THE CREEP BEHAVIOURS AFTER DIFFERENT ROLLING CONDITIONS IN NICKEL BASE ALLOY

Vrchovinský V. [1]*, Zrník J.* [1]*, Kvačkaj T.* [2]*, Wangyao P.* [3]
[1] *Dept. of Materials Science, Faculty of Metallurgy, Technical University, Košice, Slovak Rep.*
[2] *Dept. of Metal Forming, Faculty of Metallurgy, Technical University, Košice, Slovak Rep.*
[3] *Metallurgy and Materials Science Research Institute, Chulalongkorn University, Bangkok, Thailand,* e-mail: panyawat@hotmail.com

TEČENIE NIKLOVEJ ZLIATINY PO ROZDIELNYCH PODMIENKACH VALCOVANIA

Vrchovinský V. [1]*, Zrník J.* [1]*, Kvačkaj T.* [2]*, Wangyao P.* [3]
[1] *Dept. of Materials Science, Faculty of Metallurgy, Technical University, Košice, Slovak Rep.*
[2] *Dept. of Metal Forming, Faculty of Metallurgy, Technical University, Košice, Slovak Rep.*
[3] *Metallurgy and Materials Science Research Institute, Chulalongkorn University, Bangkok, Thailand,* e-mail: panyawat@hotmail.com

Abstract

Microstructure is one of the most important features to mechanical properties. It can strongly influence on strength, creep, and fatigue behaviours. Grain size is basic consideration which uniform coarse grain size is favouring creep strength, crack growth resistance, and ductility for next step forming. Optimisation and control of uniform grain size and morphology can be achieved by recrystallization process from annealing treatment after hot working. It was found that annealed microstructure resulting from only one or two steps of hot working in NiMoCr alloy was not uniform through out specimens as desired. Therefore breaking down the casting ingot structure by hot working process could provide only non-uniform recrystallized grain structure. In order to obtain more uniform grain structures an additional cold working was utilized. The following annealing process was supposed to achieve uniform recrystallized grain structure. It was found that annealed microstructures after cold working were much more uniform and homogeneous than those without any cold working. Furthermore, the uniformity of microstructure increased with higher amount of introduced deformation.

In this study, creep behaviour of modified microstructure in NiMoCr alloy, which has very similar chemical composition like in Hastelloy N, after different hot and cold working conditions and various annealing times was investigated. Creep tests were performed with stress level at 160 MPa and temperature at 710°C using uniaxial creep specimens. The results of creep tests showed that creep characteristics such as rupture strain, strain rate, and lifetime of the tested alloy were greatly dependent on the initial hot working conditions (temperature and amount of deformation in the process) and various annealing times.

Keywords: Hot and Cold workings, Recrystallization, Annealing, Nickel base superalloy, Creep

Abstrakt

Mikroštruktúra materiálu je jednou z najdôležitejších vlastností, ktoré majú vplyv na mechanické vlastnosti. Mikroštruktúra môže výrazne vplývať na pevnosť, tečenie a únavu materiálu. Veľkosť zrna je dôležitým parametrom, keď rovnomerné hrubé zrno je priaznivé pre medzu pevnosti pri tečení, odolnosť rastu trhliny a ťažnosť pre následné tvárnenie. Optimalizácia a kontrola rovnomernosti veľkosti zrna a jej morfológie môže byť získaná procesom rekryštalizácie zrna pri žíhaní materiálu po jeho valcovaní za tepla. Bolo zistené, že žíhaná mikroštruktúra zliatiny NiMoCr získaná buď jednostupňovým alebo dvojstupňovým procesom valcovania za tepla nebola rovnomerná, ako bolo požadované. Preto liaca štruktúra ingotu získaná procesom valcovania za tepla môže poskytnúť len nerovnomernú rekryštalizovanú štruktúru. Pre získanie rovnomernejšej štruktúry bolo dodatočne aplikované valcovanie za studena. Následný proces žíhania môže zabezpečiť rovnomernú rekryštalizovanú štruktúru. Bolo zistené, že žíhané mikroštruktúry dosiahnuté po procese valcovania za studena boli rovnomernejšie a homogennejšie ako tie, pri ktorých nebolo použité valcovanie za studena. Z tohto faktu vyplýva, že rovnomernosť mikroštruktúry narastá so zvyšujúcim sa stupňom deformácie.

