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## DIFFERENCE OF THE CREEP RATE OF PRE-STRAINED AND NO PRE-STRAINED SUPERALLOY N07080

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#### ABSTRACT

This paper describes the difference in the creep rate of pre-strained and no pre-strained samples of superalloy N07080. The primary strengthening mechanism of this superalloy is based on the precipitation of fine and coherent particles of the intermetallic γ' phase Ni<sub>3</sub>(Al,Ti) that ensure good creep resistance. In the case of additional strengthening of superalloy N07080 by warm plastic deformation, sometimes required by the automotive industry, its life in creep conditions will be significantly reduced. Performing the partial recrystallization annealing, after solution annealing and warm deformation, and before the final precipitation annealing, leads to a decrease in strength and an increase in the superalloy ductile properties and return of part of the lost creep life due to warm deformation.

Because of the shorter lifetime of warm-deformed superalloy N07080 samples, their creep rate is higher than that of those not warm-deformed. The creep rate at 50 % of creep rupture life of superalloy N07080 that warm rolled by 30% deformation (1080°C/8h+30% warm def.+700°C/16h) is 12,9 times higher than the creep rate of the standard heat treated superalloy. This creep rate reduces with increasing partial recrystallization temperature and for recrystallization temperature 1080°C it reaches values close to those that the superalloy possesses after standard heat treatment (1080°C/8h+700°C/16h).

Keywords: superalloys, creep, creep rate, pre-strained alloys, recrystallization

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#### 1. INTRODUCTION

The creep of metals is time-dependent plasticity under constant stress at elevated temperatures. Three different regions or stages characterize the creep. In the first stage (primary creep) the creep rate ( $\acute{\epsilon}$  =  $d\varepsilon/dt$ ) decreases with increasing strain ( $\varepsilon$ ) due to metal hardening. In the second stage (steady-state) the creep rate is constant. In the third stage (tertiary creep) the creep rate increases significantly with increasing strain until fracture occurs due to cavitations. Sometimes, the first stage leads directly to the third stage [1]. This paper describes the difference in the creep rate of pre-strained and no pre-strained samples of superalloy N07080.

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  $\gamma'$  phase  $Ni<sub>3</sub>(Al,Ti)$  that significantly increase the creep resistance. This strengthening mechanism for such

superalloy is more favorable than other strengthening mechanisms [2,3] because other possible strengthening mechanisms lead to a decrease in creep resistance. The effect of strengthening 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/8h and precipitation aging at 700°C/16h. Since a long-lasting solution annealing at high temperature causes coarsening of the grains, an increase of the strength is not possible significantly with a reduction of the grain size. The higher dislocation density after solution annealing and before precipitation aging by cold or warm deformation (work hardening) increases the strength. Recrystallization softens the alloy and fully restores mechanical properties to the values it possessed before work hardening, so the strength of the superalloy can be controlled by partial recrystallization [4,5].

Rolling of the superalloy, generally and also at elevated temperatures (warm rolling) causes an increase in the dislocation density, that is, the increase of stored energy within the superalloy. This stored energy is the driving force for recrystallization. Recrystallization can be dynamic (DRX), static (SRX), and meta-dynamic (MDRX) [6]. DRX occurs during hot deformation, SRX occurs during heat treatment in the unloaded pre-strained workpiece, and MDRX occurs by the continued growth of the nuclei formed by dynamic recrystallization [7]. Nickel superalloys have low values of the stacking fault energy (SFE), hence the dynamic recrystallization takes place discontinuously - discontinuous dynamic recrystallization (DDRX) [6,8]. The 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 size of the grain and recrystallized grain sizes [9]. Such initial structures with coarse grains exist in the superalloy N07080 after solution annealing (1080°C/8 h).

The service life of an alloy under creep conditions encompasses its entire

operational period. This period is divided into the time required for the nucleation of the cavities and the remaining time until fracture (growth and coalescence of intergranular cavities). In fully recrystallized structures, nucleation of the cavities occupies most of the life of the alloy [10]. The service life of some alloys can be drastically reduced if they are pre-strained (work hardened) before use under creep conditions [11,12,13,14]. Cavity nucleation generally occurs at sites where stress concentration occurs. Such sites are mainly grain boundaries. In the case of cavitation along grain boundaries, it mostly occurs along grain boundaries that lie transversely to the direction of tensile stress [13,15]. As the amount of deformation increases during the creep process, uneven elongation of individual grains in the polycrystalline material occurs. Unevenly deformed grains must adapt to each other. This can be achieved by sliding along the boundaries between adjacent grains [16]. Different inhomogeneities at grain boundaries such as solid particles or ledges are potential sites for stress concentration. In the pre-strained alloys, additional sites for stress concentration are sites of interaction of a slip band and a grain boundary. The internal stress caused by blocked slip bands in the pre-strained alloy can lead to accelerated cavity nucleation at elevated temperatures and a significant shortening of the creep rupture lifetime [12]. Recrystallization eliminates potential sites for rapid cavity nucleation, beacuse of that the creep rupture life is prolonged [17]. As the recrystallization processes progress, the influence of rapid nucleation of cavities on the creep rupture life decreases, and the influence of the grain size increases, i.e. the bars with coarser grain have longer creep rupture life [11,18].

