Microstructure and mechanical properties of a novel polycrystalline rhenium-containing nickel-based disc superalloy subjected to hot forging

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Abstract. A newly developed heavy alloyed polycrystalline nickel-based superalloy SDZhS-15 containing rhenium and intended as a structural material for turbine discs in gas turbine engines has been studied. Multiple homogenization and heterogenization heat treatment was developed for the superalloy to improve its hot workability. The heat treated workpieces were subjected to single-step hot forging under quasi-isothermal conditions at temperatures slightly below the γ′ solvus temperature followed by air cooling and ageing. As a result, sound forgings with an inhomogeneous, partially recrystallized microstructure were produced. The obtained forgings were used for preparation of specimens for mechanical testing. The new superalloy showed superior high strength and high temperature capability, while retaining reasonable ductility in comparison with currently known disc superalloys.

1. Introduction

Gas turbine engines (GTE) provide a challenging environment for the development of novel materials and associated processing methods. Particularly, the increase of GTE efficiency requires the use of novel materials and processes that would meet the growing operational requirements due to the increase in temperature and loading of components [1]. To meet these requirements, novel nickel-based superalloys are designed towards heavier alloying to achieve properly balanced mechanical properties, i.e. higher creep resistance along with higher strength, fatigue strength and other service properties.

In respect of polycrystalline nickel-based superalloys, the following alloy designing approaches can be distinguished:

i) an increase in the volume fraction of the γ′ phase precipitates by increased concentrations of precipitate forming elements such as Al, Ti, Nb and Ta. In this case, the γ′ solvus temperature increases approaching the melting temperature of the superalloy;

ii) alloying with elements such as rare-earth yttrium, cerium and lanthanum forming dispersed precipitates different from the γ′ phase and providing additional dispersion hardening;

iii) alloying with elements providing solid-solution strengthening, such as molybdenum, tungsten, rhenium, ruthenium, etc. In this regard, there is a risk of forming topologically close packed (TCP) phases during long-term high temperature exposure that can reduce the high temperature capability of
a superalloy. Thus, the alloying additions should be precisely chosen to avoid the formation of undesirable phases.

In the present study, a hot forging procedure was developed for the newly designed polycrystalline superalloy SDZhS-15 containing rhenium, followed by microstructure characterization and evaluation of mechanical properties. The obtained mechanical properties are to be compared with those of currently known high $\gamma'$ containing nickel-based superalloys.

2. Experimental
The experimental nickel-based superalloy SDZhS-15 with a nominal chemical composition of Ni-28(Cr, Co)-12.5(Al, Ti, Nb, Ta)-9(Mo, W, Re)-0.17(C, La, Y, Ce, B) (in wt. %) was taken as a starting material. The ingots of the superalloy with a size of $\varnothing$ 100×180 mm were manufactured by vacuum induction melting. Small pieces were cut out of the initial ingot and remelted in a laboratory arc-melting furnace under argon atmosphere to ensure good chemical homogeneity. The ingot dimensions used in the present study were approximately $\varnothing$ 45×15 mm.

To enhance the hot workability, long-term homogenization and heterogenization anneals were carried out in the temperature range of $(T_s-120 ^\circ C)\div(T_s+20 ^\circ C)$, where $T_s$ is the solvus temperature; each annealing stage was performed in a time interval of $\tau=2$-$20$ h and followed by slow furnace cooling. The heat treated workpieces with dimensions of 35×22×15 mm$^3$ were subjected to forging under quasi-isothermal conditions. To do this, the workpieces were put into a thick-walled can made of a more ductile nickel base superalloy in contrast to SDZhS-15 and having nearly the same solvus temperature as SDZhS-15. The canned workpieces were heated up to a temperature 20÷40 $^\circ$C lower than $T_s$ and forged at a strain rate of $\dot{\varepsilon}\approx10^{-2}$ s$^{-1}$ to an engineering strain of $\varepsilon\approx80\%$ using the die tool preheated to $T=930 ^\circ C$. The forged workpieces were left to cool in air then subjected to aging at $T=T_s-360 ^\circ C$ (6 h) and $T=T_s-470 ^\circ C$ (32 h) followed by air cooling.

