INFLUENCE OF WARM ROLLING AND RECRYSTALLIZATION ANNEALING ON MECHANICAL AND METALLOGRAPHIC PROPERTIES OF THE SUPERALLOY N07080

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Abstract

Additional strengthening of superalloy N07080 described in this work was achieved by warm rolling. Control of the ratio of strength and ductile properties of the superalloy is possible by appropriate selection of the amount of warm deformation and the appropriate selection of the partial recrystallization temperature. In addition, recrystallization annealing makes it possible to equalize the grain size across the cross section of the warm rolled bars, which before recrystallization differ significantly in size in the central and peripheral parts of the bars.

Introduction

The superalloy N07080 (Nimonic 80A) is a nickel-base alloy intended for use at elevated and high temperatures where significant creep may occur. The primary strengthening mechanism of this superalloy is based on the precipitation of fine and coherent particles of intermetallic γ’ phase Ni₃(Al,Ti) that significantly increase the creep resistance. This strengthening mechanism for such a superalloy is more favourable than other strengthening mechanisms [1, 2]. The effect of hardening that can be achieved by the γ’ phase depends on the amount, dispersion, and size of the γ’ phase and it is controlled by heat treatment. The standard heat treatment includes a solution annealing at 1080°C/8 h and precipitation aging at 700°C/16 h. The maximal hardness of the superalloy Nimonic 80A achieved after this treatment is around 360 HV, but certain applications in the automotive industry require higher hardness. Since a long-lasting solution annealing at high temperature causes coarsening of the grains, it is not possible to increase the hardness (additional strengthening) significantly with a reduction of the grain size.

The increase of the dislocation density after solution annealing and before precipitation aging by cold or warm deformation (work hardening) increases the hardness. Since a recrystallization as softening process after such deformation decreases the hardness, the hardness of the superalloy can be controlled by partial recrystallization [3, 4]. Complete recrystallization softens the superalloy and fully restores mechanical properties to the values it possessed before work hardening.

Rolling of the superalloy, generally and also at elevated temperatures (warm and hot rolling) causes an increase the dislocation density, that is, an increase of stored energy within the superalloy. This stored energy is driving force for recrystallization. Recrystallization can be dynamic (DRX), static (SRX) and metadynamic (MDRX) [5]. DRX occurs during hot deformation, SRX occurs during heat treatment in unloaded pre-strained workpiece, MDRX occurs by continued growth of the nuclei formed by dynamic recrystallization [6].

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Nickel superalloys have low values of the stacking fault energy (SFE), hence the dynamic recrystallization takes place discontinuously - discontinuous dynamic recrystallization (DDRX) [5, 7]. In metals with high values of SFE the continuous dynamic recrystallization (CDRX) occurs. DDRX takes place heterogeneously with clear nucleation and growth stage while CDRX takes place uniformly with no clear nucleation and growth stage. Unlike to metals with low SFE, in metals with high SFE the dissociation of the perfect dislocation into two partials dislocation is more difficult, so perfect dislocation may glide, climb and cross slip easily. This enables rearrangement (forming of the subgrains boundary) and annihilation of dislocations decreasing their density through dynamic recovery (DRV) process. In this case recrystallisation is based on progressively increasing of the misorientation of subgrain boundaries which may lead to HAGBs at larger strains (CDRX). In the case of metals with low SFE perfect dislocations are dissociated into partials, so their cross-slip or climb are more difficult. Because of that forming of the subgrain structures during deformation by DRV is difficult, so dislocation density increases to a high level with possibility of their local accumulation. On locations where dislocation density becomes larger then critical dislocation density the nuclei of the new grains will form and then grow (DDRX). Nucleation of DDRX is usually initiated on pre-existing grain boundaries. A necklace structure of equiaxed grains forms when there is a large difference between the initial grain size and the recrystallized grain size [8]. Initial structure? with coarse grains exist in the superalloy? N07080 (Nimonic 80A) after solution annealing (1080°C/8 h).

