The influence of temperature-strain rate conditions on hot workability and microstructure of powder metallurgy nickel-based superalloy EP741NP

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Abstract. The influence of temperature-strain rate conditions on the compression properties, hot workability and changes in the microstructure after the uniaxial single-step isothermal compression and the three-step isothermal compression with intermediate anneals has been studied for the powder metallurgy (PM) nickel-based superalloy EP741NP. Compression tests were carried out in the temperature range of \( T = 900-1190 \) °C to a total strain of \( \varepsilon = 1.05 \). The study of the microstructure was carried out for the cross sections of the deformed samples. The optimal strain temperature in terms of hot workability and recrystallization kinetics was defined as \( T = 1140-1160 \) °C, i.e. \( 45-25 \) °C below the \( \gamma' \) solvus temperature \( T_s \). Three-step compression experiments with intermediate anneals were performed at the optimal temperature of deformation with a strain rate in the range of \( \dot{\varepsilon} = 10^{-3}-10^{-1} \) s\(^{-1} \) to obtain improved hot workability and fast recrystallization kinetics. BSE observations and EBSD analysis showed that the three-step uniaxial compression at \( T = 1140-1160 \) °C with \( \dot{\varepsilon} = 5 \times 10^{-3} \) s\(^{-1} \) and intermediate anneals provided reasonable hot workability and the formation of a recrystallized fine-grained microstructure with predominantly high-angle grain boundaries.

1. Introduction
Powder metallurgy (PM) nickel-based superalloys are widely used for manufacturing of various parts in gas turbine engines (GTE). They are typically produced from powder, which is consolidated by hot isostatic pressing (HIP) into the desired parts. It is known that the mechanical properties of PM consolidated superalloys can be significantly improved by hot working [1-4]. However, low hot workability can impede the fabrication of high-quality wrought products from PM superalloys. In addition, hot working should provide fast recrystallization kinetics and the formation of a fine-grained microstructure that is the key to attaining enhanced mechanical properties. Therefore, the temperature-strain rate conditions of hot working should be precisely selected to ensure the most reasonable hot workability and extensive occurrence of recrystallization processes throughout the material volume.

The present study was aimed to develop the temperature-strain rate conditions of the uniaxial hot forging procedure in order to obtain improved hot workability and the formation of a fine-grained recrystallized structure in the PM nickel-based superalloy EP741NP. This Russian alloy is known as the most widely used PM superalloy for the manufacture of GTE parts.

2. Experimental
The nominal chemical composition of the alloy EP741NP is as follows: Cr - 9.3; Ti - 1.8; Al - 5.2;
Mo - 4; Nb - 2.7; Co – 15.7; W - 5.4; Hf – 0.2, B – 0.014 (wt. %). The initial material was supplied as workpieces with a size of Ø90×60 mm. The material was produced from a powder with a size of particles up to 140 µm, which was consolidated by HIP. The γ′ solvus temperature ($T_{\gamma'}$) was determined by differential scanning calorimetry and quenching experiments. It was defined as $T_{\gamma'}=1180\pm5$ °C. The obtained PM superalloy was subjected to heterogenization annealing to coagulate and spheroidize the γ′ precipitates. Small samples with a size of Ø10×15 mm were cut from the heat treated alloy to carry out compression tests.

At first, single-step compression tests were performed in the temperature range of $T=900-1190$ °C with an initial strain rate of $\dot{\varepsilon}=10^{-3}$ s$^{-1}$ to a total strain of $\varepsilon=1.05$. The true stress-true strain curves were built taking into account the gradual increase in the cross section of the sample during compression. Hot workability (deformability) was evaluated by measuring the maximal crack depth ($L$) on the lateral surfaces of the deformed samples. The strain temperature range, providing the most reasonable combination of hot workability, volume fraction and recrystallized grain size, was chosen for subsequent three-step compression tests with varying strain rate ($\dot{\varepsilon}=10^{-2}-10^{-1}$ s$^{-1}$). To increase the volume fraction of recrystallized grains, intermediate recrystallization anneals after each annealing step were additionally performed at the deformation temperature. All the deformed samples were finally cooled in air.

