Microstructure evolution of a Re-containing nickel-based superalloy by hot deformation and cold severe plastic deformation

Sh Mukhtarov, R Shakhov and A Ganeev
Institute for Metals Superplasticity Problems RAS, 39, Stepan Khalturin, Ufa, 450001, Russia
E-mail: shamil@anrb.ru

Abstract. The microstructure evolutions of the cast rhenium containing nickel-based superalloy subjected to thermomechanical treatment and severe plastic deformation by high pressure torsion were investigated. Thermomechanical treatment led to the formation a fine-grained structure with γ grain size of about 7 μm and both large precipitates of the γ’ phase with a size of 1-5 μm and dispersed precipitates with a size of about 0.2 μm. Microstructure of the superalloy subjected to high pressure torsion up to 5 revolutions was nanocrystalline with partly dissolved of the dispersed γ’ precipitates. The effect of different kinds of deformation and subsequent annealing on the microstructure, grain boundaries and microhardness changes are also discussed.

1. Introduction
Improving the chemical composition of nickel-based superalloys for an increase in operating temperature of gas turbine engines leads to a decrease in their deformability. Nickel-based superalloys are multiphase materials, which usually consist of the γ matrix phase, carbides and dispersed hardening phases as γ’ phase, γ”, δ and others [1]. Deformation processing of superalloys can lead to recrystallization of the γ matrix phase, dissolution and precipitation of dispersed phases during high temperatures [1] and the same thing occurs by severe plastic deformation at room temperature followed by annealing [2]. The development of new superalloys goes towards increasing the content of refractory elements [1], which leads to a decrease in the diffusion rate and affects the development of recrystallization. The recently developed superalloy contains of about 2 wt. % of rhenium and its chemical composition is similar to some of the second generation single-crystal superalloys. The deformation behavior, recrystallization of such complex superalloys has not been sufficiently studied. Previous studies [3] showed that a decrease in the size of the recrystallized γ grains of the investigated superalloy to 3-20 μm led to an increase in mechanical properties both at room and at elevated temperatures. The formation of a smaller γ grain size in this superalloy has not been previously obtained. In this regard, it is relevant to study the evolution of the microstructure during deformation of both hot deformation and severe plastic deformation by high pressure torsion at room temperature.
2. Material and methods

The material selected for this study was precipitation hardened with the strengthened phase $\gamma'$ (fcc, Ni$_3$Al) cast nickel-based superalloy Ni-12.5(Al,Ti,Nb,Ta)-37(Cr,Co,W,Mo,Re)-0.17(C,La,Y,Ce,B) (in wt.%). The ingots produced by induction melting had a diameter of 40 mm and a height of 15 mm. The temperature of the full dissolution $\gamma'$ phase was measured via water quenching experiments and correspond to $T_s=1220 \pm 5$ °C. The superalloy ingots were subjected to: i) heterogenization annealing in the temperature range of ($T_r$-120)+($T_r$+20) followed by slow cooling with a rate of 25 °C/min; ii) rapid cooling in air from temperature $T_r$+20. The sample subjected to annealing was compressed to an engineering strain of $\varepsilon=50\%$ with an initial strain rate of $5 \times 10^{-4}$ s$^{-1}$ at a temperature of 1175 °C and then cooled in air [3]. The samples with dimension 8 mm in diameter and 0.7 mm in height were cut out of the superalloy. These small discs were processed by HPT up to 5 revolutions at room temperature and annealed at 700 °C for 2 hours in an airtight casing.

The microstructure investigation was carried out using a Tescan Mira 3 scanning electron microscope (SEM) in backscattering electron (BSE) mode and a JEOL JEM-2000EX transmission electron microscope (TEM). Electron backscatter diffraction (EBSD) analysis was performed with a scan-step size of 3 µm from central parts of the deformed sample. The grain boundaries with misorientation more than 15° were assumed as high-angle ones. The grain boundaries having a misorientation angle of less than 2° were excluded from consideration. Vickers microhardness tests were carried out using an AFFRI DM8A digital microhardness tester at a load of 100 g and a holding time of 10 seconds.

3. Results and discussion

3.1. Microstructure of the superalloy in as-cast condition and subjected to annealing

The superalloy in the as-cast condition was characterized by a coarse-grained structure with a dendritic segregation and non-equilibrium eutectic (figure 1 a). The dispersed $\gamma'$ phase homogeneously precipitated within the $\gamma$ phase. Carbides are uniformly distributed in the microstructure with a size of about 10 µm. The as-cast superalloy was subjected to heterogenization annealing in order to improve hot workability. The annealing led to the dissolution of the nonequilibrium eutectics and some coagulation of the $\gamma'$ phase (figure 1 b). The annealing also resulted in a partial loss of coherence between the $\gamma'$ phase and the $\gamma$ matrix. Heterogenization annealing led to coagulate of $\gamma'$ particles up to 5-10 µm (figure 1 b).

