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MICROSTRUCTURE DEVELOPMENT BY HOT AND COLD WORKING PROCESSES IN NICKEL BASED ALLOY

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ABSTRACT

This research provides an attempt to achieve uniform grain structure by a recrystallization process from different hot and cold-working conditions followed by various annealing times in NiMoCr alloy. The different structure would result in high temperature mechanical properties such as ductility, fatigue, and creep strength. The microstructural changes results from all tested programs were investigated by an optical microscope. The results showed the uniformity of the recrystallized structure strongly relating to the amount of % reduction during hot working and period of annealing time. It was also found that the uniformity of the recrystallized structure increased with a higher amount of deformation and longer annealing times. Furthermore, the results illustrated that the recrystallized structure on the surface layer was finer than the interior of the sample. This could be due to the recrystallization occurring close to the specimen surface, where it was influenced by a much higher degree of deformation and heat lost. Such effects provided a very fine recrystallized structure. By conventional two step hot rolling, it could not reach completely the expected uniform recrystallized microstructure. This might be due to there being too low deformation during hot working and/or also from very heterogeneous initial structure. However, by additional cold rolling after hot working and then annealing, a more uniform recrystallized grain structure could be achieved as a result of higher deformation.

Keywords: nickel base alloy, thermomechanical processing, recrystallization, microstructure

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INTRODUCTION

Nickel base alloy NiMoCr, a nuclear resistant material and solid solution strengthening alloy, was invented as a container material for molten fluoride salts. The chemical composition of alloy was very similar to Hastelloy N. Hence, the NiMoCr alloy can be expected to have very similar performances like in Hastelloy N. Many studies stated that materials with high Mo and low Cr contents are generally superior to other materials in high temperature corrosion resistance in fluorine environment, specifically at 700-870°C for Hastelloy N (http://www.haynesintl.com).

Currently, it is an experimental alloy in the frame of the development ADTT (Accelerated Driven Transmutation Technology) loop for a molten salt-type nuclear reactor (Novy, *et al.* 1999). 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 the reactor of the nuclear power plant are also fundamental material requirements.

To study the mechanical properties of NiMoCr, we primarily considered grain size features, as it greatly influences strength, creep, fatigue crack initiation, and growth rate. The uniform coarse grain size increases creep strength, crack growth resistance, and ductility. In contrast, uniform fine grain size provides a high low cycle fatigue life and tensile yield strength (Jones and Jackman, 1999).

Since the primary manufacturing process of the sample was a casting and then a wrought process, sample microstructure contained mainly coarse grains and non-uniform structure. The initial microstructure of the sample was a typical grain coarsening behavior, as a result of the manufacturing processes, casting and wrought process, which provided a rapid non-steady cooling rate and non-uniform structure. However, grain size optimisation and control can be achieved by a hot working process, where plastic deformation at temperatures which are high enough for recovery and recrystallization to counteract strain hardening.

Hot working was purposely employed to transform coarse grain in ingot into refined and more uniform grain in semi-finished products. Therefore, microstructure development was controlled via the first-step in the manufacturing process. Furthermore, the result can vary dependently on the type of alloy (Semiatin and Bieler, 1999). Therefore, lots of works have been done to control and modify for recrystallization and grain growth as desired structures for numerous nickel based alloys. When comparing hot working temperature between Hastelloy N to NiMoCr, the study stated that Hastelloy N has a wide range of hot-working temperature (870-1180°C) than NiMoCr alloy (700-870°C) (http://www.haynesintl.com). As Hastelloy N has no effect on secondary precipitation from inclusions, such as, gamma and/or carbides in a very high amount to control recrystallization during the hot-working process.

Recently, there are a few studies on microstructure evolution during hot working of NiMoCr alloy ingot. Therefore, this study was an effort to develop the process conditions, which optimize uniform grain structure. To modify microstructure for better mechanical properties such as creep, different designed TMP conditions with various annealing times were utilised. The work also paid attention to the study of static recrystallization behaviour of the alloy which occurred after the annealing process.

