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Effect of Final Cold Rolled Microstructures on Creep Deformation Behavior in Nickel Base Alloy

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Abstract

It was found that the annealed microstructure resulting from only one or two steps of hot working in NiMoCr alloy was not uniform throughout the specimens as desired. 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 the annealed microstructures after cold working were more homogeneous than those without any cold working. Furthermore, the uniformity of the microstructure increased with a higher amount of introduced deformation. The creep tests to evaluate the high temperature strength of the alloy were carried out at a stress of 160 MPa and at a temperature of 710°C. The results showed that creep characteristics, namely rupture strain, strain rate, and lifetime, greatly depend on the initial cold working conditions (degree of reduction) carried out prior to creep test. The creep results showed that creep lifetime increased as reduction increased in a range of 4.8 to 15%. This result was probably due to the majority effect of the strain hardening in non-recrystallized or deformed grains where 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, a more uniform and finer recrystallized grains structure slightly decreased creep lifetime as an effect of smaller grain size.

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

Introduction

Nickel base alloy NiMoCr is an experimental alloy for a 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 (TME) resistances at working temperatures in the 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 favoring 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). Because mechanical properties are related to microstructures. many research works(3-20) had been carried out to investigate these relationships of microstructure-mechanical properties in nickel base alloys. The grain size optimization and control can be achieved by hot working process, the plastic deformation at temperatures which are high enough for recovery and recrystallization to counteract strain hardening. The main goal of hot working, an ingot as a semi-finished product, is to refine ingot grains. The hot working process, by which microstructure development is controlled, is strongly dependent on the type of alloys.

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Since the initial microstructure of materials from casting processing has a typical grain coarsening behaviour, which is a non-uniform structure, grain size optimization and control can be achieved by the hot working process. Many works(21-29) had been done to control and modify recrystallization and grain growth as desired uniform structures in the alloy in order to optimize mechanical properties at elevated temperatures. However, Hastelloy N which was compared to the NiMoCr alloy has a quite wide range of hot working temperatures (870-1180°C) (30), because there is no need to consider the effect of secondary phase precipitate such as gamma prime and/or carbides which control the recrystallization process during hot working.

Furthermore, the hot working followed by cold working and then the annealing process can be the way to achieve the uniform recrystallized microstructure. The desired uniform coarse recrystallized microstructure does not only provide high creep strength and crack growth resistance but also promotes resistance to thermal fatigue which 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 that decrease the creep strength and to avoid excessively large grains, which is harm ful to tensile and yield properties(31). Up to the 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 an effort had been made to develop and search for the thermomechanical processing to achieve a uniform grain structure of the alloy. To modify the microstructure for better mechanical properties, especially creep, the different design of hot working with a combination of cold working process conditions were conducted with a final recrystallization annealing process.

The purpose of this study was to examine the effect of the amount of shape-reducing deformation during cold rolling followed by annealing on the creep behavior of NiMoCr alloy. A comprehensive study of creep deformation as a function of cold working condition 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 was obtained from the 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 property of major concern in this work, creep behavior of modified microstructure in NiMoCr alloy, which has a similar chemical composition as Hastelloy N and 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 investigated material is a solid solution strengthened nickel base NiMoCr alloy. The chemical composition of the alloy in wt.% is shown in Table 1. The initial alloys was obtained from a casting process and then forged by a multi-step forging-annealing process. However, the sample structure consists of a non-uniform grain structure. In order to obtain a more uniform structure, the experiments on the hot working process followed by recrystallization annealing were conducted with various reduction percentages during cold rolling and then annealing, as details are shown in Table 2. The additional effect of several chosen cold deformation reductions were introduced after hot working procedures in order to analyze its effect on the recrystallization behaviour of the alloy and its creep behaviour. Then, the samples were machined to specimens for creep testing. The creep tests were conducted in tensile creep testing machines at applied 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

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

Table 2. Details of hot and cold working conditions

Specimen No.	Heating Temperature before Hot working	% Hot deformation	% cold deformation after Air-cooling	Annealing at 1130 °C for 25 minutes
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

Results and Discussion

Creep deformation behaviour of the alloy

It was found that the annealed microstructures resulting from only one or two steps of hot working of NiMoCr alloy were not uniform throughout the specimens as desired (21, 27, 29). Breaking down the casting ingot structure by only hot working could not provide enough uniform recrystallized grain structure of alloy required for a further manufacturing process.