V tejto práci bolo študované tečenie modifikovanej mikroštruktúry zliatiny NiMoCr, ktorá má veľmi podobné chemické zloženie ako zliatina Hastelloy N, po rozdielnych podmienkach valcovania za tepla a za studena a rozdielnych časoch žíhania. Skúšky tečenia boli urobené pri napätí 160 MPa a teplote 710°C použitím jednoosového namáhania. Výsledky skúšok tečenia ukazujú, že krípové charakteristiky, ako deformácia pri lome, rýchlosť deformácie a čas do lomu testovanej zliatiny výrazne závisí na počiatočných podmienkach namáhania (teplota a stupeň deformácie) a rozdielnych dobách žíhania.

Introduction

Nickel base alloy NiMoCr, a solid solution strengthening alloy was invented as a container material for molten fluoride salts in nuclear reactor. The alloy has the chemical composition very similar to Hastelloy N. Hence the NiMoCr alloy can be expected to have very similar performances like in Hastelloy N. Research has shown that materials which have high Mo content and low Cr content are generally superior to other materials in resisting high temperature corrosion in fluorine containing environments in the temperature range of 700 to 870°C for Hastelloy N [1]. In present, it is experimental alloy in frame of the development ADTT (Accelerated Driven Transmutation Technology) loop for molten salt-type nuclear reactor [2]. 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) resistance at working temperatures in reactor of nuclear power plant are also fundamental material requirements. For our first development, due to grain size is one of the most important features, as it can greatly influence strength, creep, and fatigue crack initiation and growth rate. The grain size is a classical consideration, with uniform coarser grain size favouring increased creep strength, crack growth resistance and ductility. And uniform fine grain structure provides higher low cycle fatigue life and tensile yield strength [3].

Since initial microstructure of materials from cast and semi-wrought processing has typical grain coarsening behaviour with very heterogeneous structure. It is widely well known that, besides the purpose for casting ingot break down and shape reduction, thermomechanical processing (TMF), such as hot working can also improve size and morphology of grain structure. By this way, microstructural features can be improved to increase mechanical properties at elevated temperature such as tensile, fatigue and creep strengths in materials. For creep strength enhancement, it has the purpose to provide a low rate of diffusion and/or plastic deformation at elevated temperatures inside the crystallites and inside the grain boundaries while preserving the compatibility of the material. Dislocation inside the crystals should have a low mobility. Grain boundary and phase boundary sliding can be limited and controlled by segregation or particles dispersions at the grain boundaries. This sliding should take place at the same rate as crystal lattice deformation to avoid the holes formation.

Due to all mechanical properties are strongly related to its microstructure [4 - 13]. Therefore, the size and morphology optimisation is classical consideration for improving creep strength by utilised the ideas above. The designed grain structure should be uniform coarsening grain size with beneficial segregation or particles dispersions at the grain boundaries resulting in higher creep strength, crack growth resistance [14 - 16]. This type of structure can be achieved by thermomechanical processing such as hot and cold working including annealing process by means of microstructure is modified by recrystallization process. Up to present, however, the microstructure evolution during the hot and cold working of ingot metallurgy of the NiMoCr alloy have been still less developed. Thus, in the present work had an effort to develop and search for the processing to achieve uniform grain structure. To modify microstructure for better mechanical properties such as creep, LCF and TMF, different designed hot working and/or combining with cold working conditions with various annealing times were utilised.

The purpose of this study was to examine the effect of amount of shape-reducing deformation during hot rolling process and annealing process on creep behavior in NiMoCr alloy. A comprehensive study of creep deformation as a function of hot working conditions of NiMoCr alloy is presented. It is well recognized that mechanical properties, such as, tensile, creep, and low cycle fatigue (LCF), strongly depend on morphology of grain structure, which obtained from hot working process, including temperature and amount of % reduction during the process as well as annealing conditions, are linked to the manner, in which developing microstructures approached in order to exploit fully the creep capabilities of the alloy. Since high temperature creep strength resistance is considered to be the most mechanical properties of major concern in this work. In this study, therefore, creep behavior of modified microstructure in NiMoCr alloy, with different condition in working history, was investigated. Creep tests were conducted at stress level at 160 MPa and temperature at 710°C using tensile creep specimens.