**Experimental Research and Test Results**

Superalloy N07080 according to standard ASTM B 637 for experimental research was produced by double melting. Primary melting was performed in a vacuum induction furnace (VIM). Remelting? was performed by electroslag? remelting (ESR) process. Achieved chemical composition of the superalloy after remelting (ESR ingot) is given in Table 1.

**Table 1:** Chemical composition of ESR ingot [9].

| Sample          | C    | Si    | Mn    | P    | S    | Fe   | Cr   | Co   | Al   | Ti   | Ni   | Balance |
|-----------------|------|-------|-------|------|------|------|------|------|------|------|------|-------|
| Top of the ingot| 0,07 | 0,84  | 0,73  | 0,007| 0,006| 3,0  | 20,4 | 2,0  | 1,26 | 2,42 |      |       |
| Bottom of the ingot| 0,08 | 0,82  | 0,76  | 0,007| 0,006| 3,0  | 21,0 | 2,0  | 1,29 | 2,42 |      | Balance |

The dimensions of the ESR ingot were φ126 mm at the bottom, φ 115 mm at the top, and the length was 305 mm. The weight of the ingot was 27.9 kg. Hot forging of ingot up to a diameter of 50 mm was performed on a hydraulic press 2 MN, and then on a pneumatic hammer 2,5 kN up to a diameter of 20 mm. The temperature interval of hot forging was between 950 °C and 1160 °C. Hot rolling (starting temperature 1160 °C) of the bars φ20 mm was carried out on light-section rolling mill SKET φ 370 mm on four different dimensions:

1. Round bars with diameter 15 mm (not intendend for additional warm rolling),
2. Horizontal oval bars 13,0 x 21,4 mm (intendend for additional warm rolling – one pass on bars φ15 mm with 10% of warm deformation),
3. Vertical oval bars 15,0 x 18,0 mm (intendend for additional warm rolling – two passes on bars φ15 mm with 20% of warm deformation),
4. Horizontal oval bars 14,0 x 25,2 mm (intendend for additional warm rolling – three passes on bars φ15 mm with 30% of warm deformation).

Additional warm rolling of the bars was carried out after performing of solution annealing at 1080°C/8 h. The starting temperature for the warm rolling was 1050 °C.

After warm rolling all bars were cooled to room temperature on still air. All solution annealed and warm rolled bars were heat treated by final precipitation aging at 700°C/16 h.

Hot rolled bars φ15 mm that not intenden for additional warm rolling were used for mechanical and metallographic testing of the superalloy after standard heat treatment (1080°C/8h + 700°C/16h). All thermal and thermomechanical treatments performed? on the bars are shown in Figure 1.
The tensile test of each test bar was performed on test pieces with a diameter of 8 mm machined from rolled bars φ15 mm. Hardness and metallographic tests were performed on a full cross section of rolled bars φ15 mm. All tests were performed on standard heat treated bars, solution annealed + warm rolled bars with different amounts of deformation (10%, 20% or 30% reduction of cross section), solution annealed + warm rolled + partially or fully recrystallized bars. Also, all tested bars were heat treated by precipitation aging before testing.

The tensile testing results are shown in Table 2, Figure 2 and Figure 3. The hardness testing results are shown in Figure 4. The microstructures of the different bars are shown in Figures 5 to 8. The grain size in standard heat treated (1080°C/8 h + 700°C/16 h) bars is between G1 and G3. These bars also have larger grains than G1. In bars that are warm rolled, there is no significant change in grain size as well as in those that are recrystallization annealed at 1000°C/1h. By performing recrystallization annealing at higher temperatures (1040°C and 1080°C), the grain size is reduced (grain size between G2 and G4). This grain size analysis does not include grains in necklaces, where the grains are up to size. In the bar 1080°C/8h + 10% warm def. + 1040°C/1h + 700°C/16h as a result of partial recrystallization, grain size is between G2 and G6 [9].