To solve this problem, an additional cold rolling process where different % reductions were introduced after hot working was conducted. Then further annealing was applied with the aim to achieve a uniform recrystallized grain structure. It was also found that the annealed microstructures after cold working were much more homogeneous than those without any cold working. Furthermore, the uniformity of the alloy microstructure increased with a higher amount of introduced deformation. The creep tests carried out at a stress of 160 MPa and at a temperature of 710 °C, to evaluate the effect of additional cold deformation.

Table 3. Creep properties of C program

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

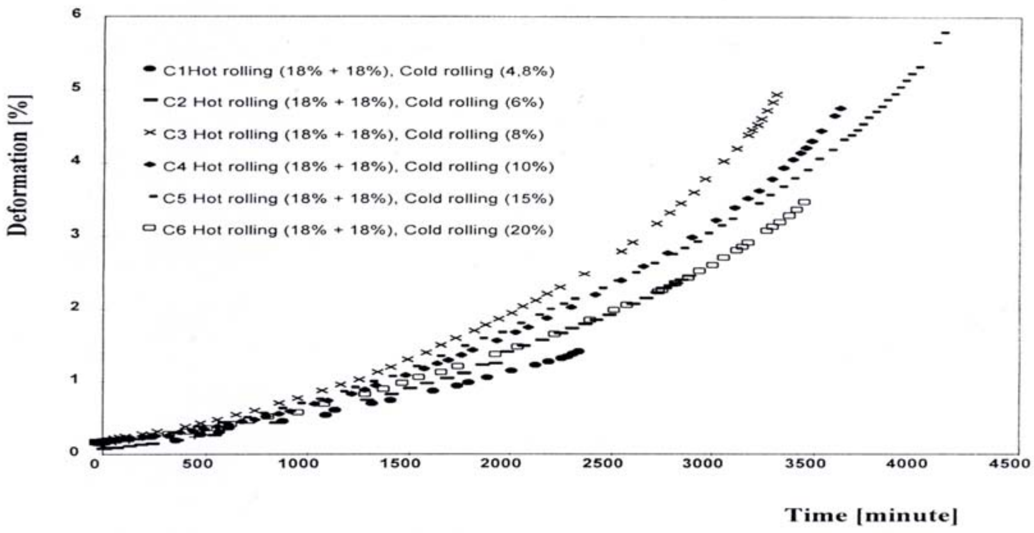

Figure 1. Creep deformation dependencies

The creep dependencies are displayed in Figures 1-3 and summaries are in Table 3, the creep test shows that the amount of cold reduction had a strong effect on alloy creep behaviour resulting specifically in creep lifetime, strain rate and total fracture strain, which depend on grain structure, as seen in Table 4.

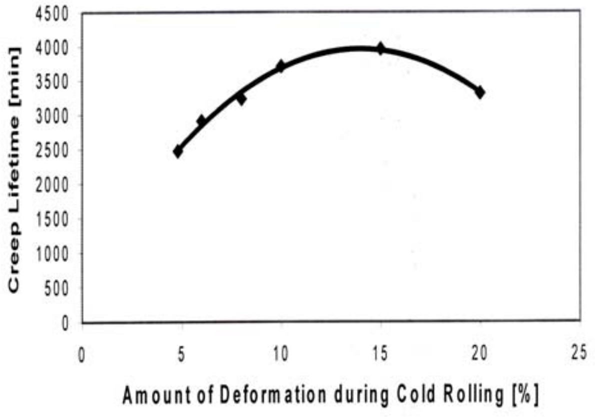

Figure 2. The relationship between creep lifetime and amount of cold reduction.

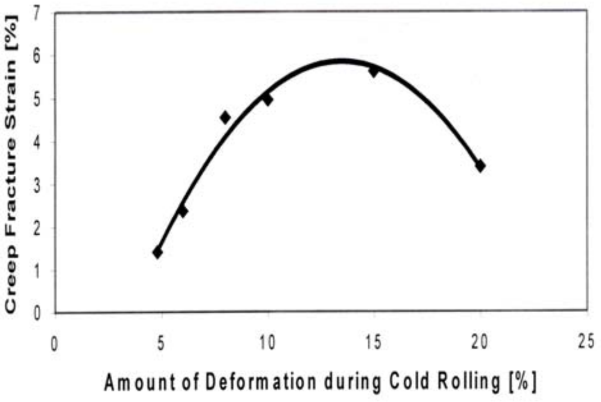

Figure 3. The relationship between fracture strain and amount of cold reduction.

