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The Analysis of Creep Behavior in an Annealed Nickel Based Alloy

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ABSTRACT

The study investigated the creep behaviour of a nickel based solid solution strengthened NiMoCr alloy after different applied hot working conditions. Creep tests were conducted at a stress level of 160 MPa and a temperature of 710°C. The results showed that creep characteristics, greatly depend on the initial hot working conditions (heating temperature and degree of deformation in the process) and annealing time carried out prior to creep. Failure mechanisms including crack nucleation and crack propagation strongly depended on the grain boundaries. Fine recrystallized structures had provided much less resistance to creep deformation.

Keywords: Nickel-base alloy, Hot working, Annealing, Microstructure evolution, and Creep

INTRODUCTION

There were many microstructural features that have been developed in order to increase creep strength in materials. They all aimed to provide a low diffusion rate and/or plastic deformation rate at elevated temperatures inside the crystallites and inside the grain boundaries while preserving the compatibility of the material. Dislocation inside the crystals would have a low mobility. Segregation or particle dispersions at the grain boundaries can control grain boundary and phase boundary sliding. This sliding would take place at the same rate as crystal lattice deformation to avoid the void formation. Size and morphology optimization is classical consideration for improving creep strength by utilizing the ideas above. The designed grain structure would contain a uniform coarsening grain size with beneficial segregation or particle dispersions such as fine carbides at the grain boundaries resulting in higher creep strength and crack growth resistance. This type of structure can be achieved by a thermomechanical processes such as hot working, annealing, by means of modified microstructures via a recrystallization process.

The purpose of this study was to examine the effect of the amount of shape-reducing deformation during the 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 the morphology of the grain structure, which is obtained from hot working parameters, including temperature and the amount of percent 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 resistance is considered to be the most mechanical properties of major concern in this work. Therefore, the creep behavior of modified microstructures in the NiMoCr alloy, which has a similar chemical composition as Hasteloy N, with different conditions in working history, was investigated. Creep tests were conducted at a stress level of 160 MPa and a temperature of 710°C using tensile creep specimens.

Materials and experimental procedure

The material was a solid solution strengthened nickel based NiMoCr alloy. The chemical composition of the alloy in wt.% was shown in Table 1. The initial alloy was produced from a casting process and then forged by a multi-step forging-annealing process. However, the as-received structure consists of a non-uniform grain structure. In order to obtain uniform structure, the experiments on hot working process followed by recrystallization annealing were conducted using two different programs as shown:

Program A: Specimens were preheated at 1,200°C for 30 minutes before two steps hot working resulted in reduction (18%+18%). Hot worked specimens were air-cooled (A1) and quenched (A2). Other deformed bands were cooled to room temperature and then annealed at 1,100°C for 3, 5, 10, 15, 25 and 50 minutes (A3-A8, respectively).

Program B: Specimens were preheated at 1,100°C for 30 minutes before two steps hot working resulted in reduction (11.3%+13.6%). Hot worked specimens were quenched (B1). Other deformed bands were air-cooled to room temperature and then annealed at 1,100°C for 3, 5, 10, 25 and 50 minutes (B2-B6, respectively).

Subsequently all samples were machined to specimens for creep testing. The creep tests were conducted in tensile creep testing machines at constant load. All creep tests were carried out at a stress level of 160 MPa and a temperature of 710°C. The elongation with time was recorded by two extensometers. The testing temperature was controlled by two Pt-PtRh thermocouples by means of a thermal compensator. The temperature was maintained within the range of ±5°C.

Table 1 Chemical composition of NiMoCr alloy (wtt.)

Ni	Mo	Cr	Fe	Al	Ti	W	Co	Si	Cu	B	S	C
72.7	17.8	6.3	2.8	0.16	0.06	0.06	0.06	0.05	0.01	0.01	0.001	0.02

RESULTS AND DISCUSSION

Creep behaviour

The creep results for TMP in program A and B are presented in Figures 1 and 2. It can be seen that various annealing times did not affect creep lifetime (≈ 2,200 minutes in program A and ≈3,000 minutes in program B). The water-quenched specimens and air-cooled specimens after hot working produced much longer lifetimes and better corresponded with a lower creep rate than the annealed specimens. This was reasonable according to the effect of the higher amount of work hardening still stored in the deformed structure (Wangyao, *et al.* 2001). Furthermore, its strength is also obtained from the non-uniform coarsening grain structure which was not fully replaced by finer dynamic recrystallized grains. Although these cooled specimens obtained high creep strength they will not be considered for further use because of their poor uniform formability for the next forming step, such as extrusion or tube drawing. Therefore, the annealing or recrystallized microstructure specimen after hot working would be further creep tested in order to seek proper manufacturing conditions that optimize the creep characteristics.