The study of the microstructure was carried out for the central part of the cross sections of the deformed samples. Optical microscopy and scanning electron microscopy (SEM) in backscattering electron (BSE) mode were used for this. Electron backscattered diffraction (EBSD) analysis was performed with a scan-step size of 0.5 µm. In this case, the γ′ precipitates were assumed as a γ phase, and the microstructure was assumed as a quasi-single-phase. The EBSD analysis was conducted using CHANNEL 5 processing software. Grain boundaries having a misorientation angle of less than 2° were excluded from consideration taking into account the measurement accuracy. Grain boundaries having a misorientation angle of more than 15° were assumed as high-angle boundaries.

3. Results and discussion

3.1. Single-step compression tests

Figure 1 represents the true stress-true strain curves, obtained as a result of compression testing of the samples. With increasing the test temperature the flow stresses decreased, and the hot workability improved reaching its maximum at around the γ′ solvus temperature. Table 1 shows the dependences of workability and microstructural parameters of the deformed samples on the test temperature. All compressed samples had lateral cracks, which became smaller with increasing strain temperature: one can see that the value of $L$ decreased with increasing strain temperature (table 1). A microstructural examination (not presented here) showed that the volume fraction ($V_{\gamma'}$) and the size of the recrystallized γ grains ($d$) increased with increasing test temperature. After compression at $T=1175$ and 1190 °C, the mean size of recrystallized grains reached $d=14$ and 65 µm, respectively. One can see that the size of recrystallized grains sharply increased near the γ′ solvus temperature that is associated with the dissolution of the γ′ phase. Taking into account the hot workability, the volume fraction and the size of recrystallized grains depending on the test temperature, the optimal strain temperatures were defined in the range of $T=1140-1160$ °C.

3.2. Three-step compression tests with intermediate anneals

Three-step compression tests with intermediate anneals were carried out at $T=1140$ °C, varying the strain rate. Figure 2 represents the appearance of samples and the corresponding EBSD orientation maps obtained from the central parts of the samples, deformed at a strain rate ranging from $\dot{\varepsilon}=10^{-4}$ to $5\times10^{-2}$ s$^{-1}$. Table 2 shows the dependences of workability (maximal crack depth on the lateral surfaces) and microstructural parameters of the deformed samples on the strain rate after a three-step compression with intermediate anneals. All compressed samples had lateral cracks. However, it should be noted that the $L$ value, characterizing the workability, has changed non-monotonically depending
on the strain rate, which reaches the smallest value after deformation at $\dot{\varepsilon}=5\times10^{-3}$ s$^{-1}$. At the same time, the volume fraction of recrystallized grains monotonically increased with increasing strain rate (table 2). Most likely, compression with a higher strain rate imparted a higher strain energy, which promoted the recrystallization processes during intermediate anneals. The fact that the size of recrystallized $\gamma$ grains decreased with increasing strain rate indicates that compression with a higher strain rate created a larger number of recrystallization centers and the formation of new recrystallized grains predominated over the growth of recrystallized grains during anneals after each compression step. The efficient occurrence of recrystallization processes under the three-step compression with intermediate anneals at $T=1140$ °C is also confirmed by the fact that predominantly high-angle grain boundaries were obtained (table 2).

![Figure 1](image1)

**Figure 1.** True stress-strain curves obtained as a result of compression tests of the samples of the alloy EP741NP at different temperatures ($\dot{\varepsilon}=10^{-3}$ s$^{-1}$).

| Deformation temperature, °C | 1100 | 1140 | 1175 | 1190 |
|-----------------------------|------|------|------|------|
| The maximal crack depth, $L$, mm | 4 | 3.2 | 2.6 | 2.2 |
| The fraction of recrystallized grains, $V_{rg}$, % | 48 | 55 | 60 | $\approx$100 |
| The mean size of recrystallized grains, $d$, µm | 1.2 | 3.3 | 14 | 65 |

**Table 1.** The maximal crack depth on the lateral surfaces and the microstructural parameters obtained for samples of the alloy EP741NP subjected to single-step compression depending on the strain temperature ($\dot{\varepsilon}=10^{-3}$ s$^{-1}$, $\varepsilon=1.05$).

![Figure 2](image2)

**Figure 2.** (a-c) The appearance of lateral surfaces of the samples and (d-f) EBSD orientation maps obtained from the central part of the samples of the EP741NP alloy subjected to three-step compression tests at $T=1140$ °C with intermediate anneals: (a, d) $=10^{-4}$ s$^{-1}$, (b, e) $5\times10^{-3}$ s$^{-1}$, (c, f) $\dot{\varepsilon}=5\times10^{-2}$ s$^{-1}$.

