ANALYSIS OF THERMOMECHANICAL PROCESSING ON CREEP DEFORMATION BEHAVIOUR OF NiMoCr ALLOY

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ABSTRACT

It was found that annealed microstructure resulting from only one or two steps of hot working of NiMoCr alloy was not uniform through out specimens as desired. Therefore breaking down the casting ingot structure by hot working could provide only non-uniform recrystallized grain structure. In order to provide more uniform grain structure an additional cold working process was introduced where different reductions were applied after hot working. The following annealing process was supposed than to achieve uniform recrystallized grain structure. It was found that annealed microstructures after cold working were much more homogeneous than those without cold working. Furthermore, the uniformity of microstructure increased with higher amount of introduced deformation. The creep tests to evaluate the high temperature properties of alloy were carried out at stress of 160 MPa and at temperature of 710°C. The creep results showed that creep lifetime increased as reduction increased in range of 4.8 to 15% reduction. This was probably due to the majority effect of the remain work or strain hardening in non-recrystallized or deformed grains where still dislocation tangles after uncompleted annealing have been presented. For the highest reduction the stored energy from deformation was quite enough to modify and make more uniform grain structure through out the samples. However the resulting more uniform and finer recrystallized grains structure slightly decreased creep lifetime as an effect of smaller grain size.

KEYWORDS

Nickel-base alloy, Hot and cold working, Annealing process, Microstructure evolution, Recrystallized structure, Creep deformation.

INTRODUCTION

In present, nickel base alloy NiMoCr is experimental alloy in frame of the development ADTT (Accelerated Driven Transmutation Technology) loop for molten salt-type reactor [1]. Besides its resistance to radiation damage (thermal neutron) during fission production and corrosion resistance in hot liquid fluoride salts, other mechanical properties such as creep, low cycle fatigue (LCF) and thermal fatigue (TMF) resistances at working temperatures in reactor of the nuclear power plant are also fundamental material requirements. For our first development, the grain size of alloy is one of the most important features, as it can greatly influence its strength, creep, and fatigue crack initiation and growth rate.

The grain structure is a classical consideration, with uniform coarser grain size favouring increased creep strength, crack growth resistance and ductility. On the other side the uniform fine grain structure provides higher low cycle fatigue life and tensile yield strength [2]. The grain size optimisation and control can be achieved by hot working process, where plastic deformation at temperatures which are high enough for recovery and recrystallization to counteract strain hardening. The main goal for hot working, an ingot as semifinished product, is to refine and uniform ingot grains. Hot working process, by which microstructure development is controlled, is strongly dependent on the type of alloys [3].

Furthermore, the hot working followed by cold working and then annealing process can be the way to achieve the uniform recrystallized microstructure. The desired uniform coarse recrystallized microstructure do not only provide high creep strength and crack growth resistance but also promote resistance to thermal fatigue which all are prerequisites for the alloy. However, control of grain size is vital and difficult. A balance must be carefully considered to avoid excessive fine grains which decrease the creep strength and also to avoid excessively large grains which harm tensile and yield properties [4]. Up to present, however, the microstructure evolution by means of the hot and cold working of NiMoCr alloy ingot has been still less developed. Thus, in the present work the effort had been brought to develop and search for the thermomechanical processing to achieve uniform grain structure of the alloy. To modify microstructure for better mechanical properties, especially as creep, the different design of hot working with combination of cold working process condition were conducted with recrystallization.

A comprehensive study of creep deformation as a function of thermomechanical processing (TMP) conditions of NiMoCr alloy is presented in this work as well. The effects of temperature during hot working, amount of deformation introduced in hot and cold working are linked to the microstructure development approach in order to exploit fully the creep capabilities of the alloy. Since high temperature creep strength resistance is considered to be the mechanical property of major concern of alloy resistance at the working condition, the creep testing was carried out to evaluate the alloy creep resistance as a function of received structure parameters.