Table 2: Results of tensile testing of the superalloy N07080 at 20°C and 700°C.

| Heat treatment and/or thermomechanical treatment | Testing at 20°C | Testing at 700°C |
|-------------------------------------------------|----------------|-----------------|
|                                                 | $R_{p0.2}$ (MPa) | $R_m$ (MPa) | A (%) | $R_{p0.2}$ (MPa) | $R_m$ (MPa) | A (%) |
| 1080°C/8h+700°C/16h                             | 785            | 1215           | 24.0   | 730            | 795           | 4.0    |
| 1080°C/8h+10% warm def.+700°C/16h               | 1076           | 1314           | 17.5   | 852            | 917           | 5.0    |
| 1080°C/8h+20% warm def.+700°C/16h               | 1131           | 1352           | 15.5   | 795            | 844           | 3.5    |
| 1080°C/8h+30% warm def.+700°C/16h               | 1161           | 1379           | 15.0   | 763            | 870           | 5.0    |
| 1080°C/8h+10% warm def.+1000°C/1h+700°C/16h     | 934            | 1264           | 22.0   | 780            | 863           | 6.0    |
| 1080°C/8h+20% warm def.+1000°C/1h+700°C/16h     | 930            | 1255           | 22.0   | 811            | 834           | 6.0    |
| 1080°C/8h+30% warm def.+1000°C/1h+700°C/16h     | 914            | 1272           | 22.0   | 792            | 835           | 6.5    |
| 1080°C/8h+10% warm def.+1040°C/1h+700°C/16h     | 855            | 1260           | 25.0   | 731            | 742           | 4.0    |
| 1080°C/8h+20% warm def.+1040°C/1h+700°C/16h     | 872            | 1261           | 25.5   | 658            | 679           | 5.5    |
| 1080°C/8h+30% warm def.+1040°C/1h+700°C/16h     | 947            | 1333           | 24.0   | 735            | 741           | 6.0    |
| 1080°C/8h+20% warm def.+1080°C/1h+700°C/16h     | 765            | 1209           | 27.5   | 741            | 813           | 4.0    |
Figure 2: Yield and tensile strength of the superalloy N07080 at 20 °C and 700 °C.

Figure 3: Elongation after break of the superalloy N07080 at 20 °C and 700 °C.
Discussion:

The results of the tensile testing at 20 °C shows that the yield strength and tensile strength of the solution annealed bars are increased by warm rolling at 1050 °C. The hardness of warm rolled bars also increases. On the other hand, the elongation decreases. As the amount of warm deformation increases, the yield strength, tensile strength and hardness increase too, while the elongation decreases. The results of the tensile testing at 700 °C shows that and the yield strength, tensile strength and elongation do not change significantly. After annealing of the warm rolled bars at 1000 °C/1h, 1040 °C/1h and 1080 °C/1h, the yield strength, tensile strengths and hardesses decrease while the elongation increases. After annealing at 1000 °C/1h, 1040 °C/1h, the values of these properties do not return to the values of the properties that the superalloy has in the standard heat treated state (1080 °C/8h + 700 °C/16h), while after annealing at 1080 °C/1h? the values of the yield strength, tensile strength, hardness and elongation are returned to values that are very close to those in the standard heat-treated bars. Therefore, annealing at 1000 °C/1h and 1040 °C/1h represents partial recrystallization while annealing at 1080 °C/1h represents complete recrystallization. The contribution to the increase of the superalloy hardness from the final precipitation aging is getting smaller with increasing of the contribution of the warm deformation on the superalloy hardness and vice versa.