MATERIAL AND EXPERIMENTAL PROGRAMS

The tested NiMoCr alloy has a chemical composition by wt. % as shown in Table 1. The obtained alloy was produced by the casting process and multi step press forging and annealing. The initial alloy was still not of uniform microstructure as desired after this manufacturing. Furthermore, it is required to make more reduction for final sheet production. Then various tests were modified from previous work (Novy, *et al.* 1999; Nemacek, *et al.* 1999; and Hosnedl, *et al.* 1998) and then performed to the alloy to simulate and find the optimal worked operation condition. The details of the simulating working conditions are shown in Table 2. Program A, specimens were heated to 1,200°C for 30 minutes and following by two steps hot worked for 18% and 18% of reduction then cooling or annealing at 1,100°C for 3, 5, 10, 15, 25, and 50 minutes. Program B, specimens were heated to 1,100°C for 30 minutes and following by two steps hot worked for 11.3% and 13.6% reduction then cooling or annealing at 1,100°C for 3, 5, 10, 25, and 50 minutes. In program C, after being heated to 1,200°C for 30 minutes and following by two steps hot worked for 18% and 18% of reduction following by air-cooling and then the samples were cold worked with a degree of deformation for 4.8, 6, 8, 10, 15, and 20%. Finally all tested specimens were observed and investigated by an optical microscope.

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 working and cold working conditions.

Specimen No.	**Heating Temperature before H.W.**	**% Hot Deformation**	**Annealing Time (min) at 1,100°C**	**% Cold Deformation after Air Cooling**	**Annealing at 1,130 °C for 25 min.**
A1	1,200°C/30min	18% +18%	Quenching	-	-
A2	1,200°C/30min	18% +18%	Air Cooling	-	-
A3	1,200°C/30min	18% +18%	3 min	-	-
A4	1,200°C/30min	18% +18%	5 min	-	-
A5	1,200°C/30min	18% +18%	10 min	-	-
A6	1,200°C/30min	18% +18%	15 min	-	-
A7	1,200°C/30min	18% +18%	25 min	-	-
A8	1,200°C/30min	18% +18%	50 min	-	-
B1	1,100°C/30min	11.3% +13.6%	Quenching	-	-
B2	1,100°C/30min	11.3% +13.6%	3 min	-	-
B3	1,100°C/30min	11.3% +13.6%	5 min	-	-
B4	1,100°C/30min	11.3% +13.6%	10 min	-	-
B5	1,100°C/30min	11.3% +13.6%	25 min	-	-
B6	1,100°C/30min	11.3% +13.6%	50 min	-	-
B7	1,100°C/30min	11.3% +13.6%	-	6%	Yes
C1	1,200°C/30min	18% +18%	-	4.8%	Yes
C2	1,200°C/30min	18% +18%	-	6%	Yes
C3	1,200°C/30min	18% +18%	-	8%	Yes
C4	1,200°C/30min	18% +18%	-	10%	Yes
C5	1,200°C/30min	18% +18%	-	15%	Yes
C6	1,200°C/30min	18% +18%	-	20%	Yes

RESULTS AND DISCUSSION

Microstructureal Analysis of Slab by Optical Microscope

In the obtained initial microstructure of the alloy, resulted from casting and ingot multi steps hot forging with repeated reheating, heterogeneity was found in the form of bimodal grain size structure, as shown in Figure 1. The calculated grain size mean diameter was $d_m \approx 0.09$ mm. The presence of such a heterogeneous structure of broken down ingot after casting could be a result either of high stability of the primary cast grain structure or due to non-uniform deformation of the ingot. The recrystallization of process could not be generated uniformly through out of ingot cross section. By analyzing the structure, in the sections cut parallel to the longitudinal axis of the slab and in a cross section of slab, the chain of very recrystallized grains were found along the grain boundaries of the coarse grains. This appearance can be used as evidence of a dynamic recrystallization process, which were originated at grain boundaries, see Figure 2. These fine grains might further nucleate preferentially at grain boundaries by either static recrystallization in periods of ingot reheating or they were results of the dynamic recrystallization process, which ran during multi-step hot pressing operation (Wangyao, *et al.* 2001).

