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MATERIALS PROPERTIES OF MODIFEIED Ni-BASED ALLOY

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The thermomechanical processing of NiMoCr solid solution nickel base superalloy is the way to considerably influence the grain size. As uniform coarse grain size increases the creep strength and crack growth resistance. In the work, the processing to achieve uniform recrystallized grain structure with variation of thermomechanical parameters is investigated. The creep behaviour of the alloy after various hot working conditions is determined. The results of creep tests showed that creep characteristics such as strain rate and lifetime were greatly dependent on the initial hot working conditions and annealing parameters.

Key words: *Ni-based alloy, hot working, annealing, recrystallization, structure, creep*

Karakteristike modificiranih nikljenih superlegura. Termomehanička obrada NiMoCr krutog rastvora nikljene superlegure odvija se na način koji značajno utječe na veličinu zrna. S jednlikim porastom grubosti zrna povećavaju se čvrstoća na puzanje i otpornost na širenje pukotine. U radu se istražuje obrada za postizanje jednolike rekristalizirane strukture zrna uz varijaciju termomehaničkih parametara. Određuje se puzanje legure nakon raznih uvjeta tople obradbe. Rezultat ispitivanja na puzanje pokazali su da su karakteristike puzanja, kao što su produljenje i životni vijek bile vrlo ovisne o početnim uvjetima tople obrade i parametrima žarenja.

Ključne riječi: *Ni-superlegura, topla obradba, rekristalizacija, struktura, puzanje*

INTRODUCTION

Solid solution strengthened nickel base high temperature alloys have a wide use in sheet, in gas turbine engine combustion chambers, transition liners, and in petroleum and chemical processing industry. In these applications, the components are often subjected to creep and low cycle fatigue.

The development of reactors that incorporate a circulating molten fluoride fuel is predicated on the availability of construction material that will contain the molten salt over long time periods and, also ensure the required mechanical properties [1, 2]. The container must be metallurgically stable and to show enhanced resisting to oxidation and grain boundary embrittlement [3].

The reference alloy system was based on nickel with as primary strengthening the addition of 15 - 20 % Mo for the increase of oxidation resistance. Because the poor mechanical properties, additions of various solid solution alloying elements were evaluated. An optimum alloy composition was selected which ensures satisfying mechanical properties and corrosion properties with Cr, Al, Ti, V, Fe, Nb, and W additions [4].

It is known that the mechanical and physical properties of wrought nickel base superalloys can be significantly altered through variation in thermomechanical (TM) processing. The role of TM processing has to be considered when mechanical properties depend on the grain size [5]. To verify the hot working characteristics of solid solution strengthened nickel base superalloys and enable the development of suitable hot working practises a laboratory test to assess hot workability is essential.

Several microstructural features were developed to increase creep strength in solid solution strengthened materials with the purpose to lower the diffusion rate and to improve the deformability at elevated temperatures while preserving the creep compatibility of the material. Grain boundary and phase boundary sliding can be controlled by segregation and particles dispersions at the grain boundaries. This sliding should take place at the same rate as crystal lattice deformation to avoid the voids formation [6]. Structure morphology and grain size and is classical consideration for improving creep strength by utilised the ideas above. To ensure a higher creep strength and crack growth resistance the designed coarse grained microstructure should be maintained with also uniform the particles dispersions.

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This type of structure can be achieved with thermomechanical processing such as hot rolling combined with annealing and control of recrystallization [7, 8].

The purpose of the presented investigation was to examine the influence of the extent of hot rolling deformation and post-deformation annealing on microstructure evolution and creep properties of the nickel base NiMoCr alloy.

EXPERIMENTAL PROCEDURE

The investigated material was wrought nickel base NiMoCr alloy. The chemical composition of the alloy in wt. % was as follow: 72,7 % Ni; 17,8 % Mo; 6,3 % Cr; 2,8 % Fe; 0,16 % Al; 0,06 % Ti; 0,06 % W; 0,06 % Co; 0,05 % Si; 0,01 % Cu; 0,01 % B; 0,001 % S and 0,02 % C. The initial alloy was obtained with casting and multi steps press forging and annealing.