MATERIAL AND EXPERIMENTAL PROCEDURE

The investigated material was wrought nickel base NiMoCr alloy. Chemical composition of the alloy in wt. % is as following: 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 from casting process and then forged by multi-steps press forging-annealing process. However, the obtained alloy structure was very heterogeneous in grain size structure. In order to obtain more uniform microstructure the various hot working conditions following with different cold working and finally annealing process were designed and carried out as shown by details in Table 1.

Table 1. Details of sequence of hot working, cold working and annealing process of NiMoCr alloy

Specimen No.	Heating temperature conditions	Hot deformation [%]	Annealing at 1,130°C for 25 min	Cold deformation [%]	Annealing at 1,130 °C for 25 min.
A1	1,100°C/30min	11.3%+13.6%	Yes	-	-
A2	1,100°C/30min	11.3%+13.6%	-	6%	Yes
B1	1,200°C/30min	18% +18%	Yes	-	-
B2	1,200°C/30min	18% +18%	-	4.8%	Yes
B3	1,200°C/30min	18% +18%	-	6%	Yes
B4	1,200°C/30min	18% +18%	-	8%	Yes
B5	1,200°C/30min	18% +18%	-	10%	Yes
B6	1,200°C/30min	18% +18%	-	15%	Yes
B7	1,200°C/30min	18% +18%	-	20%	Yes

Then all samples were machined for creep testing. The creep tests were performed in air tensile creep testing machines, allowing the applied load to remain constant during testing. All creep tests were carried out at stress level of 160 MPa and temperature at 710°C. The specimen elongation versus time was recorded by two extensometers. The testing temperature was controlled by two Pt-PtRh thermocouples by means of thermal compensator. The temperature was maintained within the range of ± 5°C.

RESULTS AND DISCUSSION

Microstructure analysis

Using optical metallography, it was found that the microstructure of annealed cold worked specimens, for both A and B programmes, was more uniform than of non-cold worked specimens. When comparing the microstructure of two specimens subjected to the same cold deformation of 6%, however of different hot working condition (specimen A2 and B3 respectively) the specimen A2 manifested the less uniform microstructure than the specimen B3 where higher amount of hot deformation specimen received. That higher amount of hot deformation in specimen B3 provided better condition for uniform recrystallization process during hot rolling. It was also possible to document that the uniformity of microstructure resulted at B programme increased slightly as specimen cold reduction increased. The most uniform microstructure was obtained when the highest reduction of 20% was applied. Applying those this reduction at rolling only few excessive grains appeared in the structure what might be related to commencement of secondary recrystallization process. Considering grain size resulting from this reduction the relatively finer grain structure was observed comparing with structures where lower cold reductions were performed.

Creep Behaviour

The creep results presented in Figure 1, 2, and 3 implies that introduction of the cold reduction had strong effect on creep deformation behaviour and affected not only the creep lifetime but also strain rate and strain to failure. The individual contribution of cold working introduced in A programme, specimen A2, resulted in increasing of creep lifetime and creep strain rate decreasing comparing to specimen A1 where any cold deformation was not introduced. This specific deformation behaviour was, with high probability, due to the of effect of strain hardening remaining from cold deformation was governing the deformation no matter that the slightly finer grain structure was observed after the thermal treatment.