Table 4. Grain size measurement

Specimen No.	C1	C2	C3	C4	C5	C6
Average Grains (mm^2)	217	238	295	351	374	486
Average grain diameter (mm)	0.071	0.067	0.061	0.057	0.051	0.046
ASTM No. (Approximately)	≈ 5	≈ 5	≈ 5	≈ 5 - 6	≈ 5 - 6	≈ 6

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

From Figures 2 and 3, it could be concluded that creep lifetime and fracture strain increased as the amount of cold reduction was increased in the range of 4.8 to 15% (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(32-361). On the other hand, those that were applied to cold reduction could not sufficiently generate a uniform recrystallized grain structure in some areas in the microstructure. However, this partially coarser structure was also resulted from an insufficient annealing time to provide a uniform recrystallized structure. Therefore, the annealing of such a 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, a coarser grain microstructure should be expected to result also in a slightly higher creep lifetime and a higher fracture strain than a uniform and finer grain one. It is proposed for the highest amount of cold reduction (20%) 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 a more uniform and finer recrystallized grain structure, and causing a slight decrease in creep lifetime and in fracture strain, as shown in Figures 2 and 3. respectively. It should be expected that at the exceeding of this deformation level (above 20% cold reduction) the effect of a finer recrystallized grain structure would be more pronounced.

Figure 4 shows the creep fracture strain was increasing as creep lifetime increased. The alloy additions, especially Mo and Cr, improve creep strength(37), usually result in increased resistance to creep fracture by the impeding dislocation glide and recovery process. It can be also expected that the location of these atoms on grain boundaries can hinder grain boundary sliding, therefore, suppressing the intergranular cracks. Furthermore, as well as affecting creep rate and rupture life, the alloying element may increase creep ductility. The longer creep lifetime allowed more deformation during the creep process as a function of time. In this case, the creep behaviour is not only influenced by only grain size. In order to explain the creep dependence, it must be taken into account the prior remaining work hardening effect after TMP as well as the effect of the annealing process.

Figure 5. Shows the dependence of the minimum creep rate ε'_m ($d\varepsilon_m/dt$) and time to fracture t_F, the relationship could be tried to expressed by the following equation:

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

where ε_f is the creep deformation at fracture, and C_1 and m_1 are material constants. The equation (1) expresses the proportionality of the strain to fracture to minimum creep strain rate multiplied by creep lifetime.

It should be noted that when the dislocation process is dominant, alloying elements in solid solution can sometimes change even the detailed dislocation mechanisms affecting creep behaviour. Two categories of single-phase alloy can be defined as.(38) : 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 Figure 1 show that normal primary curves are always observed and recorded. Thus, the alloy is reasonably expected to be class M creep behaviour regardless of the different structures resulting from TMP of the alloy.

In Figure 6, the dependence (slope of graph is more than 1) shows good correlation between time to onset of tertiary creep and creep lifetime, comparing to the correlation between minimum creep rate and creep lifetime, in Figure 7, which does not show a reliable tendency of relationship. Furthermore, it should be noted that this dependence in Figure 7 contrasted with the previous basic knowledge, which is that creep lifetime should increase with decreasing minimum creep rate. For all cases in C program, 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 a constant load creep test was performed, the stress gradually had increased as the cross section area of specimens had decreased with increasing strain. This caused the creep rate to accelerate. There could be probably 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.