## 2. EXPERIMENTAL RESEARCH AND TEST RESULTS

# 2.1 Practical work

Superalloy N07080 according to standard ASTM B 637 for this research was produced by double melting. Primary melting was performed in a vacuum induction furnace (VIM). The remelting was carried out using

the electro-slagremelting (ESR) process. The chemical composition of the superalloy after remelting (ESR ingot) is given in Table 1. The dimensions of the ESR ingot were  $\phi$ 126 mm at the bottom,  $\phi$ 115 mm at the top, and the length was 305 mm. The weight of the ingot was 27.9 kg. Hot forging of ingot to the diameter of 50 mm was performed using a 2 MN hydraulic press, and a 2.5 kN pneumatic hammer for diameters up to 20 mm. The temperature interval of hot forging was between 950°C and 1160°C. Hot rolling (starting temperature 1160°C) of the bars  $\phi$ 20 mm was carried out on a light-section rolling mill SKET  $\phi$ 370 mm on two different dimensions:

 $-$  round bars with a diameter of 15 mm (not intended for additional warm rolling),

Table 1. Chemical composition of ESR ingot [19]

 horizontal oval bars 14.0 x 25.2 mm (intended for additional warm rolling – three passes to the bars  $\phi$ 15 mm with 30% of warm deformation – reduction of crosssection in total).

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. Solution annealed, warm rolled and partially recrystallized or not recrystallized bars were heat treated by final precipitation aging at 700°C/16h. All bars  $\phi$ 15 mm were used for tensile, creep and metallographic testing. All thermal and thermomechanical treatments performed on the bars are shown in Figure 1.







Figure 1. Warm rolling in combination with corresponding heat treatments

The tensile test of each test bar was performed on test pieces with a diameter of 8 mm machined from rolled bars  $\phi$ 15 mm. The tests were performed on standard heat treated bar, solution annealed + warm rolled

bars (with 30% reduction of cross section) +precipitation aged bar and solution annealed + warm rolled + partially recrystallized (1000°C/1h) + precipitation aged

pieces) at room temperature and at 700°C (three test pieces) are shown in Table 2. The creep test of each test bar was performed on test pieces with a diameter of 8 mm machined from rolled bars with a diameter of 15 mm. All tests were performed on standard heat treated bars, solution annealed + warm rolled bars with 30% reduction of cross section, solution annealed + warm rolled +

partial or fully recrystallized bars. Also, all tested bars were heat treated by precipitation aging before testing. The creep test results at 700°C and stress of 340 MPa are shown on Figure 2. The creep rate of the same test pieces at 5%, 10%, 50%, 95% and 100% of creep rupture life for test pieces with different heat and thermomechanical treatments are shown in Table 3.

Table 2. Results of tensile testing of the superalloy N07080 on three test pieces at room temperature and three test pieces at 700°C

	Testing at room temperature			Testing at 700°C		
Heat treatment and/or thermomechanical treatment		$R_m$ (MPa)	А $(\%)$	$R_{p0.2}$ (MPa)	$\rm R_m$ (MPa)	A $(\%)$
1080°C/8h+700°C/16h	785	1215	24.0	730	795	4.0
1080°C/8h+30% warm def.+700°C/16h	1161	1379	15.0	763	870	5.0
1080°C/8h+30% warm def.+1000°C/1h+700°C/16h	914	1272	22.0	792	835	6.5



**Figure 2.** Creep diagrams (creep rate  $-$  time) for test pieces after different heat and thermomechanical treatments at temperature 700°C and stress of 340 MPa

Table 3. Creep rate at different percentages of creep rupture life at 700°C and stress of 340 MPa for test pieces after different heat and thermomechanical treatments

- Sample	Creep rupture time t(h)	Rupture elongation ε (%)	Creep rate, $\acute{\text{e}}$ x 1000 (h <sup>-1</sup> ), at				
			5%	10%	50%	95%	100%
			of creep rupture life				
$1080^{\circ}$ C/8h + 700 $^{\circ}$ C/16h	392	1.06	0.0786	0.0349	0.0098	0.0322	0.2283
$1080^{\circ}$ C/8h + 30% warm def. + 700°C/16h	22	0.56	0.0378	0.0666	0.1264	0.4561	0.4958
$1080^{\circ}$ C/8h + 30% warm def. + $1000^{\circ}$ C/1h + 700 $^{\circ}$ C/16h	83	1.69	0.0499	0.0999	0.1090	0.4141	0.7625
$1080^{\circ}$ C/8h + 30% warm def. +	219	1.39	0.1160	0.0495	0.0254	0.1283	0.8125



The microstructures of the different bars are shown in Figures 3 and 4. 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 the bar that was warm rolled with 30% deformation, there are no significant differences in grain size compared to those that partially recrystallized at 1000°C for 1 hour. Recrystallization annealing at elevated temperatures (1040°C and 1080°C) leads to a decrease in grain size, resulting in grain sizes ranging from G2 to G4. This grain size analysis does not include grains in necklaces, where the grains correspond to G8 size [16, 18]. In the warm rolled and warm rolled and partially recrystallized (1000°C/1h, 1040°C/1h, 1080°C/1h) bars the grains in the peripheral parts are larger than in their central parts (Figure 3). The grains in the central and peripheral parts of the standard heat-treated bar are similar. Since the creep test specimens were obtained by machining from the rolled bars with a diameter of 15 mm to the initial diameter of the creep test pieces of 8 mm, the microstructures of test pieces generally correspond to the microstructures of the core of the bar, Figure 4.