The grains in the bars after standard heat treatment (1080 °C/8h + 700 °C/16h) are very large. Some of them are larger than G1 (mean grain diameter > 0.3 mm). Warm rolling of solution annealed bars causes the appearance of different microstructures in the central and peripheral parts of the bars. Microstructural differences between the central and peripheral parts of the bars occur due to the existence of different stress state in the central and peripheral parts of the bars and the slower cooling of the central parts than their periphery. In the outer parts of the bars the grains are deformed. Twins are present, too. Recrystallization of the outer parts of the warm rolled bars is mainly carried out by a static recrystallization process. Recrystallization of the central parts is initiated as dynamic and/or metadynamic recrystallization. In the central parts of the warm rolled bars a necklace structure of small equiaxed grain forms at pre-existing boundaries of coarsed grains. The formation of the necklace structure is a characteristic of the continuous dynamic recrystallization process (DDRX). As the recrystallization temperature (1000 °C, 1040 °C, and 1080 °C) increases, the proportion of recrystallized structure increases, too.
| Treatment                  | Periphery of the bar | Core of the bar |
|----------------------------|----------------------|-----------------|
| Without recrystallization  | ![Image](image1.png) | ![Image](image2.png) |
| 1000°C/1h                  | ![Image](image3.png) | ![Image](image4.png) |
| 1040°C/1h                  | ![Image](image5.png) | ![Image](image6.png) |
| 1080°C/1h                  | ![Image](image7.png) | ![Image](image8.png) |

**Figure 5:** Microstructure of bars rolled with 10% of warm deformation without and with recrystallization annealing included between solution annealing and precipitation aging.
**Figure 6:** Microstructure of bars rolled with 20% of warm deformation without and with recrystallization annealing included between solution annealing and precipitation aging.
| Treatment                        | Periphery of the bar | Core of the bar |
|--------------------------------|----------------------|-----------------|
| Without recrystallization      | ![Periphery Image](image1) | ![Core Image](image2) |
|                                | ![Periphery Image](image3) | ![Core Image](image4) |
| 1000°C/1h                      | ![Periphery Image](image5) | ![Core Image](image6) |
| 1040°C/1h                      | ![Periphery Image](image7) | ![Core Image](image8) |
| Recrystallization              | ![Periphery Image](image9) | ![Core Image](image10) |
| 1040°C/1h                      | ![Periphery Image](image11) | ![Core Image](image12) |
| 1080°C/1h                      | ![Periphery Image](image13) | ![Core Image](image14) |

**Figure 7:** Microstructure of bars rolled with 30% of warm deformation without and with recrystallization annealing included between solution annealing and precipitation aging.
Taking into account the mechanical properties of the warm rolled bars annealed at 1000 °C and 1040 °C and the microstructural characteristics of the same bars, temperatures of 1000 °C and 1040 °C are the partial recrystallization temperatures. According to the achieved mechanical properties determined on the bars which are annealed at 1080 °C after warm deformation and which are close to the corresponding mechanical properties of the standard heat treated bars, the annealing temperature of 1080 °C is the temperature of complete recrystallization. The grains in the bars recrystallized at 1080 °C are generally smaller than the grains in standard heat treated bars, but there are significant differences in their size.

Conclusions:
By conducting warm deformation of the N07080 superalloy at 1050 °C after solution annealing it is possible to increase its strength and hardness. These properties can be reduced by recrystallization annealing. Complete recrystallization is possible at temperature of 1080 °C. Annealing at lower temperatures does not allow reducing strength and hardness of the warm rolled bars on values that the superalloy has after standard heat treatment. Therefore, at temperatures of 1000 °C and 1040 °C it is possible to achieve only partial recrystallization of the superalloy N07080. By appropriate selection of the amount of warm deformation and the appropriate selection of the partial recrystallization temperature, it is possible to control the ratio of strength and ductile properties of the superalloy N07080. Due to the difference in stress state and cooling rate of the central and peripheral parts of the bars after warm rolling the microstructure of these parts of the bars differ. Differences in microstructure between these parts of the warm rolled bars are reduced by conducting recrystallization annealing. Recrystallization of the outer parts of the warm rolled bars is generally carried out by a static recrystallization process, while recrystallization of the central parts begins as a dynamic and/or metadynamic recrystallization.

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