![Figure 1. SEM images of cast superalloy Ni-12.5(Al,Ti,Nb,Ta)-37(Cr,Co,W,Mo,Re)-0.17(C,La,Y,Ce,B): (a) – as-received condition; (b) – subjected to heterogenization annealing in the temperature range of ($T_r$-120)+($T_r$+20) followed by slow cooling with a rate of 25 °C/min.](image-url)
3.2. Microstructure evolution of a Re-contain nickel-based superalloy by hot deformation

Figure 2 shows the microstructure and EBSD map obtained from the central part of the compressed sample of the superalloy. The recrystallization processes occurred extensively at 1175 °C in the central part of the sample. The average size of the recrystallized γ grains in the small central part was down to 7 µm (figures 2 a). In the microstructure there were both coarse γ' grains with a size of 1-5 µm, which were not dissolved during high temperature heating before hot deformation, and dispersed γ' precipitates with a size of 0.15-0.25 µm (figure 2 a) that formed during air cooling.

EBSD analysis of the central part of the compressed sample revealed that the first stage of the deformation led to the formation of a developed substructure, which consisted of subgrains (top and bottom part of figure 2 b). An increase in the strain value led to the occurrence of recrystallization along the initial γ grain boundaries, and then near completely recrystallized microstructure was formed. The size of recrystallized γ grains was in the range of 5-50 µm. The large grains contained low-angle boundaries. The main mechanism for the microstructure refinement during compression at 1175 °C was defined as continuous dynamic recrystallization. Therefore, the hot deformation of cast rhenium containing nickel-based superalloy showed the possibility of recrystallization behaviour that was generally similar to that of other nickel-based superalloys.

Figure 2. Microstructure and EBSD map of superalloy Ni-12.5(Al,Ti,Nb,Ta)-37(Cr,Co,W,Mo,Re)-0.17(C,La,Y,Ce,B) subjected to heterogenization annealing and following hot deformation at the temperature (T=25) to strain of ε=50% with an initial strain rate of 5×10^{-4} s^{-1}: (a) – BSE image; (b) – normal-direction EBSD (inverse-pole-figure) map, the compression axis is vertical, high- and low-angle grain boundaries are indicated by black and white lines, respectively.

3.3. Microstructure evolution of a Re-containing nickel-based superalloy by severe plastic deformation and annealing

The investigation of cast superalloy subjected to rapid cooling from high temperature showed a coarse grained microstructure with γ grain size of about 300 µm and dispersed γ' precipitates. The superalloy Ni-12.5(Al,Ti,Nb,Ta)-37(Cr,Co,W,Mo,Re)-0.17(C,La,Y,Ce,B) in three conditions: i) cast condition; ii) cast alloy after heterogenization annealing; and iii) cast alloy after rapid cooling were subjected to HPT up to 5 revolutions. The studies of the microstructure revealed that HPT of samples with different initial conditions led to refined of the microstructure to nanosized. The average grain size was about 30 nm (figure 3 a). Studies of the fine structure revealed that the γ' phase did not dissolve in all conditions. Figure 3 b shows a TEM image of the sample subjected to heterogenization annealing followed by HPT and annealing at 700 °C for 2 hours. The average γ grain size increased from about 30 to 50 nm independently of the initial condition. Annealing twins were detected within some of the grains.

3.4. Effect of HPT and annealing on the microhardness of a Re-containing nickel-based superalloy

The microhardness of the superalloy Ni-12.5(Al,Ti,Nb,Ta)-37(Cr,Co,W,Mo,Re)-0.17(C,La,Y,Ce,B) in different conditions is represented in figure 4. The microhardness increases up to 6.4-6.7 GPa after
HPT. This is due to the refinement of the microstructure down to the nanocrystalline level. The microhardness increase up to 9.3-9.8 GPa for all conditions after subsequent annealing, is probably associated with the onset of polygonization that occurred before recrystallization [4]. The microhardness of the superalloy subjected to heterogenization annealing following by HPT and annealing at 700 °C is slightly higher than the microhardness of other conditions probably because of the presence of large γ' precipitates that not dissolved during HPT.

![Figure 3. TEM images of the superalloy Ni-12.5(Al,Ti,Nb,Ta)-37(Cr,Co,W,Mo,Re)-0.17(C,La,Y,Ce,B) subjected to heterogenization annealing following by: (a) – HPT; (b) – HPT and annealing at 700 °C during 2 hours.](image)

![Figure 4. Microhardness of the superalloy Ni-12.5(Al,Ti,Nb,Ta)-37(Cr,Co,W,Mo,Re)-0.17(C,La,Y,Ce,B) subjected to different treatments.](image)

4. Summary
Hot deformation of the cast superalloy Ni-12.5(Al,Ti,Nb,Ta)-37(Cr,Co,W,Mo,Re)-0.17(C,La,Y,Ce,B) at 1175 °C with an initial strain rate of 5×10⁻⁴ s⁻¹ only on ε=50% led to the formation of the recrystallized microstructure with an average grain size of about 7 µm. HPT of the samples of the cast superalloy with different initial conditions led to refined microstructure to nanocrystalline. The average grain size was about 30 nm. Annealing at 700 °C for 2 hours of the superalloy with different initial conditions subjected to HPT slightly increased the grain size up to 50 nm. The highest microhardness (9.8 GPa) was achieved in the superalloy subjected to heterogenization annealing following by HPT and subsequent annealing at 700 °C for 2 hours.

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