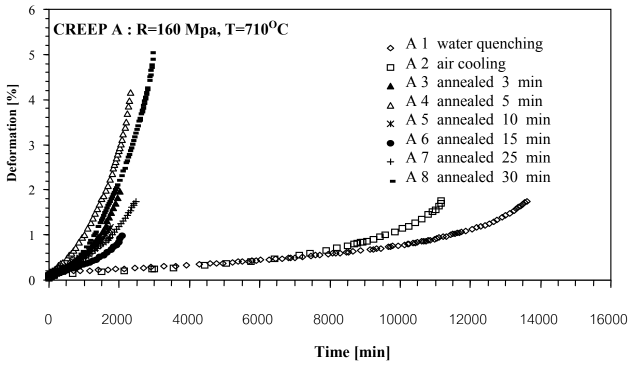

Figure 1 Creep **curves** of program A

The creep behavior of the alloy processed according to program A and B has very similar properties. Figures 3 and 4, Tables 2 and 3, summarized the relationship among creep lifetime, minimum creep strain rate and annealing time. In conclusion, specimens from program A had a higher amount of remaining work hardening than those of B due to more severe deformation during hot working. Therefore, the strength of the water-quenched specimens from program A is better than program B (Wangyao, *et al.* 2001).

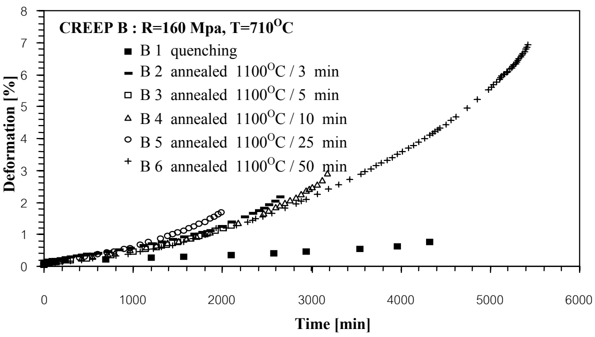

Figure 2 Creep **curves** of program B

However, creep lifetimes decreased sharply when specimens were annealed in the range of 3-25 minutes. This was because deformed grain structure was replaced by recrystallization. The effect of work hardening in deformed microstructure as seen in the creep lifetime difference between A and B was decreased as annealing time increased. As the quantity of finer recrystallized grain increased, the microstructure was easily deformed via the creep process (Wangyao, *et al.* 2000). In case of longer annealing time in the range of 25-50 minutes creep lifetime slightly increased again. This prolongation of lifetime resulted from the grain coarsening (Wangyao, *et al.* 2000; and (Wangyao, *et al.* 2001). The effect of grain growth process would be corresponding to the secondary stage of recrystallization. Also prolonged annealing time over 25 minutes would produce the coarser and more uniform grain structure, which might result in slightly increasing the creep strength, as stated in Figure 3.

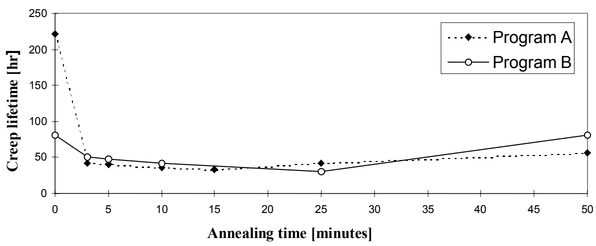

Figure 3 The relationship between creep lifetime and annealing time in program A and B