However, the obtained alloy structure was very heterogeneous. The various testing hot working conditions were carried out as shown in Table 1. The grain morphology and effect of recrystallization microstructure were investigated with metallographical examinations. Specimens from non-recrystallized and recrystallized rolled down specimens were machined for creep testing. The creep tests were performed in air with constant applied load. All creep tests were carried out at stress level of 160 MPa and temperature of 710 °C. The elongation was recorded by two extensometers. The testing temperature was controlled by two thermocouples and it was maintained within the range of ± 5 °C.

Table 1. **Details of hot working and cold working processing**
Tablica 1. **Detalji tople i hladne obradbe**

Specimen No.	Heating temperature before H. W.	% Hot deformation	Anneling time (min) at 1100 °C
A1	1,200 °C/ 30 min	18 % + 18 %	Quenching
A2	1,200 °C/ 30 min	18 % + 18 %	Air Cooling
A3	1,200 °C/ 30 min	18 % + 18 %	3 min
A4	1,200 °C/ 30 min	18 % + 18 %	5 min
A5	1,200 °C/ 30 min	18 % + 18 %	10 min
A6	1,200 °C/ 30 min	18 % + 18 %	15 min
A7	1,200 °C/ 30 min	18 % + 18 %	25 min
A8	1,200 °C/ 30 min	18 % + 18 %	50 min
B1	1,100 °C/ 30 min	11,3 % + 13,6 %	Quenching
B2	1,100 °C/ 30 min	11,3 % + 13,6 %	3 min
B3	1,100 °C/ 30 min	11,3 % + 13,6 %	5 min
B4	1,100 °C/ 30 min	11,3 % + 13,6 %	10 min
B5	1,100 °C/ 30 min	11,3 % + 13,6 %	25 min
B6	1,100 °C/ 30 min	11,3 % + 13,6 %	50 min

RESULTS AND DISCUSSION

Microstructural analysis

In the initial microstructure obtained after casting, multisteps hot pressing and annealing process it was found that it was very heterogeneous and of bimodal grain size. The explanation might be, that the multisteps press forging-annealing process did not provide uniform deformation throughout the ingot and then recrystallization process did not occur uniformly. Generally, in the microstructure very fine recrystallized grains were found beside coarse, probably primary grains. These fine grains might be generated preferentially at grain boundaries by either static recrystallization during annealing processes and/or by small amount of dynamic recrystallization occurring during the multisteps hot forging.

After hot working according to both programs A and B (specimen no. A1, A2 and B1) immediate quenching or air cooling the microstructures were still heterogeneous with a large number of twins and coarse grains in the middle of samples. However, that the deformed grains were slightly elongated in the rolling direction. This suggested a non-uniform displacement of deformation and the absence of sufficient deformation in the central part of the rolled bands.

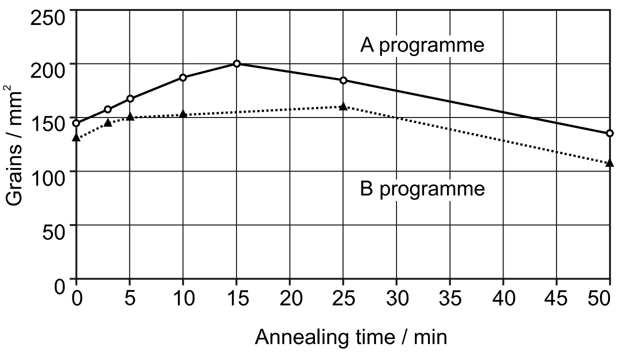

Figure 1. **The relationship number of grains/mm^2 versus annealing time**
Slika 1. **Odnos zrno/mm^2 o vremenu žarenja**

After additional hot working consisting of two steps of reductions in specimens No. A3-A8 and B2-B6, the heterogeneity in microstructure was smaller if the annealing time was increased. The recrystallization after the processing according to the A programme produced more uniform grain structure comparing to the annealed microstructures obtained with the B programme. The final grain morphology depends greatly on the degree of deformation before annealing. The greater was the degree of deformation the smaller recrystallized grain size was achieved, as shown in Table 2., 3. The relationship between the number of grains per area and annealing time is shown in Figure 1.