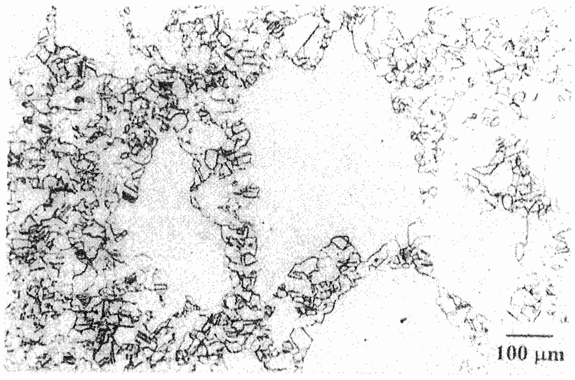

Figure 1 Initial ingot breakdown microstructure.

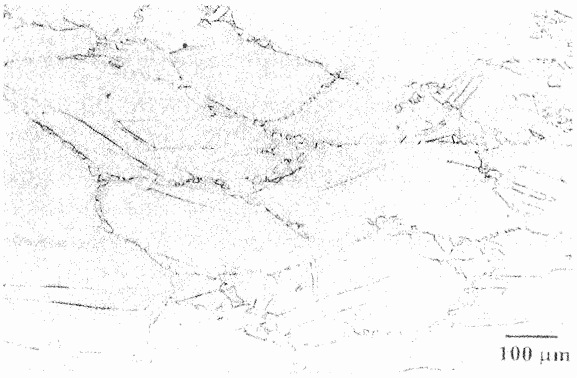

Figure 2 The fine dynamic recrystallized grains around coarser primary grains.

Structure Analysis of the Alloy after Hot Working Processing

The structure development for both designed and realized thermomechanical processing of NiMoCr alloy has been analyzed successively according to the program A and B (Table 2). In both designed programs, where a different deformation temperature and degree of deformation were selected, have been expected that these parameters would promote the recrystallization process in the alloy. To fix and evaluate that remained deformation effect on the further structural evolution then the fast cooling was performed before recrystallization annealing. It was observed, for both programs in primary schedules (specimen A1, A2 and B1) after hot working followed by immediate quenching or air-cooling, that the structures still remained in heterogeneous form according to the grain size and morphology. The structure consists of a quite large number of coarse primary grains in the central area of rolled down bands, and of the very fine dynamically recrystallized grain structure with annealing twins in their interior, Figures 3 and 4.

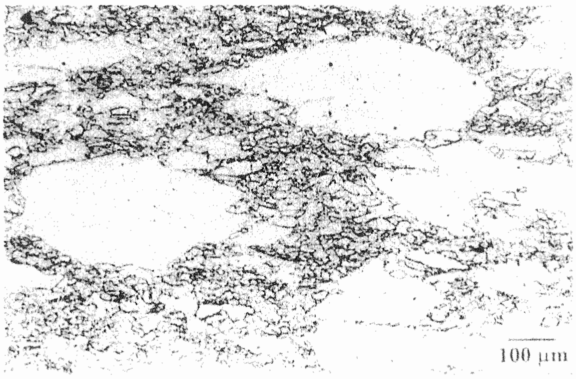

Figure 3 Micrograph of quenched microstructure, A1.

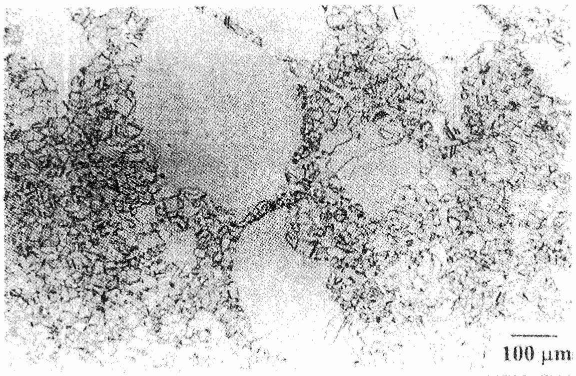

Figure 4 Micrograph of quenched microstructure, B1.