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

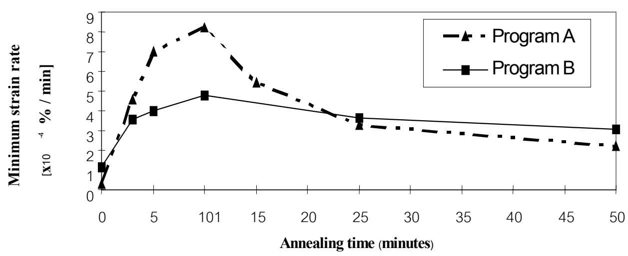

Figure 4 The relationship between minimum strain rate and annealing time

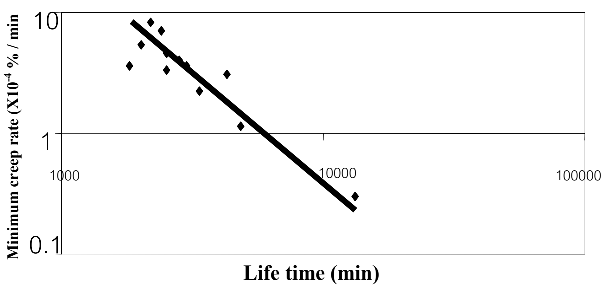

Figure 5 The relationship between minimum creep rate and lifetime

In Figure 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 that the minimum creep rate ($\dot{\varepsilon}_m$) is varying inversely with lifetime (t_f) as stated in this following form:

$$\text{Log}(\dot{\varepsilon}_m) \quad \alpha \quad 1 / (\log t_f) \;\ldots\ldots\ldots\ldots\ldots\ldots 1)$$

This implies that a 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 study, the time to failure (t_f) was usually found to increase linearly as the minimum creep rate decreased, so that:

$$t_f = M/\dot{\varepsilon}_m \text{ or } t_f \times \dot{\varepsilon}_m = \text{constant } (M) \ldots\ldots\ldots\ldots 2)$$

The expression is commonly known as the Monkman-Grant relationship (Evangelisla, *et al.* 1995). Therefore, the creep behaviour of the NiMoCr alloy can be discussed by using power law relationships for creep as:

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

An 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 (Evans and Wilshire, 1993). Moreover, for this alloy, the results showed that the Monkman-Grant relationship was not strongly affected by applied hot working conditions and annealing parameters.

Table 2 Creep properties of program A

Specimen No.	Annealing time at 1,100 °C (minute)	Creep lifetime (minute)	Fracture strain (%)	Minimum creep rate (x10^{-4} % / minute)
A1	0	13,225.5	1.75	0.2976
A2	0	11,453	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 1,100 °C (minute)	Creep lifetime (minute)	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

Creep fracture Analysis

The SEM fracture analysis of broken specimens was employed to trace crack initiation site and fracture morphology, as well as the fracture mode, with consideration to microstructure. Metallography examination along a plane normal to the crack propagation of the failure specimen after different hot working and annealing conditions had been done. It was found that different hot working conditions applied before the annealing process and introduced annealing time did not show any significant effect to the mode of creep fracture in broken specimens. The initiation of fracture crack was dominantly intergranular and the crack, one or more, nucleated on the free surface of specimens, Figure 6. In the figure, it is possible to see the heterogeneous grain structure after creep fracture. From this nucleation site, cracks would continue to propagate along the grain boundaries in the early stage of the deformation process.

Figure 6 Crack nucleation and propagation in intergranular fracture

The growth of critical crack size was normal to applied stress and continuously formed and grew in an intergranular manner to form microcracks. Eventually, they propagated to a main crack and developed into a final fracture, which was macroscopically brittle. Under the applied creep testing condition of the alloy, the grain boundaries were preferential sites to crack nucleation and propagation. Secondary microcracks were also found at the specimen surface. Corresponding intergranular fracture surfaces were very smooth. Probably, intergranular cracks developed by nucleation, growth and link-up of grain boundary cavities. Here, it will be stressed on intergranular damage because intergranular cavitation and fracture in general are a sequence of the joint action of creep deformation and diffusional process (Evans and Wilshire, 1993).

Microvoids would probably, but not always, be initiated at inclusions or small second-phase particles in grain boundaries. In this case, it might probably be small carbides. Creep deformation continued when the numbers and sizes of the cavities were critical and potential for more crack nucleation. Cavities form continuously throughout the high temperature creep process. However, according to the previous work Granet (1980), in general, the third stage of creep, the nucleation and growth of grain boundary voids and cracks usually occur at an accelerated rate. The extremely high stress normal to grain boundaries was expected to nucleate cavity. Thus, very large stress concentrations were required. In this case, they could be produced at particles in sliding grain boundaries, at the intersection of slip bands and grain boundary particles and grain boundary triple points.

In general, a large number of cavities was not expected to form at the beginning of a creep test, but rather they accumulated as time of testing proceeded. Due to under applied creep conditions, the generation of high stress concentrations is moderated by power-law creep and diffusional creep process (Evans and Wilshire, 1993). The important features of the multi-linked grain boundary surface are facets (two-grain junctions: faces of approximately polyhedral grains), edges (three-grain junctions: edges of grains), and corners (four grain junctions: vertices of grains). These could possibly be nucleation sites for cavities but the actual nucleation and/or growth conditions may be differing significantly depending on the external condition (Sklenicka, 1997).