Comparing tables 1 and 2, one can see that the three-step compression at $T=1140$ °C with intermediate anneals was more effective from the viewpoint of hot workability and recrystallization.
behavior. Indeed, the predominantly fine-grained recrystallized microstructure \((V_{rg}=70\text{-}100\%)\) was obtained after the three-step compression with intermediate anneals at \(T=1140\ \degree C\), in contrast to the single-step compression at the same temperature \((\dot{\varepsilon}=10^{-3}\ \text{s}^{-1})\), which provided only \(V_{rg}=55\%). It is worth noting that the three-step compression with intermediate anneals and \(\dot{\varepsilon}=5\times10^{-3}\text{-}5\times10^{-2}\ \text{s}^{-1}\) provided appreciably better hot workability than the single-step compression (tables 1 and 2). At the same time, the size of recrystallized \(\gamma\) grains after the single-step compression and the three-step compression with intermediate anneals at \(T=1140\ \degree C\) was found to be similar \((d=3.3\ \text{and} \ 3.7\text{-}6\ \mu m,\) respectively). This may be due to the fact that a significant volume fraction of the \(\gamma'\) phase did not dissolve at \(T=1140\ \degree C\) and stabilized the microstructure against grain growth during intermediate recrystallization anneals.

Note that the \(L\) value slightly increased with an increase in the strain rate from \(\dot{\varepsilon}=5\times10^{-3}\) to \(5\times10^{-2}\ \text{s}^{-1}\) indicating a decrease in hot workability (figure 2 b, c, table 2). Most likely, an increase in the strain rate led to impeding dynamic recrystallization and dynamic recovery. Despite the wider development of recrystallization processes during intermediate anneals after straining with \(\dot{\varepsilon}=5\times10^{-2}\ \text{s}^{-1}\), this promoted the formation of cracks on the lateral surfaces of the samples.

**Table 2.** The maximal crack depth on the lateral surfaces and the microstructural parameters obtained for the samples of the alloy EP741NP subjected to a three-step compression with intermediate anneals depending on the strain rate \((T=1140\ \degree C, \ v=1.05)\).

| The maximal crack depth and the microstructural parameters | The strain rate \(\dot{\varepsilon}, \text{s}^{-1}\) |
|-----------------------------------------------------------|------------------|
|                                                          | \(10^{-4}\) | \(5\times10^{-3}\) | \(5\times10^{-2}\) |
| The maximal crack depth, \(L, \text{mm}\)                | 3.3          | 1               | 1.6              |
| The fraction of recrystallized grains, \(V_{rg},\ %\)     | 70           | 84              | \approx100       |
| The mean size of recrystallized grains, \(d, \mu m\)      | 6            | 5.1             | 3.7              |
| The fraction of high-angle grain boundaries, \%           | 70           | 79              | 90               |

Thus, multiple hot working with intermediate recrystallization anneals at temperatures 45\text{-}25 \(\degree C\) below \(T_s\) was found to be much more effective for the PM superalloy EP741NP than single-step hot working at the same temperatures. In the former case, recrystallization processes occurred more extensively that provided the formation of a fine-grained microstructure with predominantly high-angle grain boundaries. As has been recently shown [5], the positive influence of multiple hot working and intermediate recrystallization anneals on the recrystallization behavior and hot workability can be used for manufacturing of parts made of the alloy EP741NP with a gradient microstructure and mechanical properties.

**4. Conclusions**

The influence of temperature-strain rate conditions on compression properties, hot workability and microstructure changes after the uniaxial single-step isothermal compression and the three-step isothermal compression with intermediate anneals was studied for the powder metallurgy (PM) nickel-based superalloy EP741NP. Compression tests and microstructural examination showed that the three-step uniaxial compression at \(T=1140\text{-}1160\ \degree C\) with \(\dot{\varepsilon}=5\times10^{-3}\ \text{s}^{-1}\) to a strain of \(\varepsilon=1.05\) and intermediate anneals at the same temperature provided improved hot workability and the formation of a more uniform recrystallized fine-grained microstructure with predominantly high-angle grain boundaries in contrast to the single-step uniaxial compression under the same conditions.

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