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RESEARCH ARTICLE

CREEP RUPTURE LIFE OF PRE-STRAINED SUPERALLOY N07080

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Abstract

The creep of the pre-strained superalloy N07080 is described in this work. The pre-strain was achieved by warm rolling at 1050 ^{o}C. The warm rolling was performed due to additional strengthening, i.e increasing of the superalloy hardness. The pre-strain drastically reduces the creep rupture life of the superalloy compared to the creep rupture life of the standard heat treated superalloy. The drastic reductionof the creep rupture life is result of rapid creep cavity nucleation on stress concentration sites along primary grain boundaries of the pre-strained superalloy. Recrystallization eliminates potential sites for rapid cavity nucleation and prolongates the creep rupture life.

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 $Ni_3(Al,Ti)$ 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/8h and precipitation aging at 700°C/16h. 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 alloy 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 metadimamic (MDRX) [5].

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DRX occurs during hot deformation, SRX occurs during heat treatment in unloaded prestrained workpiece, MDRX occurs by continued growth of the nuclei formed by dynamic recrystallization [6]. Nickel superalloys have low values of the stacking fault energy (SFE), hence the dynamic recrystallization takes place discontinuously - discontinuous dynamic recrystallization (DDRX) [5, 7]. 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]. Such initial structures with coarse grains exist in the superalloy N07080 after solution annealing (1080°C/8 h).

The service life of an alloy operating in creep conditions covers the entire period of its exploitation. This period is divided into the time required for the nucleation of the cavities and the remaining time until fracture (growth and coalescense of intergranular cavities). In fully recrystallized structures, nucleation of the cavities occupies most of the life of the alloy [9]. The service life of some alloys can be drastically reduced if they are pre-strained (work hardened) before use under creep conditions [10, 11, 12, 13].

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 mainly occurs along grain boundaries that lie transversely to the direction of tensile stress [12]. 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 [14]. Different inhomogeneities at grain boundaries such as solid particles or bulges are potential sites for stress concentration to occur. In the pre-strain alloy additional sities for stress concentration are sities of interaction of a slip band and a grain boundary. Internal stress coused by blocked slip bands in pre-strained alloy can lead to accelerated cavity nucleation at elevated temperature and a significant shortening of the creep rupture lifetime [11]. Grain size has a significant effect on the creep resistance of the superalloy. In general, a recrystallized coarse-grained microstructure has a longer creep life than a fine-grained microstructure [10, 15].

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 [16].

Sample	Content of elemnts in mass fraction (w/%)									
	C	Si	Mn	P	S	Fe	Cr	Co	Al	Ti	Ni
Top of the ingot	0,07	0,84	0,73	0,007	0,006	3,0	20,4	2,0	1,26	2,42	Balance
Bottom of the ingot	0,08	0,82	0,76	0,007	0,006	3,0	21,0	2,0	1,29	2,42	

The dimensions of the ESR ingot were ϕ126 mm at the bottom, ϕ115 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 ^{o}C and 1160 ^{o}C. Hot rolling (staring temperature 1160 ^{o}C) of the bars ϕ20 mm was carried out on light-section rolling mill SKET ϕ370 mm on four different dimensions:

- round bars with diameter 15 mm (not intendend for additional warm rolling),
- 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),
- vertikal oval bars 15,0 x 18,0 mm (intendend for additional warm rolling – two passeson bars ϕ15 mm with 20% of warm deformation),
- 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 carrid out after performing of solution annealing at 1080°C/8 h. The starting temperature for the warm rolling was 1050 ^{o}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 hardness, creep 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.

