Superplastic behavior of a nickel-base superalloy containing topologically close-packed phases

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Abstract. Superplastic behaviour of a polycrystalline nickel-base superalloy Ni-47(Cr,Co,W,Al,Ta,Hf)-0.2(C,B) (wt.%) in the fine-grained condition obtained via thermomechanical treatment (TMT) has been studied. TMT transformed a coarse-grained as-cast structure into a homogeneous fine-grained one. Both the initial as-cast and the thermomechanically treated conditions contained not only the matrix γ phase, the hardening γ′ phase and carbides but also topologically close-packed (TCP) phases. In spite of the presence of the TCP phases, the superalloy in the fine-grained condition demonstrated superplastic properties (δ>300% and low flow stresses) at subsolvus temperatures. Superplastic deformation led to some refinement and alignment of the TCP phase particles along the tensile axis.

1. Introduction
Development of advanced gas turbine engines is dependent on developing novel heat resistant materials, such as nickel-base superalloys, capable of operating at higher temperatures and loadings. In regard to the nickel-base superalloys, high alloying with the γ′ forming elements and the substitution elements for strengthening the matrix γ phase remain relevant [1]. However long-term operation of such superalloys (typically single-crystals or produced via directional crystallization) can lead to the formation of unfavorable topologically close-packed (TCP) phases [2-7]. At present, there are practically no data on the deformability of nickel-base superalloys containing TCP phases. The present work was aimed to study the microstructure and superplastic properties of a fine-grained nickel-base superalloy heavily alloyed with Co and W containing TCP phases.

2. Material and methods
The nominal composition of the superalloy was Ni-47(Cr,Co,W,Al,Ta,Hf)-0.2(C,B) (wt.%). The as-cast ingot with a size of Ø45 × 270 mm was manufactured and supplied by STC “Technologies of special metallurgy” Ltd, MISiS (Russia). The actual composition of the ingot was very close to its nominal composition. The γ′ solvus temperature (Tₕ) was determined via quenching experiments and shown to be equal to Tₕ ≈ 1185°C.

The as-cast superalloy was subjected to homogenization heat treatment followed by slow cooling to cause the coarsening of the γ′ phase and to improve the hot workability. After that, thermomechanical treatment (TMT) developed in our previous work [5] was carried out for small workpieces of the superalloy. The TMT consisted of unidirectional two-stage hot forging in the temperature range of
1100-1150°C with intermediate recrystalliation annealing. The forging procedure was carried out with the strain rate of \( \dot{e} = 10^{-3}-10^{-2} \text{s}^{-1} \), the total strain value during forging was \( e \approx 1.9 \).

The thermomechanically treated condition of the superalloy was used for tensile testing. To do it, the flat samples with a gauge section of \( 10 \times 3 \times 2 \text{ mm}^3 \) were cut from the fine-grained workpieces using spark cutting. All surfaces of the samples were finely ground before testing. Tensile tests were carried out at \( T = 1075-1150°C \) with an initial strain rate of \( \dot{\varepsilon} \approx 8.3 \times 10^{-4} \text{s}^{-1} \). The tensile tests were performed in air. Elongation to rupture \( \delta \), the true stress value \( \sigma_{30} \) corresponding to 30% of elongation were determined and the true stress-elongation curves \( \sigma-\varepsilon \) were drawn. The true stress was calculated taking into account the gradual narrowing of the sample cross section during tensile testing. The strain rate sensitivity coefficient of the flow stress \( m \) was determined by the strain rate switching method.

Microstructure examination was carried out on a scanning electron microscope (SEM) in the back-scattering (BSE) or secondary electron (SE) mode. To identify the elemental composition of the bright phases precipitated in the superalloy (TCP phases), energy dispersive X-ray analysis (EDS) was applied. Electron backscatter diffraction (EBSD) analysis was performed with a scan-step size of 0.1 \( \mu \text{m} \) to characterize the thermomechanically treated condition. EBSD analysis was conducted using the CHANNEL 5 processing software. The grain boundaries having misorientation angle less than 2° were not considered taking into account the measurement accuracy. The grain boundaries with misorientation angle more than 15° were assumed as high-angle ones. The specimen surfaces before studying by SEM were mechanically ground and polished.

3. Results and discussion

3.1. Effect of processing on the microstructure

Figure 1 a, b represents the microstructural images of the as-cast superalloy. One can see that the superalloy contains not only the \( \gamma \) and \( \gamma' (\text{Ni}_3\text{Al}) \) phases and carbides (rounded bright particles) but also TCP phases located predominantly along \( \gamma \) grain boundaries. The \( \gamma \) grain size was \( d_{\gamma} \approx 100 \mu \text{m} \). The \( \gamma' \) phase in the as-cast condition was mainly presented by precipitates with a size of 0.2-0.25 \( \mu \text{m} \) (figure 1b). The volume fraction of the \( \gamma' \) phase was defined to be about 60%. The volume fraction of carbides was not more than 1-2% due to the small amount of carbon. The TCP phases were observed along the \( \gamma \) grain boundaries in the form of elongated and equiaxied particles of 0.5-20 \( \mu \text{m} \) in size; its volume fraction was about 2.5%. Homogenization annealing led to precipitation of the TCP phases, which were uniformly precipitated as lamellar and equiaxed particles. The volume fraction of the TCP phases increased up to about 5% (figure 1c). The EDS analysis of the TCP phases revealed that they were enriched with W, Ta and Hf.