### 2.2 Discussion

The results of tensile tests indicate that superalloy N07080 can be additionally strengthened through warm plastic deformation. Also, carrying out partial recrystallization annealing, after solution annealing and warm deformation, and before the final precipitation annealing leads to a decrease in strength and an increase in the superalloy ductile properties. However, this method of additional strengthening leads to a significant decrease in the service life of the superalloy in creep conditions, Figure 2. The notable reduction in the creep rupture life due to pre-strain is attributed to increased stress concentration sites along primary grain boundaries, which serve as potential locations for rapid cavity nucleation. Recrystallization decreases the number of possible sites for rapid cavity nucleation, which prolongs the creep rupture life. As the recrystallization processes progress, the influence of rapid nucleation of cavities on the creep rupture life decreases, and the effect of the grain size increases, i.e. the samples with coarser grain have longer creep rupture life [15], Figure 2 and Figure 4. Increasing the temperature of the partial recrystallization annealing extends the creep lifetime of the superalloy, making it closer to that of a standard heat-treated superalloy, as shown in Figure 2. The sample that warm deformed after solution annealing (30% reduction of cross-section), without subsequent partial recrystallization, does not show the second stage of creep, but after the first stage of creep follows the third stage - the stage of cavitation, which after a relatively short time (22 h) leads to the fracture. By conducting partial recrystallization annealing, after warm deformation, the second stage of creep becomes noticeable and becomes longer as the recrystallization temperature is higher, Figure 2. Creep rates for the same creep stages for samples after different heat or thermo mechanical treatments are different, Table 3 and Figure 5. In the second stage of creep, that is, at 50% of the creep rupture life the creep rate of the sample 1080°C/8h+30% warm def.+1000°C/1h+700°C/16h is around 11 times greater than creep rate of standard heat treated sample 1080°C/1h+700°C/16h, while creep rate of the sample 1080°C/8h+30% warm def.+1040°C/1h+700°C/16h is around 2.6 times greater than creep rate of standard heat treated sample 1080°C/1h+700°C/16h. The creep rate of the sample which was partially recrystallized at 1080°C/1h (1080°C/8h+30% warm def.+ 1080°C/1h+700°C/16h) does not differ significantly from the creep rate of the standard heat treated sample 1080°C/8h+700°C/16h.



Figure 3. Microstructure of bars  $\phi$ 15 mm after treatment 1080°C/8h+30% warm def.+700°C/16h in their core and periphery Core of the bar **Periphery of the bar** Periphery of the bar





1080°C/8h + 700°C/16h 1080°C/8h+30% warm def. +700°C/16h



1080°C/8h+30% warm def. +1000°C/1h +700°C/16h



1080°C/8h+30% warm def. +1040°C/1h +700°C/16h



Figure 4. Microstructure of the creep test pieces 1080°C/8h+30% warm def. +1080°C/1h +700°C/16h

The biggest difference in the creep rate at 50% of the creep rupture life exists between the standard heat-treated sample (1080°C/8h+700°C/16h) and the sample that warm deformed without subsequent partial

recrystallization annealing (1080°C/8h+30% warm def.+700°C/16h). The creep rate of this sample is 12.9 times higher than the creep rate of the standard heat-treated sample.



and thermomechanical treatments

Similar differences in creep rate, but not in the same amounts, between the same samples, exist at 10% and 95% of the creep rupture life, Table 3 and Figure 5.

### 3. CONCLUSION

Warm plastic deformation or warm rolling can strengthen superalloy N07080. However, this additional strengthening method significantly reduces the superalloy's service life under creep conditions. Performing the partial recrystallization annealing, after solution annealing and warm deformation, and before the final precipitation annealing, leads to a decrease in strength and an increase in ductile properties of the superalloy and return of part of the lost creep life due to warm deformation. On elevated temperatures, significant creep rupture life reduction occurs due to the accelerated nucleation of cavities, especially in places where slip-bands are blocked by grain boundaries in pre-strained superalloys. Recrystallization eliminates potential sites for rapid cavity nucleation causing creep life prolongation and creep rate to decrease. The creep rate at 50% of creep ruptures life of superalloy N07080 that warm rolled by 30% deformation is 12.9 times higher than the creep rate of the standard heat-treated superalloy. This creep rate, at 50% of creep rupture life, reduces with increasing partial

recrystallization temperature, and for recrystallization temperature 1080°C it reaches values close to those that the superalloy possesses after standard heat treatment.

# Conflicts of Interest

The authors declare no conflict of interest.

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