After critical crack size opening its further propagation, it was mixed fracture modes between intergranular and transgranular cleavage in a small area portion of fracture specimens, as illustrated in Figure 7. Several processes could cause or contribute to the acceleration in creep rate during the tertiary stage. It was documented that the development of microcracks usually leaded to creep fracture and combined with mechanical instabilities, such as, necking. Only at the final stage of the rupture process, the transgranular ductile facets appeared on the fracture surface, as shown in Figure 8, in the type of dimple fracture morphology. These fracture facets were only observed close to the fracture surface in an area of unstable crack growth (fast growth at a high deformation rate).

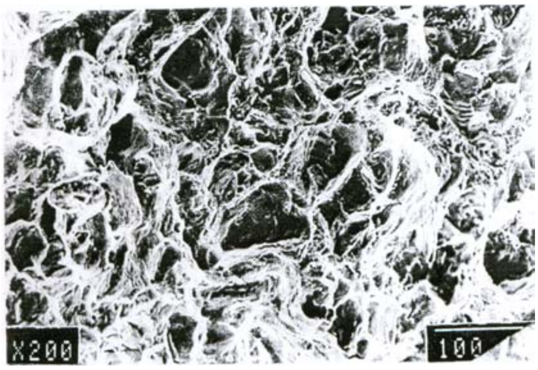

Figure 7 Mixing mode fracture facet

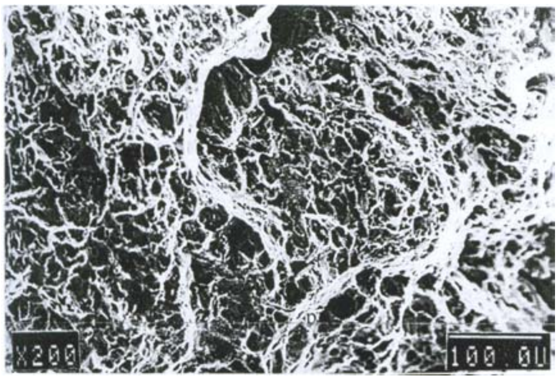

Figure 8 Crack propagation in transgranular ductile fracture (dimple)

In transgranular ductile creep fracture mode, internal cracks or voids were nucleated in grains, usually around inclusions but they could probably form in other regions of non-homogeneous microscopic strains during the creep process. The transgranular creep fracture was preceded previously and rapidly by the growth and eventual coalescence of these holes. The fracture process, which led to a considerable reduction in areas, was a macroscopically ductile fracture. A transgranular creep fracture is usually associated with a high strain rate or equivalently and immediately very high stress before the final fracture of specimens. In this alloy, when cross section areas after intergranular cracks reached the critical area, it resulted in building up enough high stress for crystallographic flow to complete the process of ductile fracture in the fast fracture state. Morphology differences were not observed in fracture mechanisms of individual fracture modes for applied different material processing conditions.

CONCLUSION

The best creep strength was not obtained in case of two steps hot working without any annealing. This, as it was observed, was due to the significant effect of residual strain hardening resulting from the hot working process. In annealed specimens, it was found that post-deformation with applied annealing periods caused the lifetime to decrease compared to quenched or air cooled conditions, where only dynamic recrystallization was supposed to appear. The positive effect of strain hardening on creep behavior of the alloy was continuously annihilated by recovery and static recrystallization during the annealing process. The mixture of very fine recrystallized and still persisting deformed grain structure could provide only low creep strength. By prolongation of the annealing period, the grains became much more uniform and coarse resulting in a slight increase of the creep lifetime. Other conclusions are as follows:

1. The higher amount of deformation during 2 steps of hot working in program A provides more uniform and finer recrystallized grain structures than those in program B resulting in a lower creep strain rate and increasing lifetime.
2. In both programs, the creep rate decreased and the annealing time increased, it implies that a longer annealing time should be utilized to allow uniform grain growth to occur for a better creep lifetime.
3. Initiation of fracture was predominantly intergranular. After critical crack size openings, its further propagation was mixed fracture mode between intergranular and transgranular cleavage in a small area portion of fracture specimens. Only in the final stage of the creep rupture process, transgranular ductile facets appeared on the fracture surface in the type of dimple fracture morphology.

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