The TMT led to a uniform development of recrystallization processes in the forged workpieces that in turn led to the formation of a fine-grained microstructure with the mean \( \gamma \) grain size of \( d_{\gamma} \approx 5 \mu \text{m} \) (figure 1d) [5]. The primary \( \gamma' \) phase not dissolved during TMT had a size of 1-2 \( \mu \text{m} \) and occupied about 20 vol.\% (figure 1d). The secondary \( \gamma' \) particles with a size of about 0.25 \( \mu \text{m} \) precipitated within \( \gamma \) grains during cooling after TMT [5]. The lamellar TCP phases after TMT were partially globularized and aligned parallel to the material flow (figure 1d).

Figure 2 shows the EBSD orientation map and corresponding misorientation angle distribution for grain boundaries obtained for the thermomechanically treated condition. One can see that mostly high-angle boundaries were obtained after TMT showing that recrystallization processes occurred extensively throughout the forged workpiece. A small peak observed near misorientation of 60° corresponded to twin boundaries that were formed immediately after completing the forging processing (figure 2b).

3.2. Mechanical behaviour

Figure 3 shows the results of tensile tests of samples in the fine-grained condition obtained via TMT. High elongations (\( \delta = 300-380\% \)), low and nearly constant flow stresses (\( \sigma = 12-18 \text{ MPa} \)) and an increased strain rate sensitivity coefficient of the flow stress (\( m > 0.3 \)) typical of superplasticity were
achieved at $T = 1100-1150^\circ C$ and $\dot{\varepsilon} = 8.3 \times 10^{-4}$ s$^{-1}$. The highest elongations ($\delta = 350-380\%$) were reached at 1100 and 1125°C. Tensile testing at 1075°C gave much lower elongation and higher flow stresses. Apparently, this tensile test did not respond to the superplastic temperature-strain rate conditions and, therefore, elongation was much smaller than 300% (figure 3).

Microstructure examination of the gauge sections of samples strained in tension at 1100-1150°C showed that $\gamma$ grain growth occurred after superplastic deformation. For instance, after tensile straining at 1100°C the mean $\gamma$ grain size grew up to $d \approx 7 \mu m$ (figure 4). The grain growth is typical of superplasticity of fine-grained alloys and results from the grain boundary sliding, which occurs in the course of the superplastic deformation. The size of the prime $\gamma'$ phase after superplastic elongation was found to be the same as prior to tensile testing and did not exceed 2 $\mu m$. Secondary $\gamma'$ phase precipitated after superplastic deformation and had also near the same size as before tensile testing. The carbides and particles of the TCP phases were slightly refined and aligned along the tensile axis. The length of lamellar TCP precipitates decreased after superplastic deformation and did not exceed 15 $\mu m$. Fragmentation of the lamellar TCP phase particles promoted the formation of voids near the fracture zone of the superplastically strained samples. Most likely, this provoked premature failure and limited the superplastic elongation. Nevertheless, in spite of the presence of the TCP phases, the fine-grained condition provided superplastic properties of the superalloy at subsolvus temperatures ($T = 1100-1150^\circ C$).

![Microstructure images](image-url)

**Figure 1.** The microstructure images of the superalloy: (a, b) the as-cast condition (BSE), (c) the cast condition subjected to homogenization heat treatment (SE), the condition obtained after TMT (BSE).
Figure 2. Normal-direction EBSD (inverse-pole-figure) map (a), and the corresponding misorientation-angle distribution for grain boundaries (b) obtained for the superalloy after TMT. The forging axis is vertical, high- and low-angle grain boundaries are indicated by black and white lines, respectively.

Figure 3. The temperature dependences of (a) the true stress vs. elongation and (b) the total elongation and the flow stress ($\sigma_{30}$) obtained as a result of tensile tests at $T = 1075-1150^\circ$C ($\dot{\varepsilon} = 8.3 \times 10^{-4}$ s$^{-1}$).

Figure 4. The BSE images obtained from the gauge area near the fracture zone of the superalloy sample after tensile testing at $T = 1100^\circ$C ($\dot{\varepsilon} = 8.3 \times 10^{-4}$ s$^{-1}$). The primary $\gamma'$ phase, TCP phases and carbides are arrowed.
4. Conclusions
The heavily alloyed nickel-base superalloy containing TCP phases was subjected to homogenization heat treatment and thermomechanical treatment, which led to a uniform occurrence of recrystallization processes and formation of a fine-grained structure in the forged workpieces. In spite of the presence of the TCP phases, the superalloy in the fine-grained condition exhibited superplastic behavior at subsolvus temperatures. In the temperature range of 1100-1150°C at strain rate of \( \dot{\varepsilon} \approx 8.3 \times 10^{-4} \) s\(^{-1}\), the fine-grained samples of the superalloy showed high elongations (\( \delta = 300-380\% \)), low and nearly constant flow stresses (\( \sigma = 12-18 \) MPa) and increased values of the strain rate sensitivity coefficient (\( m > 0.3 \)). After superplastic deformation the TCP phase particles were refined and aligned along the tensile axis.

Acknowledgments
The present work was funded by the Russian Foundation for Basic Research (Research Project No 18-08-00997-a). The work was performed using the facilities of the shared services center «Structural and Physical-Mechanical Studies of Materials» at the Institute for Metals Superplasticity Problems of Russian Academy of Sciences.

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