Experimental Material and Procedures

The investigated material was wrought nickel base alloy, NiMoCr alloy. Chemical composition of the alloy in wt. % is as follows: 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 multi steps press forging-annealing process. However, the obtained alloy structure was very heterogeneous. Then various testing hot working conditions were designed and carried out as shown by details in Table 1. 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 at 160 MPa and temperature at 710°C. The elongation with 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

1 Creep Behaviour in programs A and B

The creep results for TMP in programs A and B are presented in Figs. 1 and 2. It can be seen that various annealing times do not affect strongly on creep lifetime (≈ 2200 minutes in program A but do affect in program B in some degree). The water-quenched specimens and air-cooled specimens after hot working provided much longer lifetimes and better strength with lower creep rate than other annealed specimens. This was reasonable according to the effect of higher amount of work hardening still stored in deformed structure. Furthermore, its strength also obtained from non-uniform coarsening grain structure where was not fully replaced properly by finer dynamic recrystallized grains during hot working and fine static recrystallized grains during short-term annealing. Regard to these cooled specimens, which obtained high creep strength, they will not be considered for further use because of their dificulty in uniform formability for next forming step, such as extrusion or tube drawing. Therefore, annealed or recrystallized microstructure specimen after hot working should further be conducted in creep test for proper manufacturing conditions that could optimize creep characteristics.

Table 1 Details of hot working and cold working conditions

Specimen No.	Heating Temperature before Hot Working	% of Hot Deformation	Annealing Time (mins.) at 1100°C	% of Cold Deformation after Air Cooling	Annealing at 1130°C for 25 mins.
A1	1200°C/30 min	18 % +18 %	Quenching	-	-
A2	1200°C/30 min	18 % +18 %	Air Cooling	-	-
A3	1200°C/30 min	18 % +18 %	3 min	-	-
A4	1200°C/30 min	18 % +18 %	5 min	-	-
A5	1200°C/30 min	18 % +18 %	10 min	-	-
A6	1200°C/30 min	18 % +18 %	15 min	-	-
A7	1200°C/30 min	18 % +18 %	25 min	-	-
A8	1200°C/30 min	18 % +18 %	50 min	-	-
B1	1100°C/30 min	11.3 % +13.6 %	Quenching	-	-
B2	1100°C/30 min	11.3 % +13.6 %	3 min	-	-
B3	1100°C/30 min	11. 3% +13. 6%	5 min	-	-
B4	1100°C/30 min	11.3% +13.6%	10 min	-	-
B5	1100°C/30 min	11.3 % +13.6 %	25 min	-	-
B6	1100°C/30 min	11.3 % +13.6 %	50 min	-	-
C1	1200°C/30 min	18 % +18 %	-	4.8 %	Yes
C2	1200°C/30 min	18 % +18 %	-	6 %	Yes
C3	1200°C/30 min	18 % +18 %	-	8 %	Yes
C4	1200°C/30 min	18 % +18 %	-	10 %	Yes
C5	1200°C/30 min	18 % +18 %	-	15 %	Yes
C6	1200°C/30 min	18 % +18 %	-	20 %	Yes

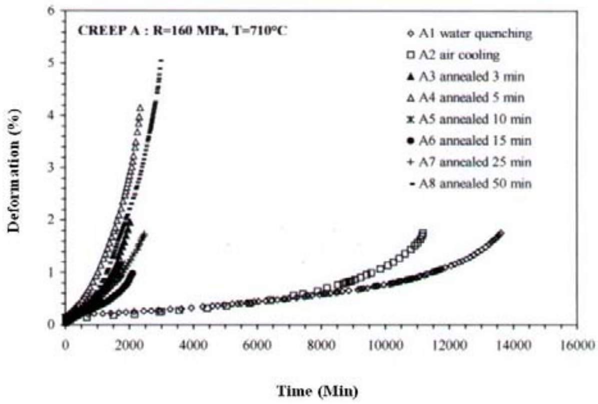

Fig.1 Creep curves of program A

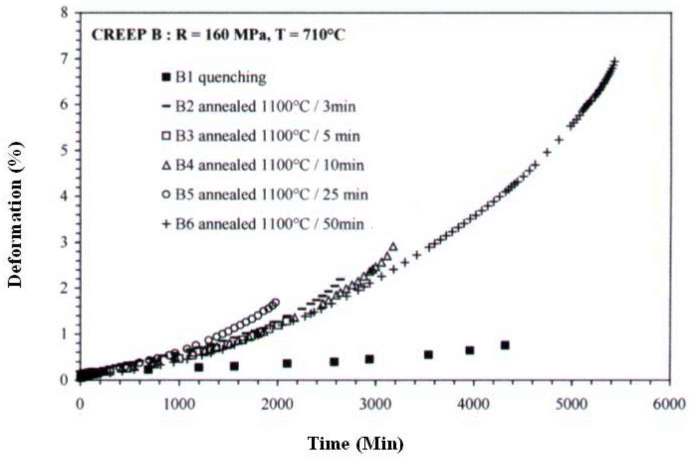

Fig.2 Creep curves of program B

Table 2 Creep properties of program A

Specimen No.	Annealing time at 1100 °C (minutes)	Creep lifetime (minutes)	Fracture strain (%)	Minimum creep rate ($x10^{-4}$ %/minute)
A1	0	13225.5	1.75	0.2976
A2	0	11453	1.8	0.3147
A3	3	2521	2.05	4.571
A4	5	2401.5	4.15	7.029
A5	10	2192	1.22	8.235
A6	15	2016	1.06	5.412
A7	25	2523	1.75	3.326
A8	50	3360	5.11	2.231