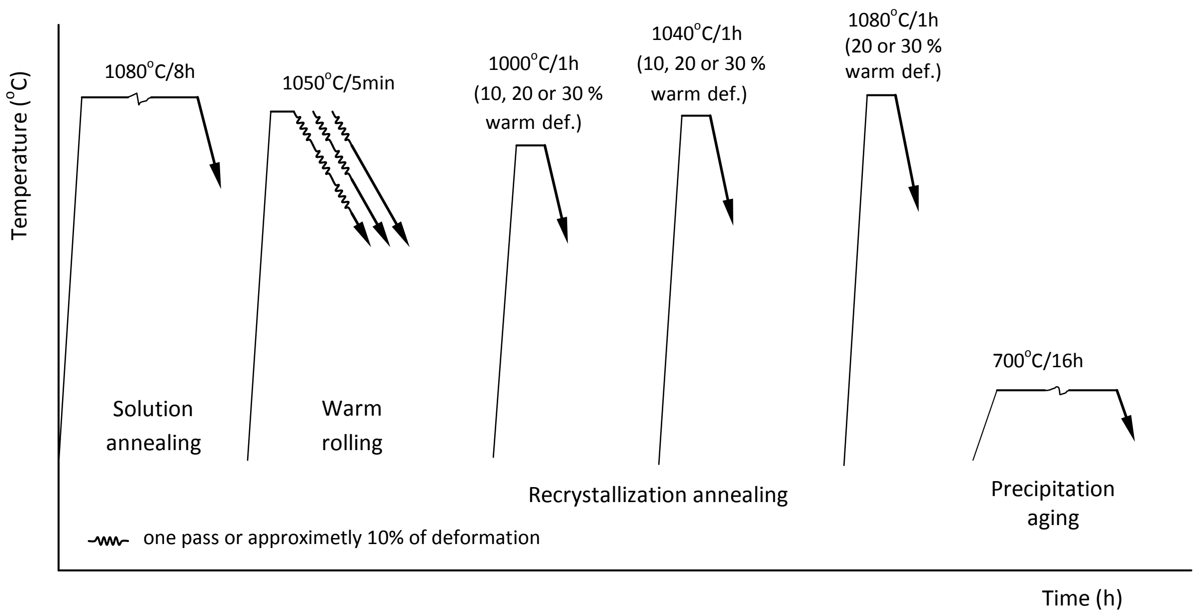


Figure 1:- Warm rolling in combination with corresponding heat treatments.

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. Hardness tests were performed on a full cross section of rolled bars with a diameter of 15 mm. All tests were performed on standard heat treated bars, solution annealed + warm rolled bars with diferent amount of deformation (10 %, 20% or 30% reduction of cross section), solution annealed + warm rolled + partially or fully recrystalized bars. Also, all tested bars were heat treated by precipitation aging before testing. Hardness testing results are shown in Figure 2, while creep test results are given in Table 2.

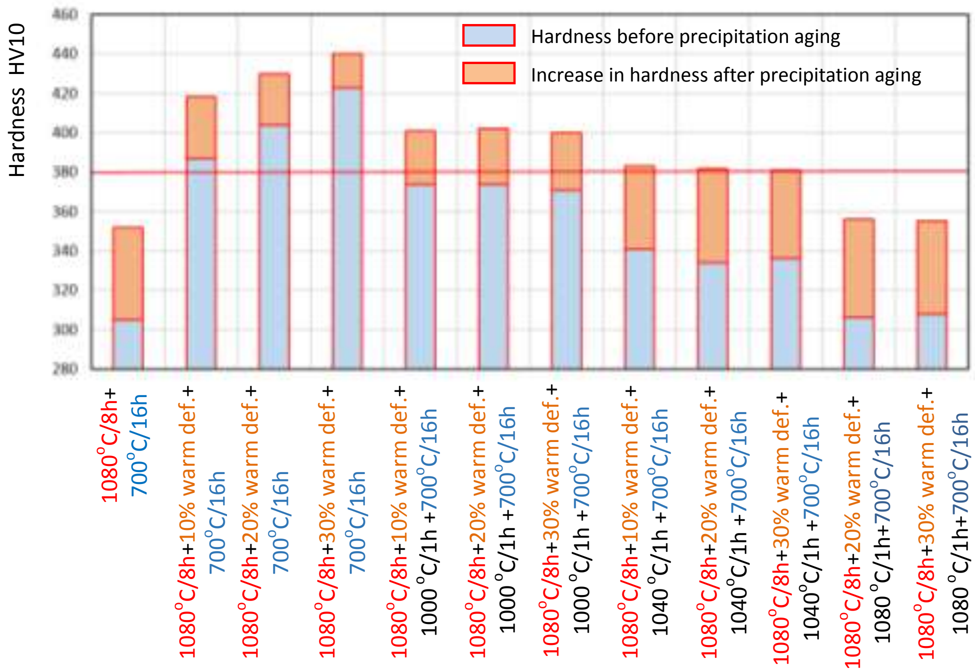
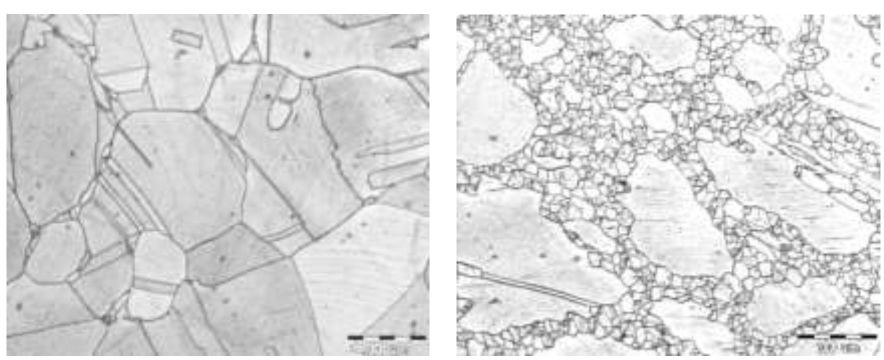


Figure 2:- Hardness of the superalloy N07080 after different heat and thermomechanical treatments.