The microstructural examination was carried out for the cross sections of the forged workpieces. Scanning electron microscopy (SEM) in backscattering electron (BSE) mode was used for this. The electron backscattered diffraction (EBSD) analysis was performed with a scan-step size of 0.5 $\mu$m. In doing so, the $\gamma'$ precipitates were assumed as a $\gamma$ phase and the microstructure was assumed as a quasi-single-phase. The EBSD analysis was conducted using the CHANNEL 5 processing software. Grain boundaries having a misorientation angle less than 2$^\circ$ were excluded from consideration taking into account the measurement accuracy. Grain boundaries having a misorientation angle more than 15$^\circ$ were assumed as high-angle boundaries. X-ray diffraction (XRD) measurement was made using Cu-K$_a$ radiation.

The obtained forgings were used for preparation of samples for mechanical testing. Tensile and long-term strength tests were carried out using flat samples with a gauge section of 15×3×1.5 mm$^3$. All specimens for mechanical testing were prepared by electrospark cutting followed by fine grinding of working surfaces.

3. Results and discussion

3.1. Initial as-cast material
The X-ray diffraction analysis of the as-cast alloy revealed only peaks corresponding to the $\gamma$ and $\gamma'$ phases (figure 1 a). Thus, any undesirable phases, including TCP ones, were not detected in the superalloy (within the sensitivity of the XRD analysis).

Figure 1 b represents the BSE images of SDZhS-15 in the as-cast condition. A typical dendritic microstructure was obtained. Nonequilibrium eutectic colonies resulted from dendritic segregation were clearly distinguished (figure 1 b). The size of the elongated dendrites was in the range of (10-20)×(50-100) $\mu$m, the arm spacings were about 20 $\mu$m. The $\gamma'$ phase precipitates had a size mostly in the range of 0.1-0.2 $\mu$m and predominantly cuboidal morphology; there were also coarse coagulated $\gamma'$
particles with a size of 1-2 µm. White particles were carbides, which had a size of 0.5-5 µm (figure 1 b). The volume fraction of the γ' phase was visually 65-70%.

Figure 1. (a) XRD spectrum and (b) BSE image obtained for the superalloy SDZhS-15 in the as-cast condition: (a) TCP phases were not detected in the superalloy, (b) arrows show nonequilibrium eutectics; white particles are carbides, dark particles are γ' precipitates formed during cooling of the ingot.

3.2. Effect of hot working on microstructure and mechanical properties

In order to improve the hot workability, the as-cast superalloy prior to hot forging was subjected to heat treatment, which included multiple homogenization and heterogenization anneals. This reduced the level of dendritic segregation and led to coagulation and spheroidization of the γ' precipitates.

To ensure nearly isothermal conditions and all-round compression, hot forging was fulfilled in a thick-walled can made of a nickel-based superalloy, which was more ductile and had a solvus temperature similar to that of SDZhS-15. The heat treated canned workpieces were preheated at a temperature 20÷40 °C lower than Ts and forged under quasi-isothermal conditions to a strain of ε≈80%, then removed from the cans and aged. Sound forgings almost free of any lateral cracks were produced using this technique.

Figure 2 shows the EBSD orientation map and BSE images obtained from the central part of the forged workpiece. One can see that coarse non-recrystallized γ grains occupied about 70 vol.%. A large number of low-angle boundaries were observed in these grains suggesting that the dynamic recovery occurred very extensively (figure 2 a). This is confirmed by the fact that the fraction of high-angle boundaries was only 45.2%. Nevertheless, fine recrystallized grains with a size of dγ=3-20 µm, occupying about 30 vol.% were formed that was the result of dynamic recrystallization (figure 2a). Thus, a partially recrystallized microstructure was obtained after canned forging, in spite of the higher strain rate and quasi-isothermal forging conditions. Apparently, this was promoted by all-round compression provided during canned forging. The microstructure of the forged material contained coarse and dispersed precipitates of the γ' phase with a size of 1-3 µm and about 0.15-0.2 µm, respectively. Large precipitates were not dissolved at the forging temperature, whereas the dispersed γ' precipitates appeared during cooling of the forged workpiece. Note that the fine γ' precipitates had a rounded morphology. This can be ascribed to the effect of a higher strain rate and a slower cooling rate in the case of canned forging. White carbide particles appeared after ageing. They were mostly located near the coarse γ' precipitates along the γ grain boundaries and had a size of 0.2-2 µm.