Table 3 Creep properties of program B

Specimen No.	Annealing time at 1100 °C (minutes)	Creep lifetime (minutes)	Fracture strain (%)	Minimum creep rate ($x10^{-4}$ %/minute)
B1	0	4815	0.75	1.153
B2	3	3002	2.25	3.571
B3	5	2821	1.33	3.978
B4	10	2526	2.91	4.762
B5	25	1813	1.41	3.654
B6	50	4291	4.72	3.081

The creep behaviours of the alloy processed according to and programs A and B have quite very similar manner. Data showed in Figs. 3 and 4, as well in Tables 2 and 3, summarise the relationships among creep lifetime, minimum strain rate and annealing time. It can be concluded that creep strength of water-quenched specimens from program A was better than those from program B. This was probably due to the higher effect of work hardening introduced in program A from higher deformation during hot working process than those in program B where total deformation was lesser. However, in both programs, creep lifetimes decreased very sharply when specimens were annealed in range of 3 - 25 minutes where deformed grain structures were replaced by recrystallization.

The effect of work hardening in deformed microstructure was also eliminated quickly by recrystallization during annealing process and then the creep lifetime difference between programs A and B was decreased. When volume portion of finer recrystallized grains was increased rapidly, such kind of microstructure was then easily deformed by creep process. In case of the longer annealing time in range of 25 - 50 minutes were, the creep lifetimes slightly increased again. The reason of an increase of the time to fracture should result from the grain coarsening. The effect of grain growth process would be corresponding to the secondary stage of recrystallization. Also the prolonged time of annealing over 25 minutes would produce the more coarsening and uniform grain structure, which resulted in better creep strength, as stated in Fig. 3.

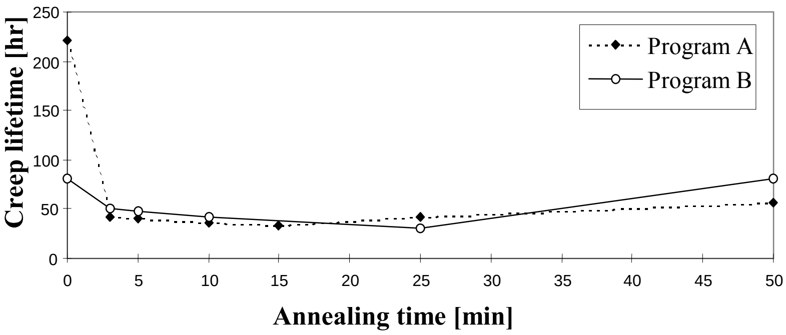

Fig.3 The relationship between creep lifetime and annealing time in programs A and B

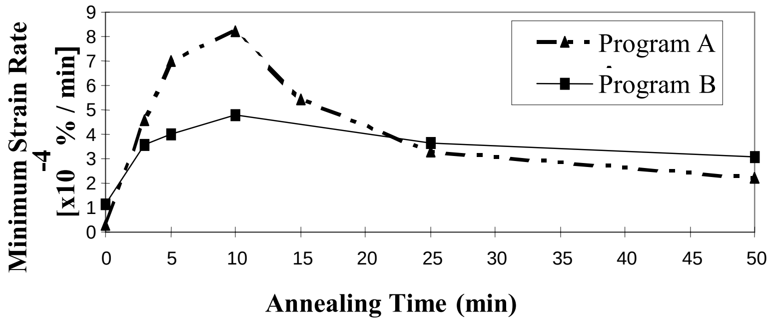

Fig.4 The relationship between Minimum strain rate and annealing time

Fig. 4 presents the relationship between the creep strain rate and annealing time. It implies that for short annealing period, creep strain rate curve increased sharply as annealing time increased till it reached the peak of both curves. This was due to the combination between new finer recrystallized and less remnant strain hardening in recovered grain structure, which was more susceptible to easier and faster creep deformation. Then after the peak points, strain rate curves start to decrease slowly as possible effect of new coarsening structure, which consists of grain growth (grain size effect). Comparing curves of both programs, it was found that the minimum creep strain rate increase from beginning point to peak points according to the increase of annealing time in program A was higher than that in program B, as the result of faster releasing stored energy. This resulted in more finely recrystallized grains generated more rapidly during annealing process in the range of 0 - 10 minutes in specimens of program A. Such microstructure might be the reason for the lower creep deformation resistance in program A comparing to program B, under the same condition. However, if annealing was longer than 10 minutes, both curves then started slightly decreasing and similar as effect of grain growth.