Table 2:- Results of creep testing of the superalloy N07080 after different heat and thermomechanical treatments at 700 ^{o}C and stress of 340 N/mm^2.

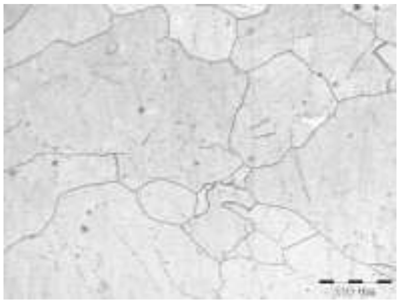
Sample	Creep rupture time, t (h)		Rupture elongation, ε (%)		Creep rate at 70% of creep rupture life, $\acute{\varepsilon}$ x 1000 (h^{-1})	
1080^{o}C/8h+700^{o}C/16h	392	368	1,06	1,05	0,0136	0,011
	344		1,03		0,0090	
1080^{o}C/8h+10% warm def. + 700^{o}C/16h	14	19	0,42	0,54	0,2790	0,285
	24		0,66		0,2908	
1080^{o}C/8h+20% warm def. + 700^{o}C/16h	18	17	0,58	0,59	0,3047	0,303
	16		0,60		0,3008	
1080^{o}C/8h+30% warm def. + 700^{o}C/16h	22	18	0,56	0,55	0,2486	0,310
	14		0,53		0,3720	
1080^{o}C/8h+10% warm def. +1000^{o}C/1h + 700^{o}C/16h	43	48	1,10	1,00	0,2185	0,164
	53		0,90		0,1098	
1080^{o}C/8h+20% warm def. +1000^{o}C/1h + 700^{o}C/16h	36	40	1,34	1,26	0,2537	0,257
	44		1,17		0,2601	
1080^{o}C/8h+30% warm def. +1000^{o}C/1h + 700^{o}C/16h	63	73	1,80	1,75	0,2382	0,245
	83		1,69		0,2524	
1080^{o}C/8h+10% warm def. +1040^{o}C/1h + 700^{o}C/16h	83	132	1,00	1,12	0,0674	0,052
	181		1,14		0,0381	
1080^{o}C/8h+20% warm def. +1040^{o}C/1h + 700^{o}C/16h	140	168	0,81	1,08	0,0346	0,043
	196		1,35		0,0512	
1080^{o}C/8h+30% warm def. +1040^{o}C/1h + 700^{o}C/16h	105	162	1,50	1,46	0,1091	0,073
	219		1,39		0,0365	
1080^{o}C/8h+20% warm def. +1080^{o}C/1h + 700^{o}C/16h	319	321	1,32	1,19	0,0124	0,010
1080^{o}C/8h+30% warm def. +1080^{o}C/1h + 700^{o}C/16h	323		1,08		0,008	

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 (Figure 3). The grains in the peripheral parts of the warm rolled bars are large, while in the central parts of the warm rolled bars the grain size depends on whether a recrystallization annealing has been performed and at what temperature. Since the creep test specimens were obtained by machining from the rolled bar ϕ15 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 bars core (Figure 4). The largest grains are in standard heat-treated bars (between G1 and G3, some of them larger then G1), they are slightly smaller in warm rolled and fully recrystallized bars (between G2 and G4), even smaller in warm rolled and partially recrystallized (1040^{o}C/1h) bars. In warm rolled and warm rolled and partially recrystallized bars (1000 ^{o}C), except coarse grains, there are very small grains in the necklaces (G8) [16].

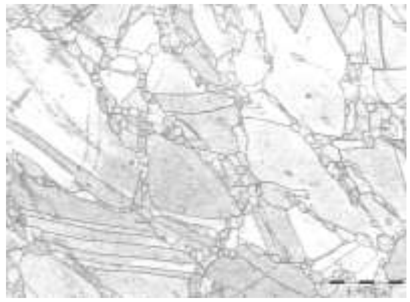


Periphery of the bar Core of the bar

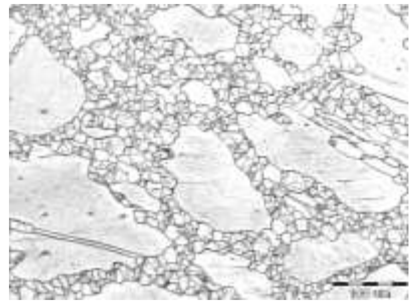
Figure 3:- Microstructure of bars- ϕ15 mm after treatment 1080^{o}C/8h+20% warm def.+700^{o}C/16h in their core and periphery.