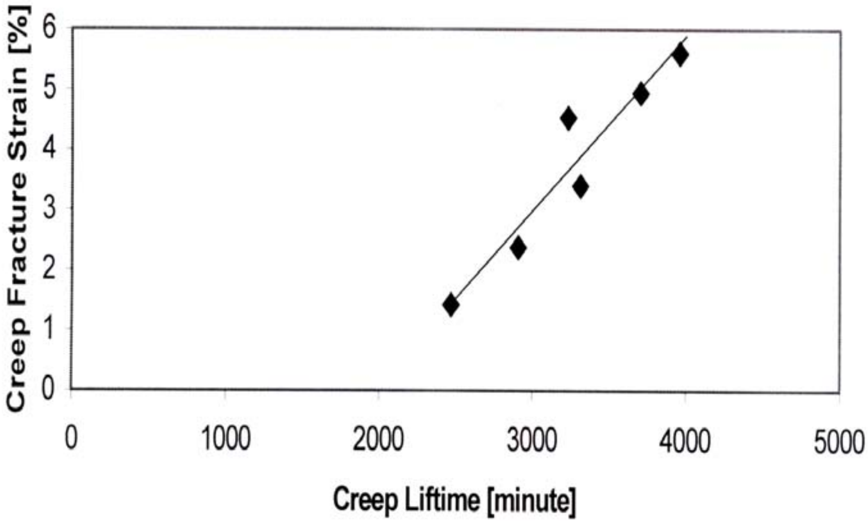

Figure 4. The relationship between creep lifetime and creep fracture strain.

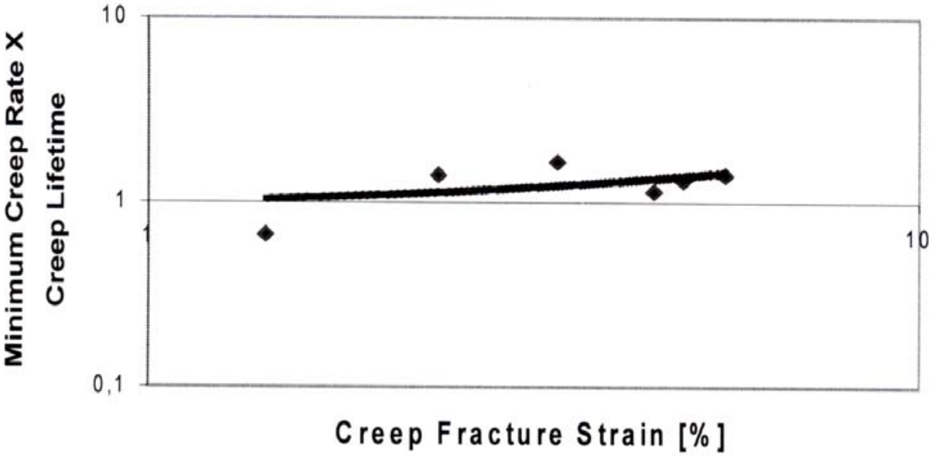

Figure 5. Dependence of ratio of minimum strain rate x time to fracture on fracture strain.

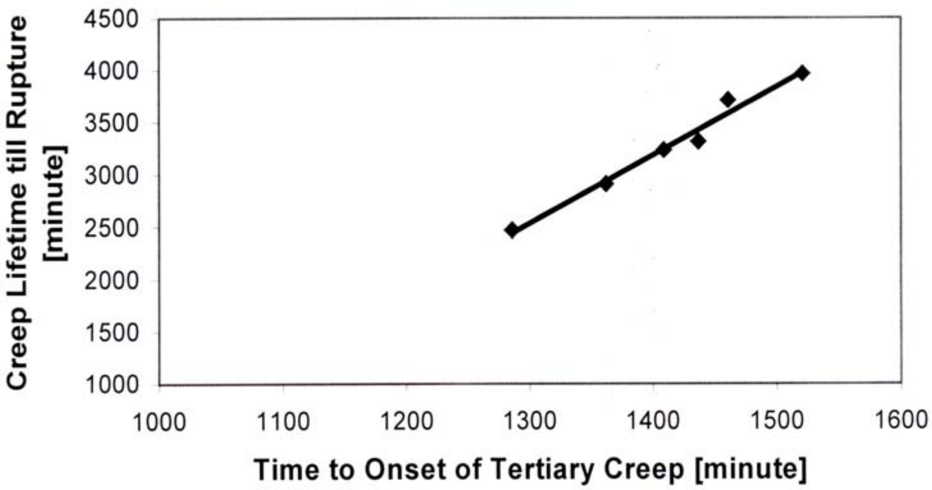

Figure 6. The relationship between time to onset of tertiary creep and creep lifetime