Creep behaviour

The creep results for TM processed specimens according to the A and B programmes are presented in Figure 2. and 3. evidence that various annealing times did not effect greatly the creep lifetime (≈ 2000 min). The specimens only quenched or air cooled immediately after hot working had much longer lifetime and correspondingly lower creep rate than those of all annealed specimens. Considering this fact the creep behaviour was explained in terms of the effect strain hardening on creep behaviour. Furthermore, it was concluded that the higher strength resulted also from the initial non-uniform coarse grain structure of the alloy. The specimens with the highest creep strength could not be used further because of their poor uniform formability made than unsuited for further forming including extrusion and tube drawing. For this reason, for application, only annealed or nearly recrystallized microstructures will be investigated further, since alloy formability, creep and fatigue properties are required.

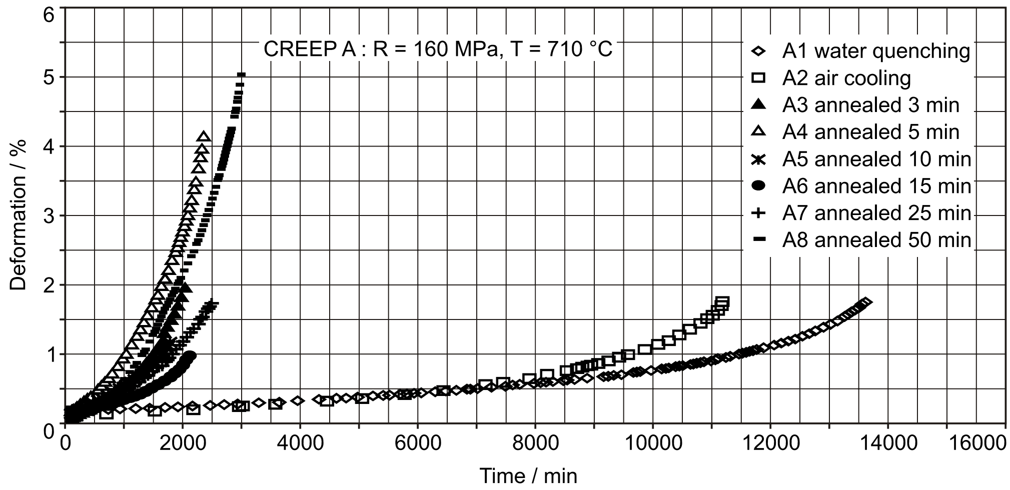

Figure 2. **Creep dependency for specimens from the A programme**
Slika 2. **Ovisnost puzanja za uzorke iz programa A**

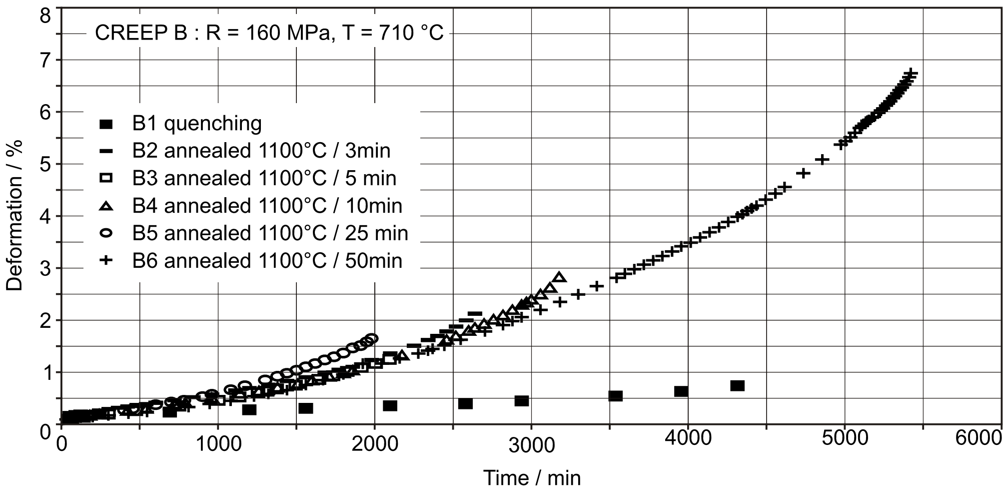

Figure 3. **Creep dependency for specimens from the B programme**
Slika 3. **Ovisnost puzanja za uzorke iz programa B**

The creep behaviour of the alloy processed according to and A and B programmes was very similar. The diagrams in Figure 4. and 5. summarise the relationship between creep lifetime, minimum strain rate and annealing time. The result shows that the creep strength of water quenched specimens from A programme was better than those from B programme. The explanation is again the higher work hardening in specimen after hot working. However, as creep results show, in both programmes creep lifetime decreased very sharply when specimens were annealed in range of 3-25 minutes where deformed grain structure was replaced by almost recrystallized structure. The effect of work hardening was eliminated quickly by recrystallization, which decrease also the creep lifetime difference between A and B pro-