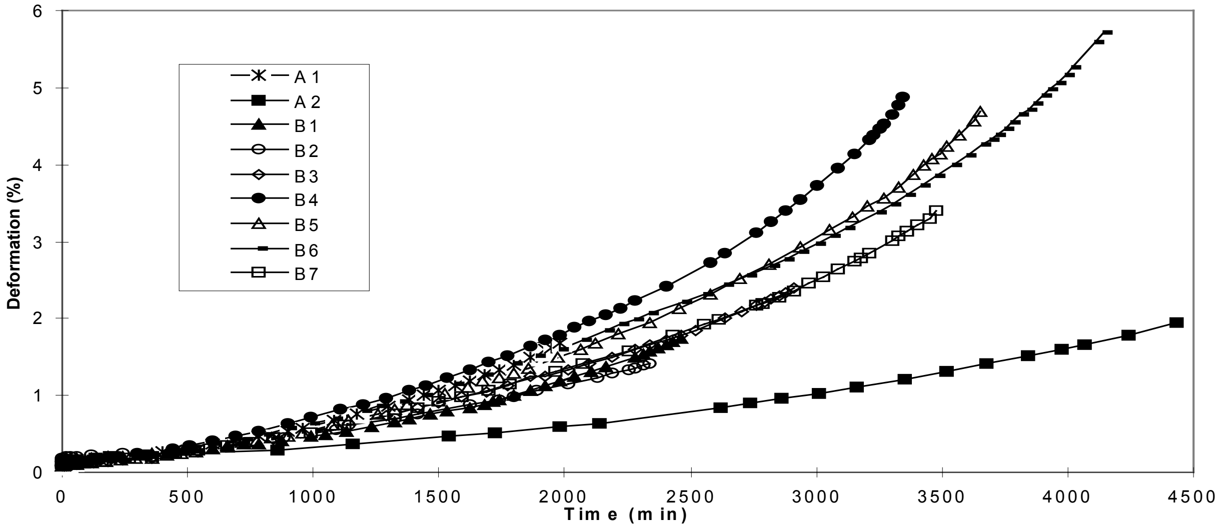

Fig. 1. Creep dependencies.

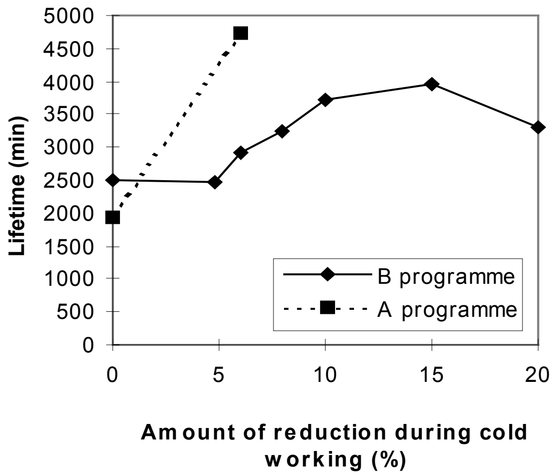

Fig. 2. The relationship between creep life and amount of reduction.

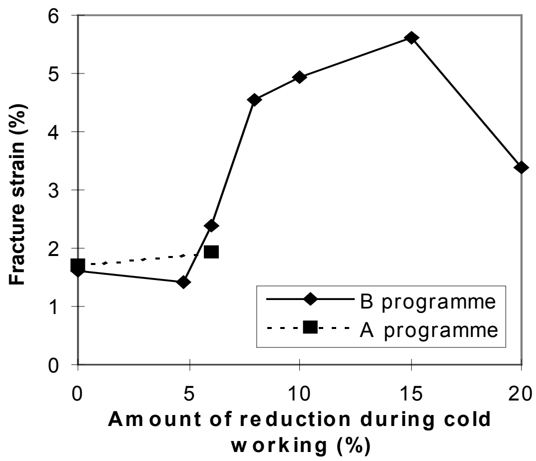

Fig. 3. The relationship between strain to failure and amount of reduction.

From Figure 2 and Figure 3, it could be documented that creep lifetime and fracture strain increase as reduction increases in range of 4.8-15% in B programme (specimens B2 -B6). This was probably due to the majority effect of the remain work or strain hardening in some volume portion of non-recrystallized or still deformed grains as a result of uncompleted recrystallization process within the specimen volume. In addition, these applied reductions might be providing not enough driving force through out the specimen, because of deformation heterogeneity, to guarantee the recrystallization to run uniformly across specimen. As a result of such condition the received structure was not uniform. However the applied annealing condition followed cold deformation allowed then the grain grew rapidly in some areas where lattice strain or stored energy was partially high. Such coarse grain microstructure then resulted in higher creep lifetime and strain to failure than uniform finer one. In case of the highest amount of reduction was realised, we propose that specimen deformation was uniformly distributed through out the sample. Due to the more uniform and finer recrystallized structure received, resulting of the slight decreasing of creep lifetime appeared. It should be noted if cold work would be over this criterion (20%) the effect of finer recrystallized grain structure would more pronounced.

