests were carried out in air without any protection against oxidation in the temperature range of $T=1100$-$1200^\circ$C with an initial strain rate of $\dot{\varepsilon}=5\times10^{-4}$ s$^{-1}$. Elongation to rupture, $\delta$, the true stress value $\sigma_{30}$ corresponding to 30% of elongation and true stress-elongation curves $\sigma-\varepsilon$ were determined. The true stress was calculated taking into account the gradual narrowing of the sample cross section during tensile testing. The strain rate sensitivity coefficient $m$ was defined at $1125$-$1175^\circ$C by varying the strain rate employed.

Solid state pressure welding experiments were carried out using the fine-grained cylindrical specimens Ø6 mm × 8 mm, which were cut out of the forged workpieces. Prior to welding experiments the specimen surfaces were mechanically polished so that the roughness of the welded surfaces was the same. Pressure welding experiments were conducted under isothermal conditions in the argon atmosphere at $T=1150^\circ$C using compression with the initial strain rate of $\dot{\varepsilon}=5\times10^{-4}$ s$^{-1}$ to an engineering strain of $\varepsilon=20$ and 50%. The pressure welding experiment under the same conditions was also performed using a foil (with a thickness of 0.2 mm) of the superalloy as an interlayer, which was obtained by high pressure torsion (HPT) at room temperature by 5 turns of a thin fine-grained specimen with an initial size of Ø10 mm × 1 mm [10].

3. Results and discussion

3.1. Microstructure characterization

The study of the microstructure evolution during thermomechanical treatment of this superalloy showed that the formation of a fine-grained structure occurred as a result of continuous dynamic recrystallization of the $\gamma$ phase [9]. After thermomechanical treatment, the microstructure was mostly recrystallized with a $\gamma$ grain size of 5-15 $\mu$m. There were also individual large grains with a size of 30-100 $\mu$m. In the course of the thermomechanical treatment, precipitation and coarsening of incoherent primary $\gamma'$ phase occurred along $\gamma$ grain boundaries as well. The size of the primary $\gamma'$ phase was $d_{\gamma'}=1-5$ $\mu$m, their volume fraction was defined as about 15% (figure 1a). Dispersed coherent particles of the secondary $\gamma'$ phase with a mean size of about 0.2 $\mu$m were precipitated within $\gamma$ grains during cooling after thermomechanical treatment (figure 1). The volume fraction of the $\gamma'$ phase was defined as 68% [9]. A high volume fraction of the $\gamma'$ phase resulted from a high content of the $\gamma'$-forming elements. The carbide particles were sometimes observed, their volume fraction was about 1.5%. HPT of the fine-grained state led to the formation of a nanocrystalline (partially fragmented) microstructure [10]. The nanocrystalline foil obtained via HPT was used as the interlayer to facilitate the diffusion bonding during pressure welding.

3.2. Superplastic behaviour

Figure 2 demonstrates the results of tensile tests. Superplastic elongations ($\delta$$>$$300\%$), low flow stresses ($10$-$27$ MPa) and a higher strain rate sensitivity coefficient ($m$$>$$0.3$) typical of superplasticity were obtained at $T=1125$-$1175^\circ$C and $\varepsilon'=5\times10^{-4}$ s$^{-1}$. The highest elongation ($\delta$$<$$400\%$) was reached at 1150 and 1175$^\circ$C. Microstructure examination of the sample tensile strained at 1175$^\circ$C detected $\gamma$ grain growth up to $d_{\gamma}$=15-20 $\mu$m (figure 3), which occurred as a result of grain boundary sliding and is typical of the superplastic behavior of fine-grained alloys. After tensile testing at 1125-$1150^\circ$C the $\gamma$ grain size was not appreciably changed. This suggests that dynamic recrystallization (first of all in relatively large $\gamma$ grains) occurred during superplastic flow at 1125 and 1150$^\circ$C along with the $\gamma$ grain growth resulted from grain boundary sliding. Therefore, the flow stresses at 1125 and 1150$^\circ$C slightly decreased as the elongation increased from 25 to 150%. Note that the flow stress changes could also be associated with minor dissolution and precipitation of the $\gamma'$ phase during tensile testing. This might occur because the
initial strain rate decreased gradually with increasing the sample length during tensile testing. The tensile test at 1200°C led to a fast γ grain growth already prior to testing because of dissolution of the γ′ phase. That resulted in rapid fracture of the sample without any narrowing (figures 2).