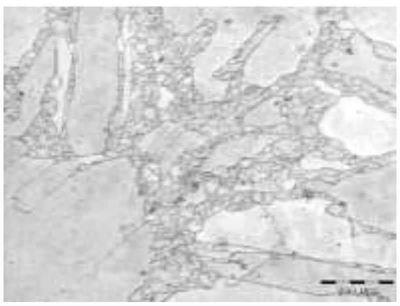
1080^{o}C/8h+
700^{o}C/16h



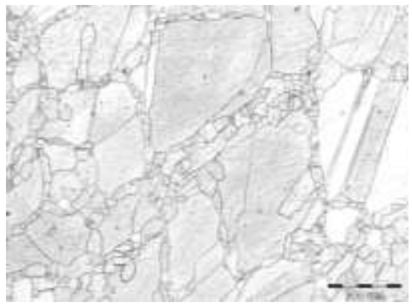
1080^{o}C/8h+10% warm def.+
700^{o}C/16h



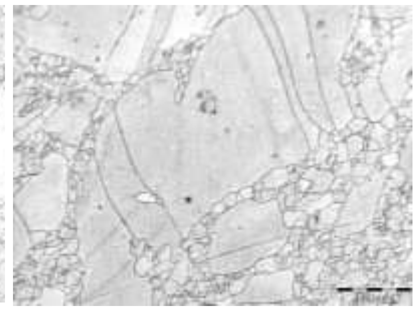
1080^{o}C/8h+20% warm def.+
700^{o}C/16h



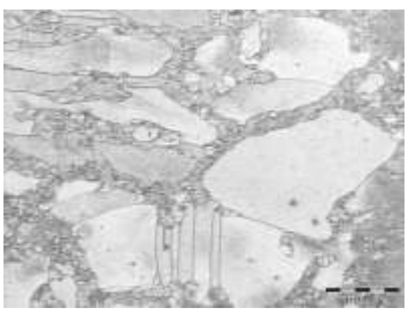
1080^{o}C/8h+30% warm def.+
700^{o}C/16h



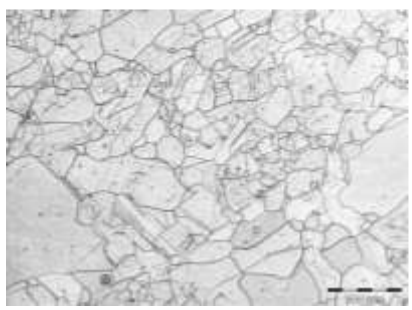
1080^{o}C/8h+10% warm def.+
1000^{o}C/1h +700^{o}C/16h



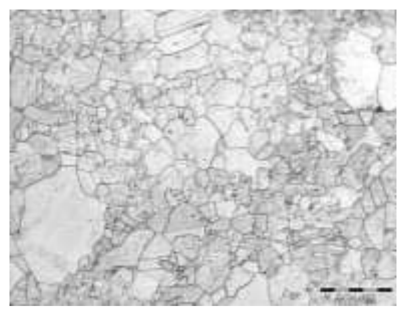
1080^{o}C/8h+20% warm def.+
1000^{o}C/1h +700^{o}C/16h



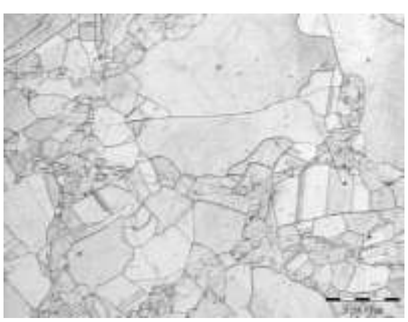
1080^{o}C/8h+30% warm def.+
1000^{o}C/1h +700^{o}C/16h



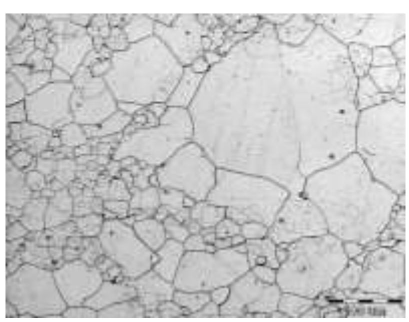
1080^{o}C/8h+10% warm def.+
1040^{o}C/1h +700^{o}C/16h



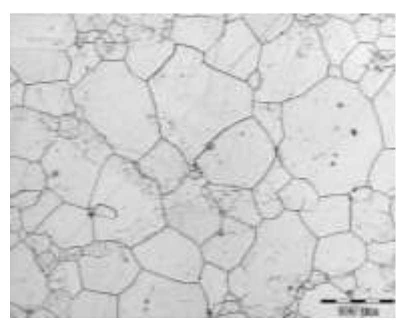
1080^{o}C/8h+20% warm def.+
1040^{o}C/1h +700^{o}C/16h



1080^{o}C/8h+30% warm def.+
1040^{o}C/1h +700^{o}C/16h



1080^{o}C/8h+20% warm def.+
1080^{o}C/1h +700^{o}C/16h



1080^{o}C/8h+30% warm def.+
1080^{o}C/1h +700^{o}C/16h

Figure 4:- Microstructure of the creep test pieces.