It should be noted that the strain hardening resulting from hot rolling deformation should be reduced mainly by dynamic recovery. Dynamic recrystallization provided additional softening, possible partial grain refinement and an increase in hot ductility. Additionally, it was possible to recognize that large primary grains, were slightly aligned parallel to the rolling direction, as illustrated in Figure 3. However, the direction of grain alignment in the microstructure was more pronounced in bands rolled down according to the condition of program A applied. This implies that the microstructures in program A were distorted or deformed in a higher amount than those of program B . However, this fact can provide evidence about non-uniform displacement of deformation within band cross section and on the absence of sufficient deformation within the central part of the rolled bands to break down the coarse grains regardless of program A or B.

When the annealing process was done immediately after hot working for both rolling schedules (specimen A3-A8 and B2-B6), the grain size heterogeneity in structure was decreased as annealing time increased. The recrystallization structure advancement due to the annealing process resulted in a more uniform grain structure of the alloy, as can be observed in Figures 4 and 5. Comparing the results on structure evolution according to both programs, it was concluded that the resulted recrystallized structure in program A were more uniform, as to consider the grain size and morphology, than those received in program B.

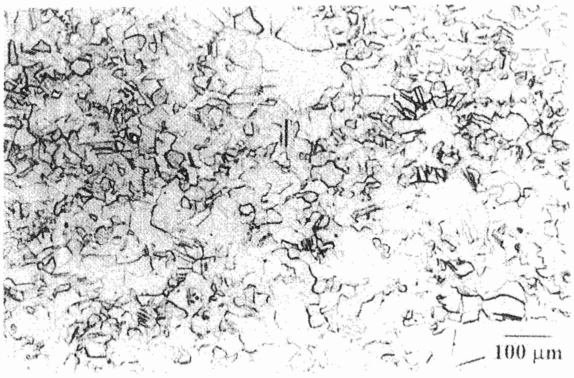

Figure 5 Micrograph of annealed microstructure, A8.

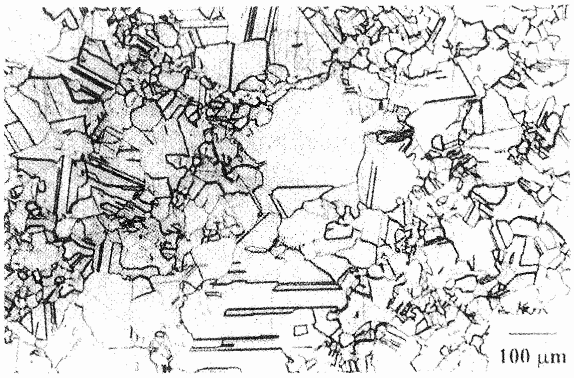

Figure 6 Micrograph of annealed microstructure, B6.

Considering this fact, it might imply that the final grain size depends greatly on the degree of introduced deformation strains (in program B), lesser number of the nuclei would be formed per unit volume and, moreover, the incubation time for nuclei formation was longer, therefore, driving force for nucleation period was lower. In consequence, the smaller number of nuclei led to coarser final recrystallized grain structures as in program B comparing to the same annealing time of program A, Tables 3 and 4. However, prolongation of the annealing time, no matter the program, was successful to break down the stable coarse primary grains as well. The resulted grain structure morphology within these coarse grains was different comparing to morphology of fine grains but recrystallization of these grains proceeded as in documented in Figure 5 (program A) and Figure 6 (program B). However, the recrystallization was more advanced when higher deformation was applied in program A.

Table 3 Grain size measurement systems in program A.

Specimen No.	**A1**	**A2**	**A3**	**A4**	**A5**	**A6**	**A7**	**A8**
Average grains/ mm^2	144	141	157	168	187	201	184	136
Average grain diameter	0.086	0.087	0.083	0.081	0.077	0.074	0.078	0.088
ASTM (approximately)	≈4	≈4	≈4	≈4.5	≈4.5	≈5	≈4.5	≈4

Table 4 Grain size measurement systems in program B.

Specimen No.	**B1**	**B2**	**B3**	**B4**	**B5**	**B6**
Average grain/ mm^2	131	144	149	153	160	108
Average grain diameter	0.089	0.086	0.085	0.084	0.083	0.099
ASTM (approximately)	≈4	≈4	≈4	≈4	≈4	≈4

During the process of recovery and recrystallization, the point defects and dislocations should gradually approach the much lower concentrations in annealing Wangyao, *et al.* (2001), and considerable rearrangement occurred with the result that the defects should disappear, energy was released in the form of heat. Initially, recrystallization involved the growth of the new crystals into distorted grains but later for longer annealing times, there usually was grain growth into recrystallized grains or bigger recrystallized grains ate the smaller ones. The larger recrystallized grains absorbed continually smaller ones to reduce surface energy until the stable grain size was reached. This behavior usually occurred at high temperature and larger grain size as an initial condition.