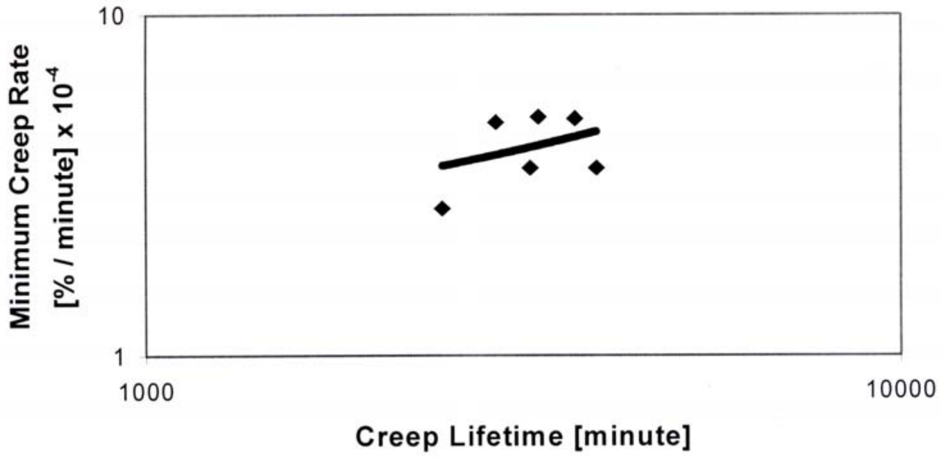

Figure 7. The relationship between creep lifetime and minimum creep rate

Microstructural Investigation of Creep Failure Specimens

The microstructural changes in longitudinal sections of crept specimens after they were subjected to different conditions of thermomechanical processing was studied by a light microscope. The results showed that creep deformation caused very negligible changes in shape and grain size.

Only areas very close to the fracture surface were observed and this is also where found individual grains with visible manifestation of deformation were found. The strains (elongation), which were below 6% in all tests, did not result in the grain elongation, regardless to the applied high creep stress. Anyhow, the largest region of deformation was near the fracture surface, Figure 8. These results of microstructure showed that they did not change from the initial microstructure.

However, in all specimens, few secondary crack openings were found along the grain boundaries located either on the specimen surface or interior specimens, as shown in Figure 8. These surface cracks were interior cracks initiated at grain boundaries, as seen in Figure 9. They were dominantly nucleated and grew along grain boundaries perpendicularly toward the applying stress (longitudinal specimen axis).

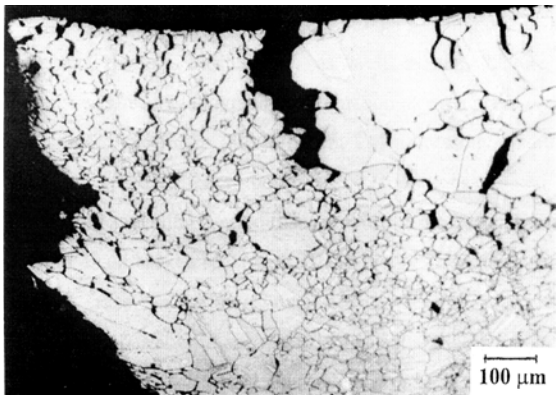

Figure 8. Crack networks close to fracture surface (left side) and free surface (up), X 100

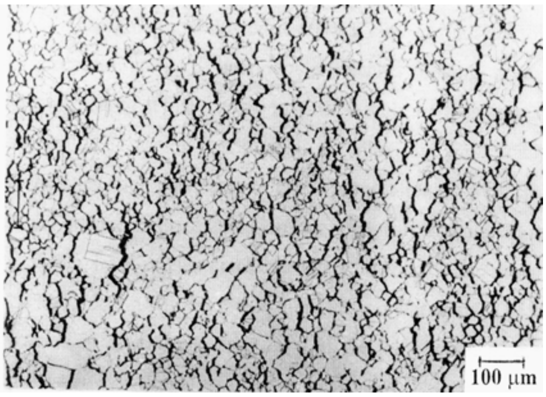

Figure 9. Crack networks in center of crept specimen, X 100

Few features of deformation in grains or slip bands were observed occurring near the fracture surface. Fracture profile lines confirm the dominant propagation of the main crack was along the grain boundaries. There were not traces of oxidation on the crack surface in the fractured specimens.

Although several processes contribute to the acceleration in the tertiary creep stage, it should be emphasized that it is usually the nucleation and growth development of microcracks, which leads to creep fracture. Under high temperature creep conditions, these cracks normally formed and grew along the grain boundaries, so that failure occurred in an intergranular mode. In general, intergranular cracking is observed in two forms. The wedge or the triple point cracks are commonly found at high-applied stress, whereas the other form of cracks by nucleation, growth and link-up of grain boundary cavities generally developed at low stresses.