Discussion:-

The hardness of the standard heat treated bars (1080°C/8h+700°C/16h) is approximately 350 HV. The hardness of these bars can be increased by warm rolling at 1050 °C. The hardness of the solution annealed bars increases with the increase in the amount of deformation, so after warm rolling with the amount of deformation of 30% and final precipitation aging (700°C/16h), the hardness increases to 440 HV. The hardness of strengthened bars can be reduced by recrystallization annealing performed before final precipitation aging. If the strengthened bars are recrystallized at 1080 °C/1h, their hardness will be reduced to approximately 350 HV. Because of that the recrystallization at 1080 °C is full recrystallization. On the other hand, recrystallization at temperatures of 1000 °C and 1040 °C allows partial recrystallization of the superalloy N07080. The hardness of partially recrystallized bars is higher than hardness of solution annealed bars and fully recrystallized bars. Creep test pieces from the bars that were warm rolled without subsequent recrystallization, as well as test pieces from bars that were warm rolled and partially recrystallized at 1000 °C are characterized by the presence of the necklace structure. The necklace structure is not present in the test pieces from warm rolled bars recrystallized at 1040 °C and 1080 °C. The grains are generally finer in the bars recrystallized at 1040 °C than in the bars recrystallized at 1080 °C.

The creep test results (Table 2 and Figure 5) indicate that the creep life of the bars is drastically reduced by conducting warm rolling at 1050 °C compared to standard heat treated bars. The creep rupture life of warm rolled bars is only 5% of the creep life of standard heat treated (1080°C/8h+700°C/16h) bars. Part of the lost creep life due to warm deformation can be recovered by conducting recrystallization annealing. Warm rolled bars that are not subsequently recrystallized have the least elongation after creep rupture. Recrystallized bars have greater elongation after creep rupture. The creep rates of different bars were compared at achieved creep life of 70% of the total creep rupture life. Warm rolled bars that are not subsequently recrystallized have an average of 27x higher creep rate than standard heat treated bars. Warm rolled and recrystallized bars at 1000 °C have an average of 20x higher creep rate than standard heat treated bars. Warm rolled and recrystallized bars at 1040 °C have an average of 5x higher creep rate than standard heat treated bars. Warm rolled and recrystallized bars at 1080 °C have the creep rate that is close to the creep rate of standard heat treated bars.

Creep strain - creep time and creep rate - creep time diagrams of the some bars after different thermomechanical treatments and/or heat treatments are shown on Figure 6. Creep diagrams for the warm rolled bar without subsequent recrystallization (1080°C/8h+30% warm def.+700°C/16h) show indications of the presence of only the tertiary creep stage without the noticeable presence of the primary and secondary creep stage. Creep diagrams for warm rolled bar with subsequent partial recrystallization at 1000 °C (1080°C/8h+30% warm def.+1000°C/1h +700°C/16h) clearly shows the existence of the tertiary creep stage with indications of the presence of the primary creep stage without the presence of the secondary creep stage. Creep diagrams for warm rolled bar with subsequent partial recrystallization at 1040 °C (1080°C/8h+20% warm def.+1040°C/1h +700°C/16h) clearly shows the existence of the tertiary and the secondary creep stage with indications of the presence of the primary creep stage. Creep diagrams for warm rolled bar with subsequent partial recrystallization at 1080 °C (1080°C/8h+20% warm def.+1080°C/1h +700°C/16h) clearly shows the existence all creep stages (primary, secondary and tertiary creep stage) like standard heat treated bar (1080°C/8h+700°C/16h).

The drastic reduction of the creep life of the warm rolled bars, which are not subsequently recrystallized, indicates that the formation of the necklace structure of small grain during DDRX/MDRX does not eliminate potential sites for rapid cavity nucleation at grain boundaries. By conducting partial or complete recrystallization, i.e with the progress of recrystallization processes, the number of potential sites for rapid cavity nucleation is reduced and becasue of that the creep life is extended. As the recrystallization processes progress, the influence of rapid nucleation of cavities on the creep life decreases, while the influence of other microstructural characteristics gains in importance, such as grain size. Fully recrystallized warm rolled bars have a shorter creep life than standard heat treated bars because they have finer grains. Creep strain - creep time and creep rate - creep time diagrams of the test pieces tested by creep14h (1080°C/8h+30% warm def. + 700°C/16h) and 100h (1080°C/8h+30% warm def. + 1040°C/1h+700°C/16h) are shown on Figure 7. After the indicated number of creep hours, the tests were interrupted and the test pieces were used to make metallographic samples (Figure 8). Although the creep times are significantly different for these two test pieces, the appearance and position of the cavities are similar to each other. The cavities appear along grain boundaries that lie transversely to the direction of tensile stress in creep test pieces. This direction is parallel to the direction of maximum deformation during rolling. This means that of all the possible potential sites for cavity nucleation at all grain boundaries, only those sites located at the transverse grain boundaries can be the nuclei of the creep cavities.