As heating at the recrystallization temperature was prolonged, regardless of the annealing temperature, grains became gradually larger, some being absorbed by others. This is known as crystal growth and it manifested the secondary recrystallization, which had already started rapidly in the range of 10-15 minutes annealing time. The driving force for this recrystallization step was a decrease in surface energy not due to stored deformation energy (Courtney, 1990; and Honeycomb, 1984). Hence, uniform grain growth structure, which needed a much longer annealing period, could not be obtained in alloy by these applied short periods of annealing time. Respecting that resulted microstructure from obvious two steps hot rolling and the selected annealing time could not satisfy the desired completed uniform microstructure requirement. The recrystallization process effectiveness of the alloy at given thermomechanical conditions, was performed with the aim of grain size and grain density per area. The relationships between grain structure and annealing time in program A and B are shown in Figures 7 and 8, and in Tables 3 and 4.

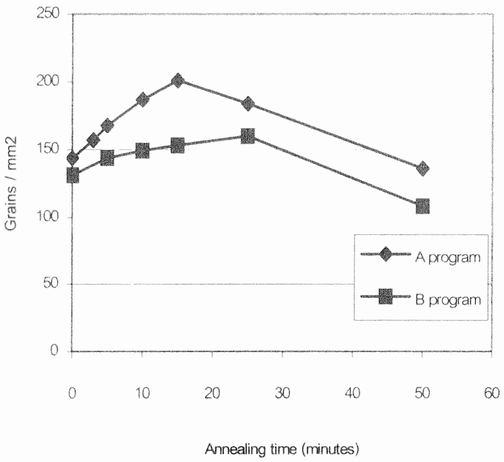

Figure 7 The relationship between number of grians/mm^2 and annealing time of program A and B.

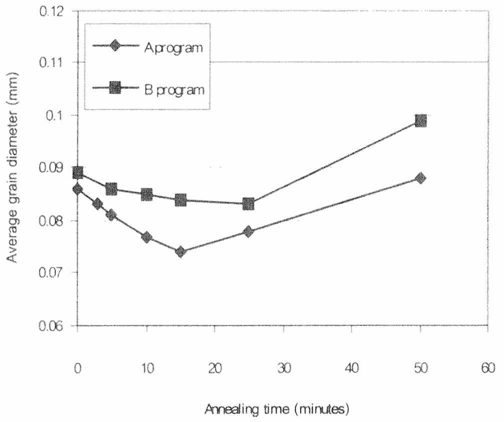

Figure 8 The relationship between grain size and annealing time of Program A and B.

Structure Analysis after Hot and Cold Working Processing

The additional cold working followed by the hot working process was an experimental attempt to modify and improve the structure of NiMoCr alloy with the aim to enhance the secondary recrystallization process. The increase in total amount of deformation in program A by introducing post annealing cold reduction of various degrees was expected to support the additional grain growth and homogeneous structure in grain size. Structure investigation was to distinguish the grain evolution changes resulting during successive recrystallization process of specimens with different deformation imposed on them.

It was found that the alloy structure of all recrystallized specimens where cold deformation was introduced, even for % of cold reduction lower than 10%, had already quite very uniform microstructure, as can be seen from structure documentation (specimen C1-C6 in program C). It might be the additional effect of deformation, during hot rolling and then annealing, provided the fine grain structure before next step cold rolling and annealing. The additional cold working followed by annealing provided a much more uniform and finer structure characteristic than that received in only two steps hot worked specimens (A1-A8 and B1-B6) as illustrated in Figures 9 and 10 (C1 and C6).