In Fig. 5, regardless of considering the annealing time in both programs, the dependence shows approximately the relationship between minimum creep rate and lifetime. It was found the relationship that minimum creep rate ($\dot{\varepsilon}_m$) is varying inversely with lifetime (t_f) as stated in this following form:

$$\mathrm{Log}\,(\dot{\varepsilon}_m) \quad \alpha \quad 1 / (\log t_f) \tag{1}$$

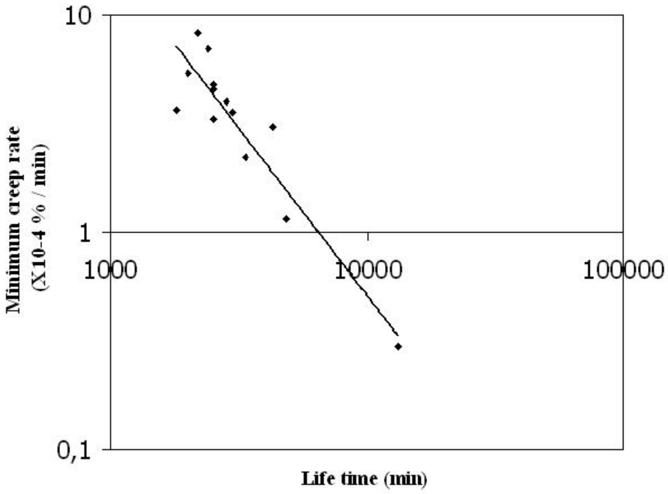

Fig.5 The relationship between minimum creep rate and lifetime

This implies that lower minimum creep rate, as a result of stronger microstructure, provides longer creep lifetime. Generally, when creep failure occurred in an intergranular behavior, as it was found in the present study, the time to failure (t_f) was usually found to increase linearly as the minimum creep rate decreased, so that:

$$t_f = M / \dot{\varepsilon}_m \quad \text{or} \quad t_f \ \mathrm{x}\ \dot{\varepsilon}_m = \text{constant (M)} \tag{2}$$

The expression is commonly known as the Monkman-Grant relationship [17]. Therefore, the creep behavior of the NiMoCr alloy can be discussed by using power law relationships for creep as:

$$1 / t_f \ \ \alpha \ \ \dot{\varepsilon}_m = A.\sigma^n \exp(-Q_c / RT) \tag{3}$$

The important conclusion could be drawn from these received results from the NiMoCr alloy when creep failure occurred in the intergranular manner. The Monkman-Grant relationship (Eqn. 2) was valid and indicated that the rate of intergranular damage development was controlled by the deformation rate. This could be considered in any micromechanism to account either for wedge-type or cavitation creep failure [18]. Moreover, for this alloy, the results showed that the Monkman-Grant relationship was not strongly affected by applied hot working conditions and annealing parameters.

2 Creep Behaviour in program C

It was found that the annealed microstructures resulting from only one or two steps of hot working of NiMoCr alloy were not uniform through out specimens as desired. Therefore, this breaking down the casting ingot structure by only hot working could not provide enough uniform recrystallized grain structure of alloy required for further manufacturing process. To solve this problem, an additional cold work (rolling) process where different % reductions were introduced after hot working was realized. Then further annealing process was applied with aim to achieve uniform recrystallized grain structure more available for alloy semifinal processing. It was also found that annealed microstructures after cold working were much more homogeneous than those received without any cold working [19]. Furthermore, the uniformity or alloy microstructure increased with higher amount of introduced deformation. The creep tests carried out at stress of 160 MPa and at temperature of 710°C, were to evaluate the effect of additional cold deformation.