**Figure 1.** The microstructure images of the Ni-30(Cr,Co,Re)-12(Al,Ti,Ta,Nb)-7.5(W,Mo) superalloy in the fine-grained condition obtained by thermomechanical treatment: (a) SEM, BSE, (b) TEM, bright field. The arrows show the primary γ′ phase.

**Figure 2.** The temperature dependences of (a) the true stress vs. elongation and (b) the total elongation and the flow stress obtained as a result of tensile tests at T=1125-1200°C (έ=5×10⁻⁴ s⁻¹).

### 3.3. Pressure welding experiments

Pressure welding experiments showed that the solid-phase joint was formed at a temperature of T=1150°C only after compression to the strain of 50% (figure 4a), whereas the strain of 20% did not result in bonding formation. Even after compression to the strain of 50% separate voids were distinguished within the bonding zone (figure 4a). At the same time, the use of the nanocrystalline foil as the interlayer between the welded samples allowed decreasing the strain value required for bonding formation. After compression to the strain of 20% the diffusion bonding was formed, although some voids were observed along the welding joint (figure 4b). During pressure welding of samples with the nanocrystalline interlayer, a noticeable growth of γ grains and coarsening of γ′ particles occurred in the vicinity of the bonding zone that can be attributed to extensive operation of grain boundary sliding in the interlayer during compression. The growth of γ grains and coarsening of γ′ phase in the vicinity of the bonding zone occurred faster than in the base fine-grained material because of the strain localization within the nanocrystalline interlayer during compression. Apparently, this was a result of a high
thermodynamic non-equilibrium of the nanocrystalline interlayer obtained via HPT at room temperature.

Figure 3. The BSE image obtained from the gauge area near the fracture zone of the superalloy sample after tensile testing at T=1175°C (\( \varepsilon = 5 \times 10^{-4} \text{ s}^{-1} \)).

Figure 4. The BSE images of the solid-phase joints obtained by pressure welding of the superalloy samples at T=1150°C (\( \varepsilon = 5 \times 10^{-4} \text{ s}^{-1} \)): (a) \( \varepsilon = 50\% \); (b) \( \varepsilon = 20\% \) with using the nanocrystalline interlayer. The solid-phase joints are arrowed.

4. Conclusions
In the present work superplastic behavior and solid state weldability were investigated for the heavily alloyed superalloy Ni-30(Cr,Co,Re)-12(Al,Ti,Ta,Nb)-7.5(W,Mo) (wt.\%) in the fine-grained condition obtained via thermomechanical treatment. The Ni-30(Cr,Co,Re)-12(Al,Ti,Ta,Nb)-7.5(W,Mo) superalloy in the fine-grained condition demonstrated superplastic properties in the temperature range of 1125-1175°C (by 45-95°C below the \( \gamma' \) phase solvus temperature). High elongations (\( \delta = 350-400\% \)), low flow stresses (10-27 MPa) and increased strain rate sensitivity coefficient (\( m > 0.3 \)) typical of superplastic behavior of fine-grained alloys were observed in this temperature range.

The pressure welding experiments performed in the argon atmosphere for the fine-grained superalloy samples revealed extremely difficult formation of a sound solid-phase joint. More or less acceptable joint was attained only after compression at T=1150°C (\( \varepsilon = 5 \times 10^{-4} \text{ s}^{-1} \)) to the strain of \( \varepsilon = 50\% \). It was revealed that the use of the nanocrystalline interlayer (produced by HPT at room temperature) between the welded fine-grained samples improved the weldability and reduced the strain value required for the formation of the solid-phase joint down to \( \varepsilon = 20\% \).
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