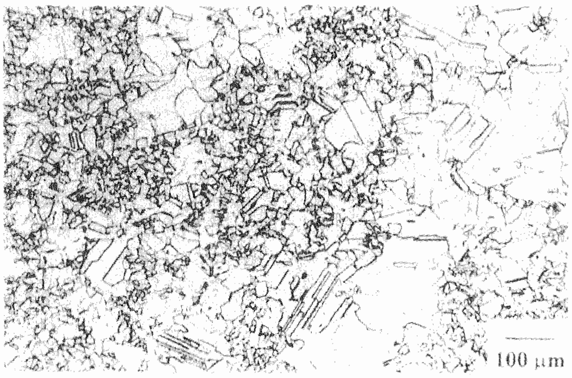

Figure 9 Micrograph of annealed 4.8% cold reduction, C1.

Figure 10 Micrograph of annealed 20% cold reduction, C6

The results of grain size measurement corresponding to structures received from program C are shown in Table 5. According to the previous work (Tillack, *et al.* 1989), it stated that the amount of any previous cold work has critical influence on the ductility of nickel and nickel alloys after annealing. If only a small amount of cold work is applied, for example at about 10% of reduction in rolling pass, full ductility for deep drawing and spinning cannot be restored by annealing because of excessive grain growth due to critical strain, even though the hardness is reduced to that of soft materials. A minimum, of approximately 20% cold working, is required to guarantee the maximum ductility and softness of the alloy after the annealing process.

Table 5 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 (approximately)	≈ 5	≈ 5	≈ 5	≈ 5 -6	≈ 5 -6	≈ 6

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

Summarizing the effect of additional cold deformation and comparing with the structure characteristic received in program A the finer structure was received if program C has been realized. This might be due to the respected applied cold reductions, which is no evidence of an onset of a secondary stage of recrystallization. The manifested structure uniformity resulted from a received procedure condition according to program C increased slightly as the amount of specimen's reduction increased from (4.8%-20%). The relationships among the average grain size and number of grain per area shown in Figures 11 and 12. The most uniformed grain structure was received from a highest applied reduction of 20%. Only few large grains were observed in the structure, which could imply that secondary recrystallization could be suppressed at these processing conditions. Comparing the grain

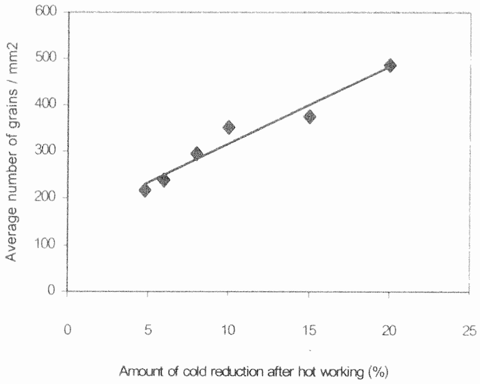

Figure 11 The relationship between average grain size and amount of cold deformation in Program C.

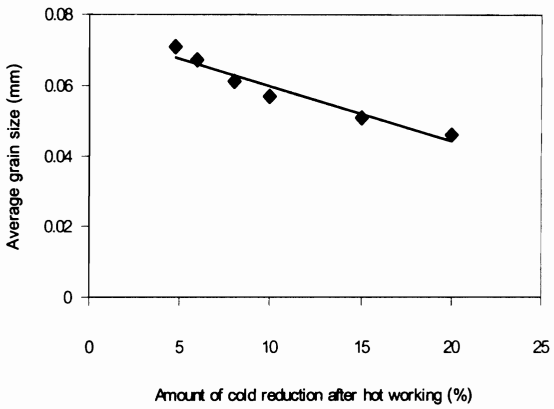

Figure 12 The relationship between average number of grains per mm^2 and amount of cold deformation in program C.

size result (Table 5) of maximum cold reduction, the structure was relatively much finer to other structures where lower deformation reductions were introduced during cold rolling. The resulted uniform structure is expected to provide good and uniform ductility property for the next step forming of alloy such as sheet cold rolling, extrusion and tube drawing, the processing expected to be used for finalizing of alloy's products.