Table 4 Creep properties of program C

Specimen Number	Amount of cold working after hot working (%)	Creep Lifetime, t_F (minute)	Fracture strain, ε_F (%)	Minimum Creep rate ($x10^{-4}$)
C1	4.8	2471	1.42	2.689
C2	6	2911	2.38	4.828
C3	8	3234	4.54	3.542
C4	10	3708	4.95	4.959
C5	15	3961	5.61	3.542
C6	20	3315	3.4	5.011

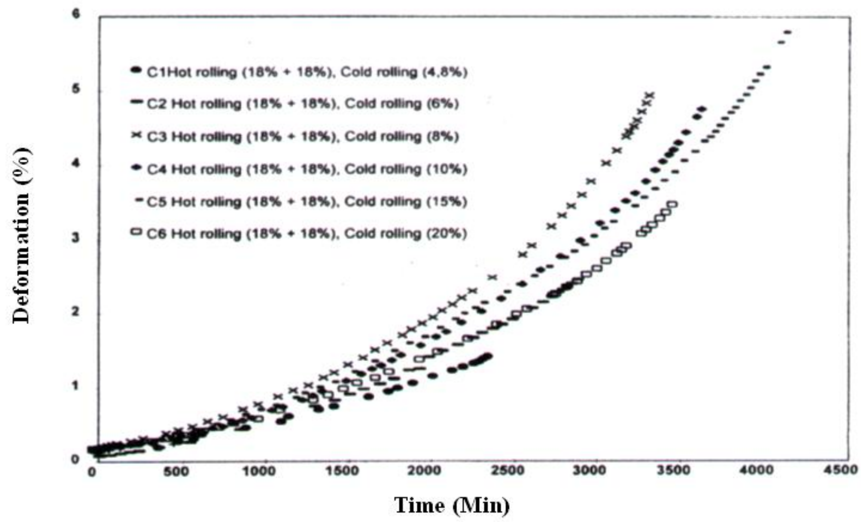

Fig.6 Creep deformation dependencies

For program C, the creep dependencies display in Figs. 6 - 8 and summaries are in Table 4, the creep test results show that amount of the applied reduction during cold rolling had strong effect on alloy creep behaviour resulting specifically in creep lifetime, strain rate and total fracture strain, i.e., influence on all those properties, which depend on grain structure.

From Figs. 7 and 8, it could be concluded that creep lifetime and fracture strain increased as amount of cold reduction was increasing in range of 4.8 to 15 % according to program C (specimen No. C1 - C5). Evaluating this effect of cold deformation on recrystallization, this result was probably due to the majority effect of the remained cold working or strain hardening in non-recrystallized or deformed grains after uncompleted annealing [20 - 24]. On the other hand, those applied cold reduction could not sufficiently generate uniform recrystallized grain structure in some areas in microstructure. However, this partial coarser structure was also resulted from insufficient annealing time to provide uniform recrystallized structure. Therefore, annealing of such deformed specimen with applied cold reduction likely allowed locally non-uniform rapid grain growing in some areas where lattice strain or stored energy was partially higher. On the other side, such more coarsening grain microstructure should be expected to result also in slightly higher creep lifetime and higher fracture strain than uniform and finer grain one. It is proposed for the highest amount of the applied cold reduction (20 %, specimen No. C6) that sample deformation would be homogeneous already and the stored energy is stored properly and uniformly through out the samples. Then, by considering this fact, this might result in more uniform and finer recrystallized grain structure, and on the other hand causing a slight decrease in creep lifetime and also in fracture strain. It should be expected that at exceeding of this deformation level (above 20 % cold reduction) the effect of finer recrystallized grain structure would be more pronounced.