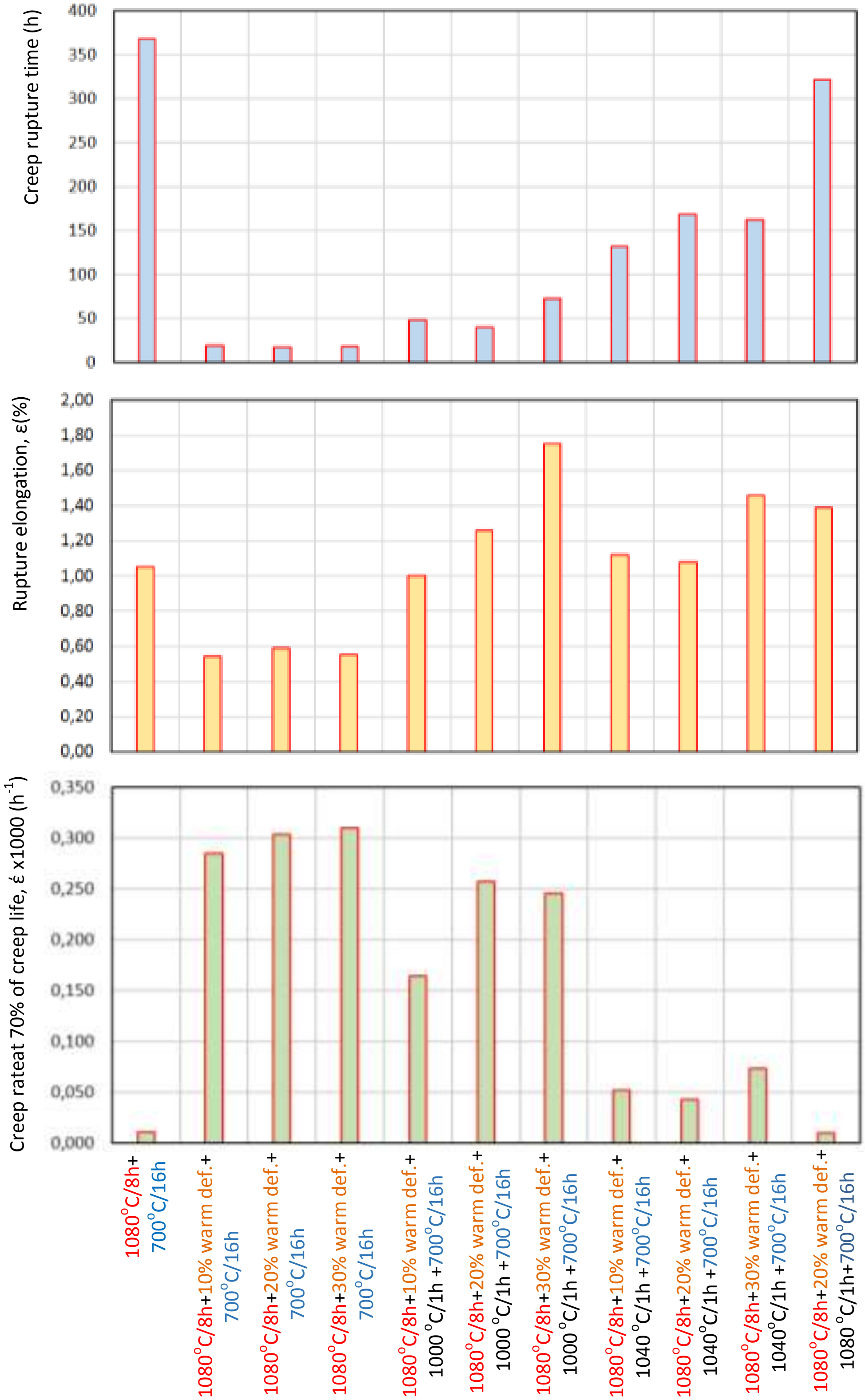


Figure 5:- Creep rupture time, rupture elongation and creep rate at 70% of creep rupture life of the superalloys N07080 after different heat and thermomechanical treatments