The forged and aged workpieces were used to prepare specimens for mechanical testing. Table 1 represents the mechanical properties of the superalloy SDZhS-15 after multiple heat treatment, canned forging and ageing in comparison with those of other industrial superalloys applied as disc materials for GTE. It is seen that the strength properties and creep resistance obtained for SDZhS-15 are higher than those of the known disc superalloys. Note that the specimens tested at T=650 °C and a loading of P=1200 MPa for 100 hours were not fractured. This indicates a significant potential of the new
superalloy in terms of creep resistance. It is worth noting that the superalloy showed fairly high strength, while retaining lower but quite reasonable ductility (table 1).

Thus, preliminary heat treatment improving hot workability was developed for the new nickel base superalloy SDZhS-15. Note that the composition of the superalloy is close to the composition of some single-crystal superalloys and non-deformable cast superalloys [1-4]. This suggests that heavy alloying of nickel-based superalloys with refractory elements, including rhenium is not an obstacle to the following wrought processing. Hot workability was improved due to the reduction of dendritic segregation, coagulation and spheroidization of the $\gamma'$ precipitates. This allowed forging the superalloy under quasi-isothermal conditions with relatively high strain rates, which provided a partially recrystallized microstructure as a result of dynamic recrystallization. The forged material was cooled in air from the forging temperature and then aged. As a result, both fine and coarse $\gamma'$ precipitates were obtained. The material with the microstructural state obtained demonstrated superior strength and long-term strength at $T=650$ °C, while retaining a fairly reasonable ductility compared to the currently known disc superalloys. As expected, further optimization of hot working and strengthening heat treatment might additionally enhance the mechanical properties of the new developed superalloy.

![Figure 2.](image)

**Figure 2.** (a) EBSD orientation map and (b) the corresponding distribution of the misorientation-angle for grain boundaries obtained from the central part of the workpiece, subjected to preliminary multiple heat treatment and canned forging; (a) high- and low-angle grain boundaries are indicated by black and white lines, respectively.

**Table 1.** Mechanical properties of the superalloy SDZhS-15 in comparison with those of other nickel base disc superalloys (RT – room temperature).

| Superalloy      | UTS, MPa | YS, MPa | $\delta$, % | Long-term strength at $T=650$ °C, 100 hours, MPa |
|-----------------|----------|---------|-------------|-----------------------------------------------|
| SDZhS-15 (present work) RT | 1708     | 1250    | 11          | >1200                                         |
| $T=650$ °C      | 1486     | 1080    | 8           |                                               |
| N18 RT [5]      | 1620     | 1080    | >20         | >1000                                         |
| Alloy 10 RT [6] | 1650     | 1210    | 12-17       |                                               |
| AF115 RT [7]    | 1646     | 1190    | 20          | 1030                                          |
| TMW-4 RT [8]    | 1700     | 1210    | 9           | -                                             |
| ME3 (Rene104) RT [9] | 1580     | 1150    | 9-18        |                                               |
|                  | 1650     | 1150    | 21          | -                                             |
| FGH100 RT [10]  | 1600     | 1172    | 19          | $T=705$ °C, 897 MPa, the creep life $\tau=42.8$ h |
| $T=705$ °C      | 1345     | 1090    | 20          |                                               |
4. Conclusions
The preliminary multiple heat treatment has been developed for the new ingot-metallurgy high $\gamma'$ containing nickel-based superalloy SDZhS-15, which improved its chemical homogeneity, provided coagulation and spheroidization of $\gamma'$ precipitates and improved hot workability of the as-cast superalloy. Successful hot forging of the new superalloy using a can made of a nickel-based superalloy under quasi-isothermal conditions was fulfilled at temperatures slightly below the solvus temperature. As a result, an inhomogeneous and partially recrystallized microstructure was produced in the forged workpiece. In spite of the fact that the obtained microstructure was far from being optimal, the new superalloy subjected to canned hot forging followed by ageing demonstrated superior mechanical properties. Further efforts should be directed at optimizing the processes of hot working and strengthening heat treatment in order to produce a more homogeneous and properly designed microstructure.

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