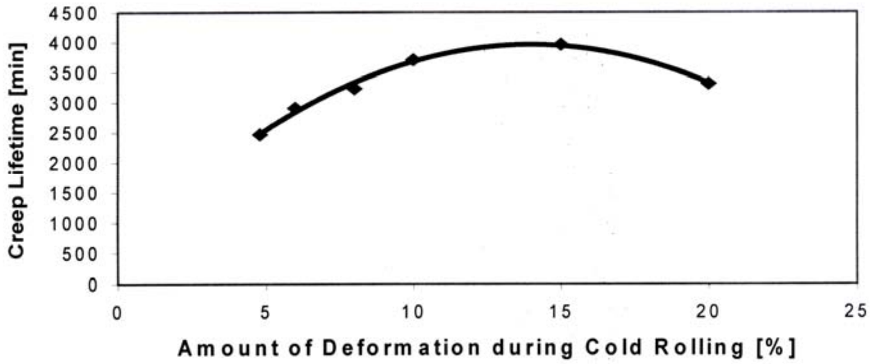

Fig.7 The relationship between creep lifetime and amount of cold reduction

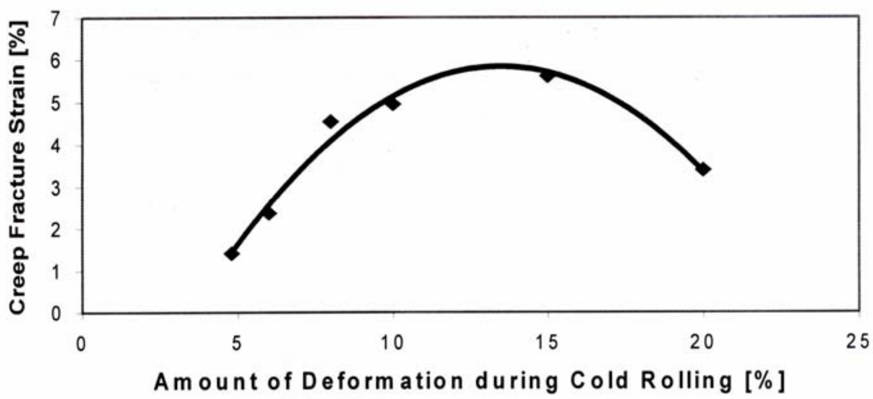

Fig.8 The relationship between fracture strain and amount of cold reduction

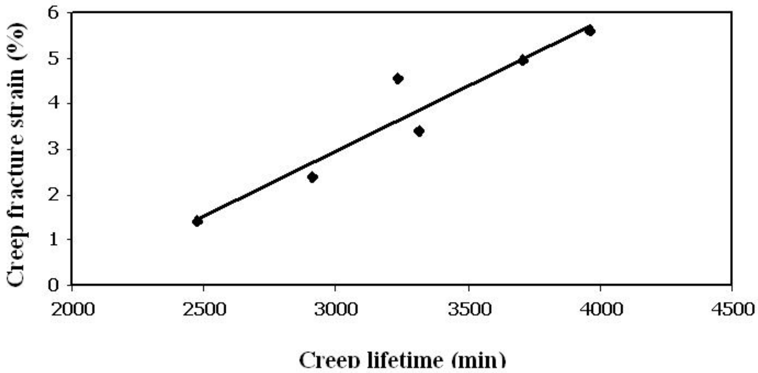

Fig.9 The relationship between creep lifetime and creep fracture strain

As Fig. 9 shows the creep fracture strain was increasing as creep lifetime increased in program C. The alloying elements, especially Mo and Cr, do improve creep strength [20], usually also result in increased resistance to creep fracture by impeding dislocation glide and recovery process at elevated temperatures. It can be also expected that the location of these atoms with carbon as carbides on grain boundaries can hinder or retard the ability of grain boundary sliding. Therefore, they suppress the intergranular cracks. Furthermore, as well as affecting creep rate and rupture life, alloying may also increase creep ductility. The longer creep lifetime, as strong creep strength, allowed more deformation during creep process as a function of time. In this case, with strong probability, the creep behaviour is not only influence by only grain size. In order to explain the creep dependence, it must be taken into account and consider prior remaining work hardening effect after TMP as well as effect of annealing process.

From the results, which show in Fig. 10, the dependence of the minimum creep rate $\dot{\varepsilon}_m$ ($d\varepsilon_m/dt$) and time to fracture t_F, the relationship could be tried to expressed by the following equation:

$$\log [(d\varepsilon_m/dt).t_F] - m \log \varepsilon_F = C \qquad (4)$$

where ε_f is the creep deformation at fracture, and C and m are material constants. The equation (4) expresses the proportionality of the strain to fracture to minimum creep strain rate multiplied by creep lifetime.

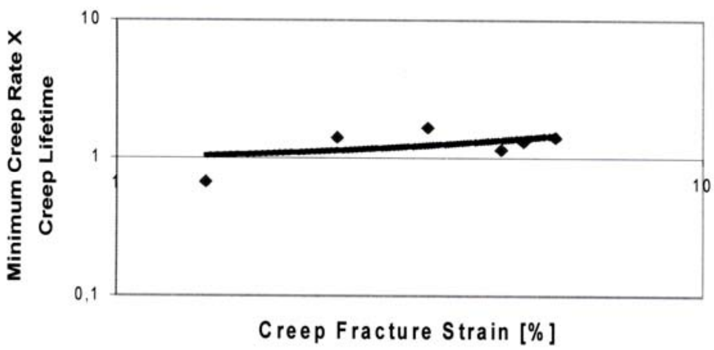

Fig.10 Dependence of ratio minimum strain rate x time to fracture on fracture strain

It should be noted that when dislocation process is dominant, alloying elements in solid solution can sometimes change even the detailed dislocation mechanisms affecting creep behaviour. Therefore, two categories of single-phase alloy can be defined as. [19]: a) Class M alloys show creep characteristics similar to pure metals and b) Class A alloys show anomalous creep behaviour. Most solid solution alloys show class M behaviour. For this NiMoCr alloy, creep curves from Figs. 1, 2 and 7 show that normal primary curves are always observed and recorded. Thus, the alloy is reasonably expected to be class M creep behaviour regardless the different structures resulting from TMP of the alloy.