Table 2. Grain size measurement

Specimen No.	A1	A2	B1	B2	B3	B4	B5	B6	B7
Average Grains/mm^2	162	188	196	217	238	295	351	374	486
Average grain diameter (mm)	0.082	0.077	0.075	0.071	0.067	0.061	0.057	0.051	0.046
ASTM (approximately)	≈ 4 -5	≈ 4 -5	≈ 4 -5	≈ 5	≈ 5	≈ 5	≈ 5 -6	≈ 5 -6	≈ 6

(The grains/mm^2 of initial sample before thermomechanical processing was 137 grains/mm^2)

Comparing A and B programmes as regards the creep strength of cold worked specimen the creep strength of A2 specimen was much higher than in case of all cold worked B specimens. This behaviour might be due to the different hot working condition applied before cold working and annealing process. Then lower amount of deformation during hot rolling in programme A provided lower strain hardening than those of B programme. Then the lower stored energy in microstructure should result in more coarsening grain structure than those of B programme. Besides the strength remained from work hardening in still deformed grains the detected microstructure coarsening of A programme (see Table 2) should also result in better creep strength resistance. Regarding this results for further experiment the only hot and cold working conditions in B programme will be considered and selected in processing of the alloy. It is expected that the more uniform microstructure resulting from B programmes should provide improved formability of alloy required for advanced alloy forming such as tube extrusion.

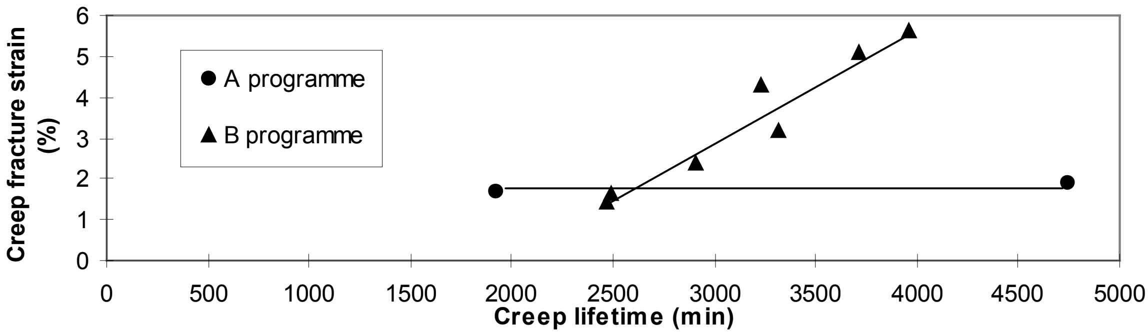

Fig. 4. The relationship between creep lifetime and creep fracture strain

Figure 4 it shows that creep fracture strain is increasing as creep lifetime increases according to B programme. Comparing A and B programmes, the results shows that fracture strain of B programme increases much more sharply than that of A programme. This could be probably due to dominant effect of more amount of finer recrystallized grain structure in B programme, which was resulted from higher amount of hot deformation applied. (Such microstructure can creep more easily than that of A programme), and/or it might be also due to the minor effect of remain strain hardening in A programme was annihilated more slowly than those in B programme. In B programme, because greater deformation from hot and cold work the higher driving force is supposed to carry out the process of recrystallization.