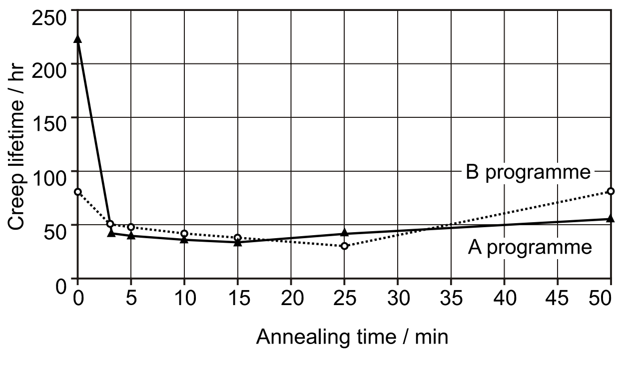

Figure 4. **The relationship creep lifetime versus annealing time for specimens from the programmes A and B**
Slika 4. **Odnos vremena puzanja o vremenu žarenja za uzorke iz programa A i B**

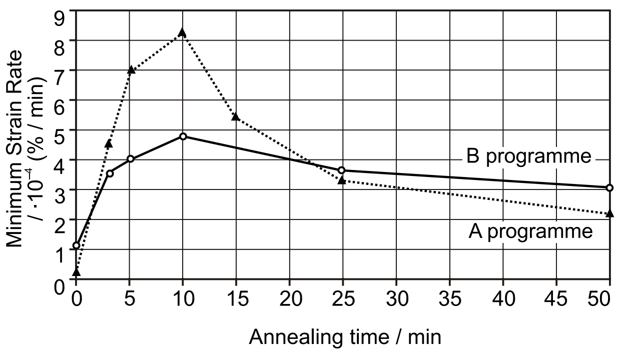

Figure 5. **Relationship minimal strain rate versus annealing time**
Slika 5. **Odnos minimalnog produljenja o vremenu žarenja**

grammes. When volume share of finer recrystallized grains increased gradually as the annealing time was prolonged over 25 minutes and the creep lifetime began to increase slightly, due to the grain coarsening. The prolonging of the annealing time over 25 minutes produced a slightly coarse and uniform grain structure, as shown in Table 2. and Table 3., and a small increase of creep strength.

Table 2. **Grain size for specimens of the A programme**
Tablica 2. **Veličina zrna za uzorke A programa**

Specimen No.	A1	A2	A3	A4
Average grains / mm^2	144	141	157	168
Average grain diameter	0,086	0,087	0,083	0,081
Specimen No.	A5	A6	A7	A8
Average grains / mm^2	187	201	184	136
Average grain diameter	0,077	0,074	0,078	0,088

Table 3. **Grain size for specimens of the B programme**
Tablica 3. **Veličina zrna za uzorke B programa**

Specimen No.	B1	B2	B3	B4
Average grains / mm^2	131	144	149	153
Average grain diameter	0,089	0,086	0,085	0,084
Specimen No.	B5	B6		
Average grains / mm^2	160	108		
Average grain diameter	0,083	0,099		

CONCLUSIONS

The results obtained for TM processing of NiMoCr alloy allow to conclude that the best creep strength was surprisingly obtained in case of applied two steps hot working without following annealing, and it was probably due to the effect of residual strain hardening resulting from hot working process. The annealing after hot working independently of the hold time, decreased the life time in comparison to the quenched or air cooled specimens with a microstructure modified only with dynamic recrystallization. The positive effect of strain hardening on creep behaviour of the alloy was changed with recovery and static recrystallization annealing.

The mixture of very fine recrystallized and deformed grain structure did ensure only a low creep strength. In order to improve the creep strength of the alloy the more uniform and coarse structure should be maintained by annealing time prolongation. The higher deformation during two steps hot working in A programme produced more uniform and finer recrystallized grain than those in B programme with lower creep strain rate and lifetime. After annealing, a fully recrystallized structure was not obtained independently of the deformation procedure before the annealing. To improve the creep properties of the alloy it is necessary to perform the recrystallization process in a way ensuring an uniform and coarse grained microstructure.

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