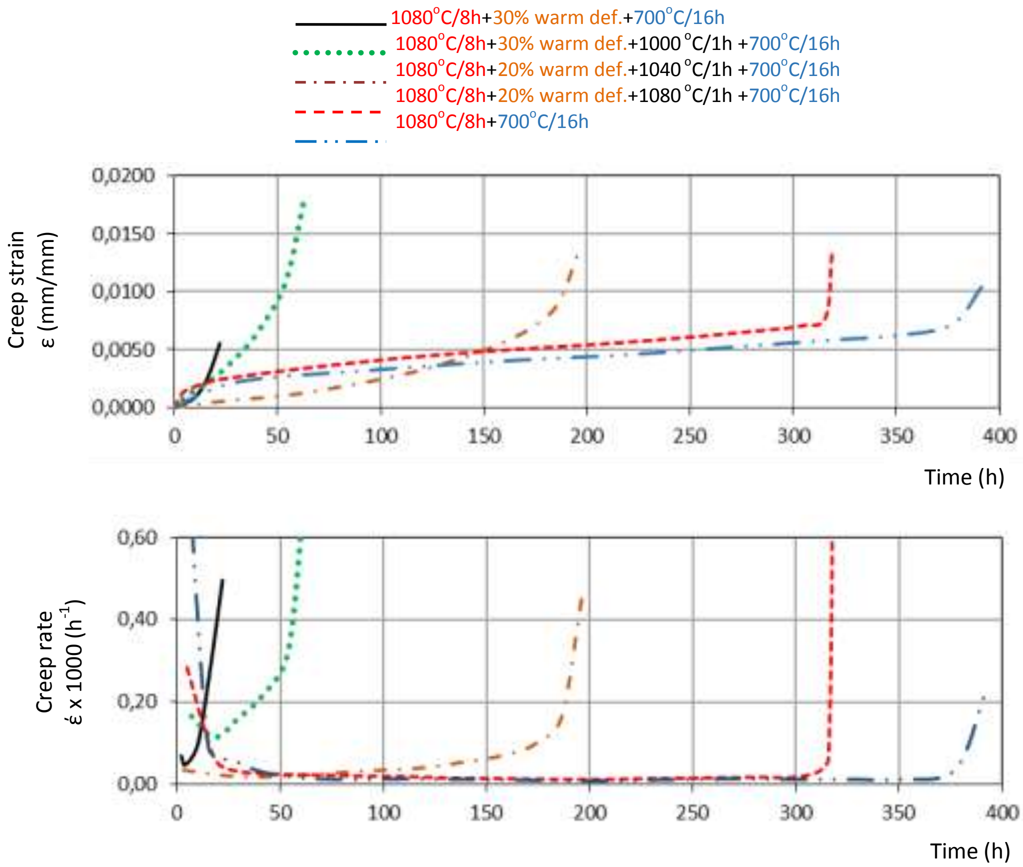


Figure 6:- Creep strain - creep time and creep rate - creep time diagrams of the some bars after different heat and thermomechanical treatments.