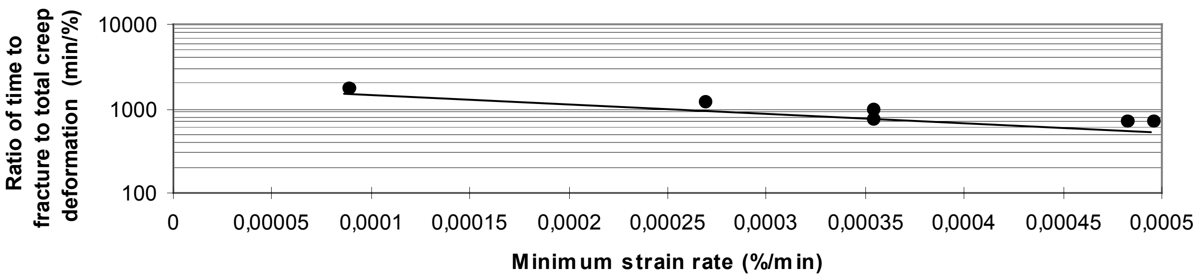

Fig. 5. Dependence of ratio of time to fracture to total creep deformation on minimum creep rate.

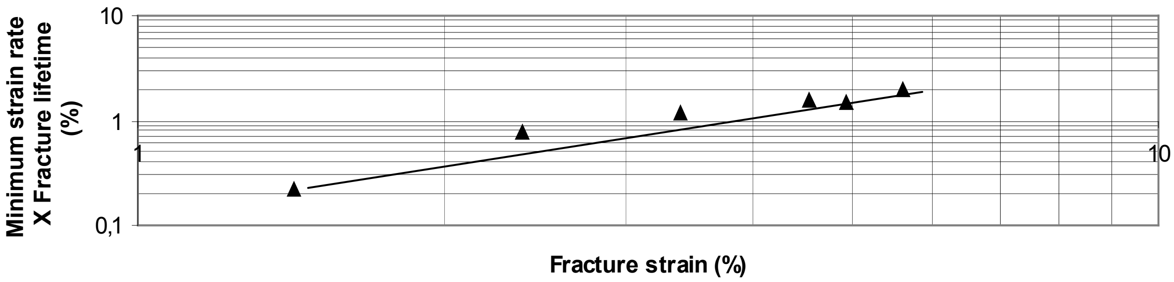

Fig. 6. Dependence of ratio minimum strain rate x time to fracture on fracture strain

From the results which are shown in Figures 5 and 6, the minimum creep rate ε_m ($d\varepsilon_m/dt$) and time to fracture t_f can be expressed by the following equations, respectively:

$$\log (t_F / \varepsilon_F) + m_1.(d\varepsilon_m/dt) = C_1 \quad (1)$$

$$\log (d\varepsilon_m/dt).\ t_f - m_2 \log \varepsilon_f = C_2 \quad (2)$$

where eqn. (1) has very similar form as proposed equation by Dobes-Milička [5],

$$\log (t_f / \varepsilon_f) + m \log (d\varepsilon_m/dt) = C \quad (3)$$

where ε_f is the deformation at fracture (or rupture), and C and m are material parameters. The proportionality between of the minimum creep rate and time to fracture to the total creep deformation could be considered, as shown by equation (1), which, however, according to our results, provides more precisely closer relationship than equation (3). Similarly, the proportionality of the strain to fracture to minimum creep strain rate multiplied by creep lifetime could be also expressed by equation (2).

CONCLUSION

On the basis of experimental results obtained from creep tests, the following conclusion can be noted. The introduction of different cold reduction during working followed after hot working had strongly beneficial effect on microstructure uniformity development characteristics and alloy creep strength. Although, the highest applied cold reduction (20%) did not provide the longest creep lifetime but the resulted uniform recrystallized microstructure was the most desired for further alloy forming process. It was proposed that this applied amount of cold reduction should be utilised in defined working process to restore the plastic properties of alloy after annealing process. Only the modified annealing process where the temperature and time should be involved to developed more coarse and uniform microstructure is expected to provide the improved creep properties of alloy.

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