One or both of crack types tends to form mainly but not exclusively on grain boundaries oriented at right angles to the tensile stress axis. Moreover, with both types of the intergranular cracks, the voids and microcracks are found to nucleate early in the creep stage life. The numbers and sizes of the cavities and number of cracks increase as creep continues. Intergranular creep fracture then occurs when the cavities and/or microcracks eventually link up to form a main crack and propagate under the applied tensile stress.

Conclusion

1. Using a light microscope for structure investigation, it was found that the microstructure of annealed cold worked specimens (C1-C6) was much more uniform than those of only two steps of hot worked specimens. The uniformity of the microstructure increased slightly as the amount of reduction increased. The most uniformed microstructure was obtained from the highest reduction of 20%. Only a few large grains were observed in the structure. The microstructure also consists of very uniform annealing twins in recrystallized grains. Comparing the grain size received form this highest amount of cold reduction it was relatively finer than other microstructures where lower cold reductions were applied. This uniform microstructure is supposed to provide very good uniform ductility properties for next step forming such as extrusion and tube drawing.

2. It could be summarized that creep lifetime and fracture strain increased as the amount of cold reduction was increased in the range of 4.8 to 15% (specimen No. C1-C5). This result was probably due to the majority effect of the strain hardening in non-recrystallized or deformed grains after partial

annealing. On the other hand, those reduction degrees could not sufficiently generate uniform recrystallized grain structure in some areas due to the fact that its partial structure had a relatively lower stored energy. However, this partial coarser structure was also resulted from insufficient annealing time to provide a uniform recrystallized structure. Therefore, the annealing of such a deformed specimen with cold reduction likely allowed locally non-uniform rapidgrain growing in some areas where lattice strain or stored energy was partially high.

3. For the highest amount of cold reduction (20%), the stored energy was stored properly and uniformly through out the samples. Then, this resulted in a more uniform and finer recrystallized grain structure, which resulted in slightly decreased creep lifetime and lower fracture strain. It should be noted that over this criterion (above 20 % cold reduction) the effect of finer recrystallized grain structure would be more pronounced. This decrease in creep strength of sample, which had previous highest cold reduction might have a higher effect of dynamic recovery and/or recrystallization during the creep process. Such cold worked microstructure, which was supposed to have very dense dislocations, should induce dynamic recrystallization during high temperature deformation resulting in a shorter creep lifetime.

4. Initiation of fracture crack was predominantly intergranular and the cracks, one or more, nucleated on the free surface of specimens. From this nucleation site, the cracks would propagate along the grain boundaries in the earlier stages of the deformation process. The growth of this critical crack size was normal to applied stress and continuously formed and grew in an intergranular manner, which is brittle. Probably, intergranular cracks developed by nucleation, growth and link-up of grain boundary cavities. After critical crack size opening its further propagation was a mixed fracture mode between intergranular and transgranular cleavage in a small area portion of fracture specimens. Several processes could contribute to the acceleration in creep rate during the tertiary stage. It was was usually the development of microcracks leading to creep fracture and combining with mechanical instability, such as the necking, which results in a localized reduction in cross-sectional area. Only at the final stage of the rupture process transgranular ductile facets appeared on the fracture surface in the type of dimple fracture morphology. The morphology differences were not observed in the fracture mechanisms of individual fracture modes for applied different material processing conditions.

5. The introduction of different amount of cold reduction after hot working had beneficial effect on microstructure development and creep behaviour of the NiMoCr solid solution alloy. Although, the highest cold reduction of 20% could not provide the longest creep lifetime but the result of recrystallized and uniformed microstructure was the most desired. To carry out the deep drawing processes, it can be recommended, that this TM process should be utilized to provide the required alloy formability. To combine improved creep properties with good formability the only modified annealing condition, namely temperature and time should be optimized to achieve the more uniform and coarser microstructure.

6. The grain morphology (size and shape) could strongly influence creep strength behaviour, especially if grain size was the primary consideration. The uniform coarser grain size favored creep strength enhancement. On the other hand, the uniform finer grain structure provides better formability in next step forming. This review will introduce examples of microstructurally induced property response to show the trend of creep deformation behaviour. However, particular emphasis was placed on creep-rupture behaviour, but tensile, low cycle fatigue (LCF), high cycle fatigue (HCF), thermomechanical fatigue (TMF) and environmental effects should be considered in the further works.

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