It must be noted that immediately followed by hot-working, static recrystallization had a lower driving force (the strain energy) than that of the cold worked structure. Therefore, the hot worked structure may degrade during a slow heat up in annealing so that it gave rise to greater completion times and coarser grain sizes. The reformed substructure, static recrystallization might take place after dynamic recrystallization (DRX) mainly by continued growth of DRX nuclei formed just before deformation ended. Such metadynamic recrystallization (MRX), which had no incubation time, usually produced a larger grain structure than the original DRX ones (Sakai, 1995). Therefore, all grain sizes obtained from program C were finer and more uniform than those obtained from program A and B.

As results show in program C, the uniformity in grain size of microstructure increased with a higher amount of introduced cold deformation. This was due to an increased additional applied deformation generating a higher amount of lattice strain energy or should store deformation energy in microstructure for active site nucleation such as slip line interactions, deformation twin interactions or residua of sub-grains and in areas close to grain boundaries especially at the triple points at grain boundaries, which could be proposed sites for new grain nucleation (Wangyao, *et al.* 2000; and Wangyao, *et al.* 2002). Foremost, the higher amount of cold reduction provides more uniform and finer final microstructure of alloy, which could be received by the introduction of additional cold deformation. The positive of program C was that the very fine recrystallized microstructure at surface layers was not observed like in A and B programs. This might be due to the amount of additional cold deformation during the rolling process displaced uniformly enough stored energy in the alloy samples resulting in more uniform recrystallization process could occur through out the entire specimens.

It is well known that the grain morphology (size and shape) strongly influence tensile strength, creep strength and fatigue crack initiation and its growth rate. Specifically, the grain size is the primary consideration, where the uniform coarser grain size favors creep strength enhancement. On the other hand, the uniform finer grain structure provide much better in low cycle fatigue life and tensile yield strength of solid solution strengthened nickel base alloys (Furrur, *et al.* 1999; Torster, *et al.* 1997; Livesey, *et al.* 1985; Shen, *et al.* 1995; and Heilmaier, *et al.* 1997). This review would introduce examples of structurally induced property response to show general trends. However, there can be exceptions to these trends, particular emphasized in this present research work.

In all programs, a feature of microstructure in the recrystallized state was the presence of annealing twins in the grain structure. The twin markings may run completely or partly across crystals, as shown in many figures. Straight-edged twins were very commonly observed within many grains of recrystallized face-centered cubic alloy. In fact, they were a sure indication of recrystallization. In general, in face-centered cubic metals or alloys, twins did not usually appear until after recrystallization, which are so-called annealing twins. At areas close to the specimen surface, where the amount of deformation during forming was much higher than interior structure provided the very fine and uniform recrystallized grains.

However, next step cold working operations, as we already know that the coarse-grain metallic materials are not suitable for most next step cold working operations. Moreover, coarse grain in the high nickel alloys cannot be refined by only thermal treatments. Therefore, it can be removed only by cold working efficiently to effect recrystallization to a smaller grain size during a subsequent annealing treatment. Maximum workability is obtained with material that has been annealed without any grain growth occurring. The average grain diameter should not exceed 0.064 mm. (ASTM No. 5) (Tillack, *et al.* 1989). This gives the best combination of ductility to allow high deformation, strength to withstand the action of tools, and surface quality to facilitate polishing.

CONCLUSIONS

1. In both program A and B (specimen A1, A2, and B1) after hot working and then immediately quenching or air-cooling, the microstructure was still heterogeneous as well with large number of internal annealing twins and coarse grains in the middle of the band samples. However, it was possible to see clearly that the deformed grain morphology is aligned parallel to the rolling direction.
2. When the annealing process was done right after hot working, the heterogeneity in microstructure was decreased as an increase of annealing time.
3. The recrystallized structure with a longer annealing time resulted in more uniform grain structure. The obtained microstructures of program A were more uniform than those of in program B.
4. The final grain size depended greatly on the degree of deformation during hot working. The greater of the degree of deformation, the finer the recrystallized grain size was obtained.
5. The microstructure of annealed cold work specimens in program C was much more uniform than those of only two steps hot worked specimens in program A and B following the annealing process.
6. The uniformity of microstructure according to program C, increased slightly as the amount of reduction increased. The most uniform microstructure was obtained from the highest cold reduction of 20%. The obtained microstructure is supposed to provide a much better uniform ductility property for the next step forming such as extrusion and tube drawing comparing to received microstructure according to A and B programs.

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