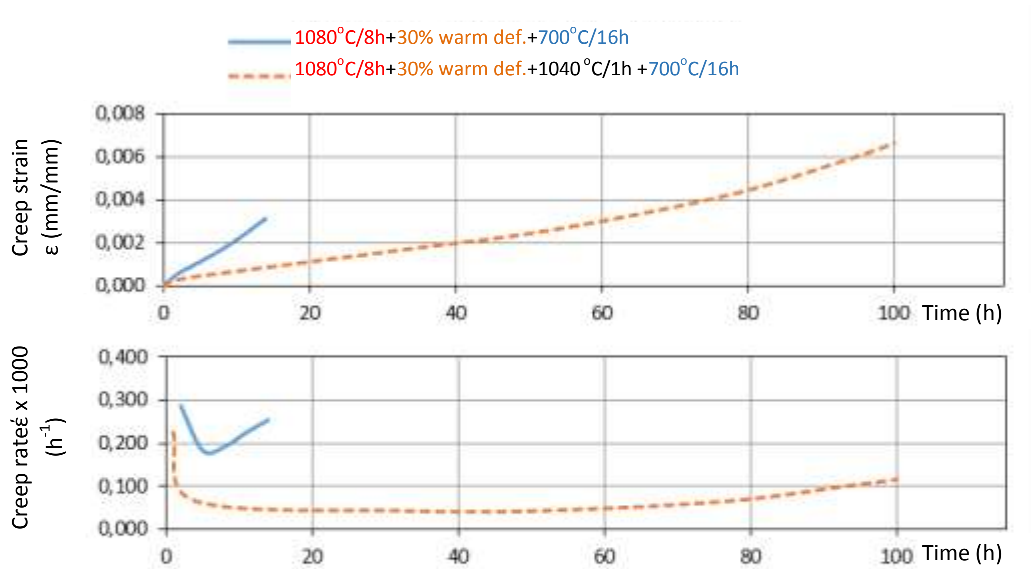
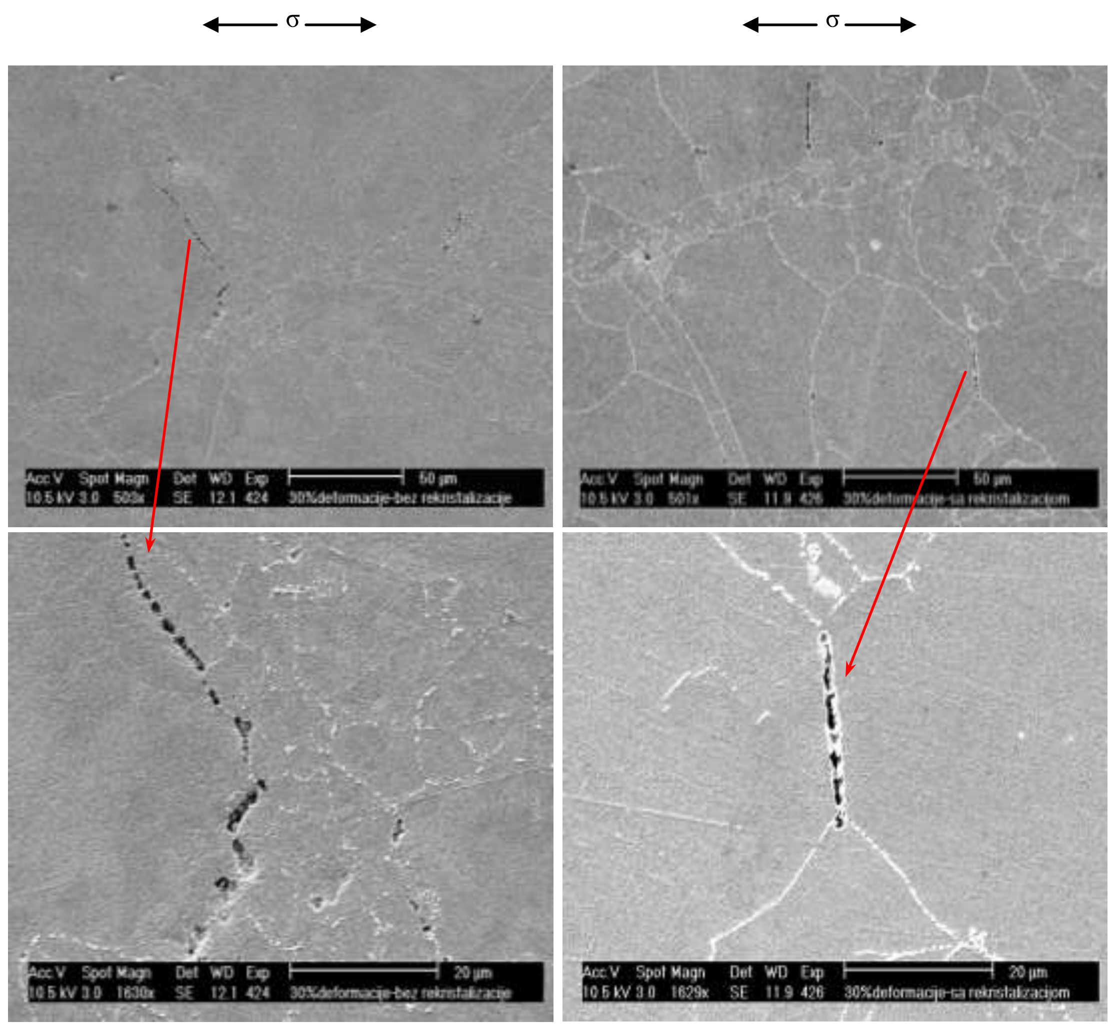


Figure 7: Creep strain - creep time and creep rate - creep time diagrams of the test pieces tested by creep 14h and 100h (after the indicated number of creep hours, the tests were interrupted and the test pieces were used to make metallographic samples)



a) 1080°C+30% warm def. +700°C/16h without recrystallization after 14h of the creep

b) 1080°C+30% warm def. + partial recrystallization at 1040°C/1h+700°C/16h after 100h of the creep

Figure 8:- SEM, cavity arrangement after a) 14h of the creep (1080°C+30% warm def.+700°C/16h – without recrystallization annealing) and b) after 100h of the creep (1080°C+30% warm def. + partial recrystallization at 1040°C/ 1h + 700°C/16h).

Conclusions:-

Warm deformation of the N07080 superalloy at 1050 °C after solution annealing enables its additional strengthening. But this strengthening results in a drastic reduction in creep rupture life compared to creep rupture life of standard heat treated bars. Part of the lost creep rupture life due to pre-strain by warm rolling compared to creep rupture life of standard heat treated bars can be recovered by conducting recrystallization annealing. As the recrystallization processes progress the creep rupture life prolongate. Therefore, the hardness and creep rupture life of the superalloy N07080 is possible to control by the amount of the warm deformations and partial recrystallization. Drastic shortening of the creep rupture life due to pre-strain is result of the increasing the number of sites on primary grain boundaries as stress concentration sites, i.e potential sites for rapid cavity nucleation. Recrystallization eliminates potential sites for rapid cavity nucleation, so the creep rupture life prolongate. As the recrystallization processes progress, the influence of rapid nucleation of cavities on the creep rupture life decreases, while the influence of the grain size increases, i.e the bars with coarser grain have longer creep rupture life.

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