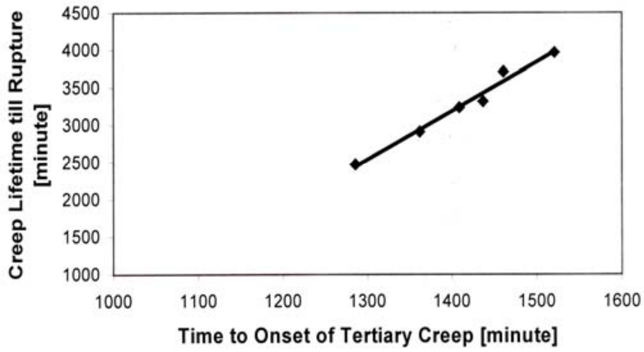

Fig.11 The relationship between time to onset of tertiary creep and creep lifetime

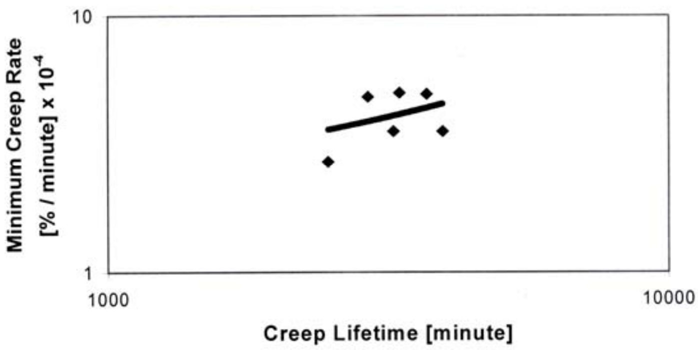

Fig.12 The relationship between creep lifetime and minimum creep rate

In Fig. 11, the dependence (slope of graph is more than 1) shows a good correlation and well-defined of properties between time onset of tertiary creep and creep lifetime, comparing to the correlation between minimum creep rate and creep lifetime, Fig. 12, which does not show sufficient reliable tendency of relationship. Furthermore, it should be noted that this dependence in Fig. 12 was contrast with the previous basic knowledge, which creep lifetime should increase with decreasing of minimum creep rate. For all cases in program C, creep at high temperature nearly always terminates in fracture. When normal creep curves were recorded, the first observable indication of fracture is the acceleration in creep rate marking the onset of the tertiary stage. When constant load creep test preformed, the stress gradually had increased as the cross section area of specimens had decreased with increasing strain. This was to cause the creep rate to accelerate. There could be probably also, generally, other possible causes for this acceleration such as microstructural instability, which includes grain growth, or (dynamic) recrystallization in the single-phase alloy and/or the nucleation and growth of internal microcracks which develop until the numbers and sizes of the microcracks were enough to increase creep rate.

Conclusions

1) The best creep strength was not surprisingly obtained only in case of two steps hot working without any annealing. This, as it was observed, was reasonably due to significant effect of residual strain hardening resulting from hot working process. In annealed specimens it was observed that all post-deformation with applied annealing periods caused lifetime decreasing comparing to quenched or air cooled conditions, where only dynamic recrystallization was supposed to appear.
2) The positive effect of strain hardening on creep behaviour of the alloy was continuously annihilated by recovery and static recrystallization during annealing process. The mixture of very fine recrystallized and still persisting deformed grain structure could provide only low creep strength. By prolongation annealing period the grains became much more uniform and coarsened resulting in slightly increase of the creep lifetime. Other conclusions are following:
3) The higher amount of deformation during 2 steps hot working in program A provides more uniform and finer recrystallized grain structure than those in program B resulting in lower creep strain rate and lifetime.
4) In both programs, the longest annealed specimen for 50 min. still have heterogeneous microstructure, this implies that longer annealing time should be utilised to allow uniform grain growth occurring for better creep lifetime.
5) On the basis of experimental results obtained from creep tests, the following conclusion can be noted. The introduction of % reduction during cold working after hot working had strongly effect on microstructure development and creep behaviour. Although, the highest tested % reduction (20%) could not provide the longest creep lifetime but its recrystallized microstructure was the most uniform. It can be suggested that by this % reduction during cold working should be utilised. Only the modified annealing process such as temperature and time should be studied and optimised more to achieve the most proper uniform coarsening microstructure, which should also provide the best creep lifetime.

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