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TEM ANALYSIS OF DISLOCATION SUBSTRUCTURE DEVELOPMENT AFTER THERMOMECHANICAL PROCESSING IN NICKEL BASED ALLOY

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ABSTRACT

Paper describes the effect of different thermomechanical processing (TMP) conditions on the evolution of the recrystallized substructure of NiMoCr alloy. The hot conditions and various heat treatment parameters were to affect the recrystallization process of the alloy. The changes in microstructure as results from tested programs were investigated by a transmission electron microscope. The progress in the recrystallization process in dependence of the applied strain and temperature parameters was investigated through dislocation structure development in thin foils. The results showed that the uniformity and the percentage amount of recrystallized area (low density of dislocation in substructure) increases when a longer annealing time and/or higher amount of deformation during TMP were employed.

Keywords: TEM Analysis, Nickel based alloy, Dislocation substructure, and Thermomechanical Processing

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INTRODUCTION

NiMoCr alloy is a relatively new solid solution strengthened nickel based alloy with attractive properties for its specific application for use in a nuclear reactor. Development and use of the alloy requires an understanding of its metallurgy in terms of microstructure relating to its properties. The alloy microstructure can be developed and/or modified to reach optimum mechanical properties by many technological methods such as thermomechanical processing (TMP) or hot working. In order to modify the microstructure for better mechanical properties such as creep, LCF and TMF, differently designed hot working processes have been done together, utilized with various annealing times.

Generally, in the hot working process, dynamic, metadynamic, and static recrystallization can occur during the process. Dynamic recrystallization occurs during the actual deformation. Whereas under metadynamic recrystallization conditions, the partially recrystallized grain structure, immediately observed after deformation, transforms to more fully recrystallized structure by growth of recrystallization nuclei, which formed during the deformation (Jones and Jackman, 1999). In case of static recrystallization, it occurs in reheating or during the annealing process when deformation proceeded. The attention was paid to study the effect of static recrystallization of the alloy on the structure evolution applying different annealing times. The transmission electron microscopy (TEM) has been used to detect the progress in development of microstructure characteristics.

MATERIALS AND EXPERIMENTAL PROGRAMS

To investigate the effect of hot deformation and annealing time parameters at 1,100°C involved after TMP conditions of the alloy (See details in Tables 1 and 2) on the development of the recrystallized structure, the TEM investigation of thin foils was used at accelerating voltages of 200 kV. The thin foils for TEM were prepared by cutting along the longitudinal axis of worked specimens to make plate-like discs with a thickness of 0.5 mm. Subsequently, the specimens were ground to a thickness of about 0.1 mm. Then thin foils were prepared by electropolishing at a potential of 25-30 V in solution of $HClO_4$ acid (10% by volume) in methanol. The temperature of the solution was less than -30°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 working conditions.

Specimen No.	Heating Temperature before H.W.	% Hot Deformation	Annealing Time (min) at 1,100°C
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

Table 2-Continued

Table 2 Details of hot working conditions.

Specimen No.	Heating Temperature before H.W.	% Hot Deformation	Annealing Time (min) at 1,100°C
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

RESULTS AND DISCUSSION

An extensive TEM analysis was carried out to study the evolution of the recrystallization process through dislocation structure development after the different parameters of thermomechanical processing (TMP) and various annealing periods. From the received TEM results, it can be concluded that the effect of TMP parameters to affect the progress of recrystallization, can be expressed by different dislocation substructure characteristics. These TEM results can also support the results received from light microscopy and SEM (ECC mode) analysis of specimens from the previous work of Wangyao, *et al.* 2003.

In quenched and air-cooled specimens for both programs, as expected, the very dense and homogeneous dislocation tangles persisted in the interior of the grains and represented the deformed state, as shown in Figures 1 and 2. However, it is possible to notice that the deformed substructure resulting from a higher amount of reduction during hot working (Figure 1) caused slightly higher dislocation density than that subjected to the lower total hot reduction (Figure 2). These results of very dense dislocation structure are the features of plastic deformation, which were also confirmed by results, received by SEM and image analysis of previous work of Wangyao, *et al.* (2001), where fully complex bend contours are the evidence of retrained structure in specimens.

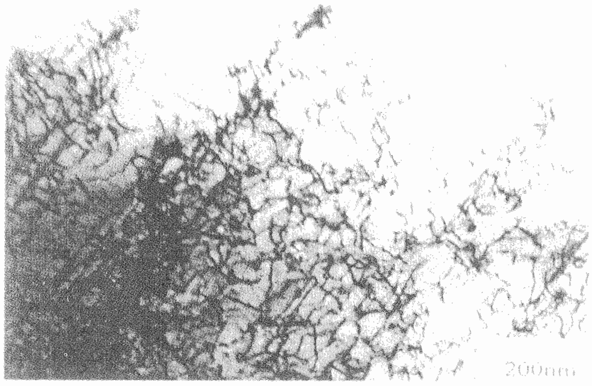

Figure 1 The very dense and homogeneous dislocation tangles in deformed grain, A1.

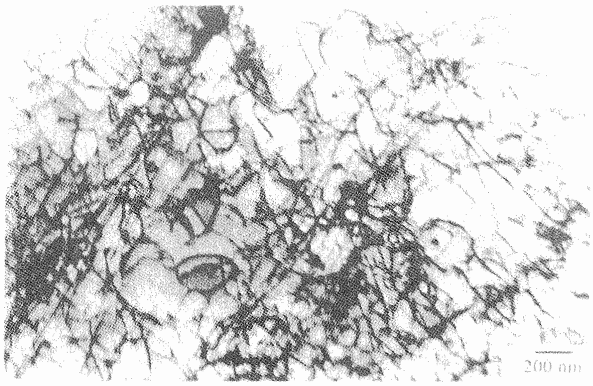

Figure 2 The very dense and homogeneous dislocation tangles in deformed grain, B2.

Not regularly, the dislocation-particle interactions were noticed in some grains within slip bands in the matrix, in which interactions occurred during hot deformation, Figure 3. In this research work the TEM analysis could not confirm clearly the evidence of dynamic recrystallization but it ought to be due to the local character of the TEM method. However, the characteristics of the evolution of new grain structure under conditions of dynamic recrystallization together with the associated development of dislocation substructures might be classified generally into three categories, according to the previous work of Sakai T. : (1) dynamic recrystallization (DRX) nuclei appearance, (2) growing DRX grains containing dislocation gradient, and (3) critically work hardened large DRX grains with fairly homogeneous dislocation substructure and more uniformly developed substructure.

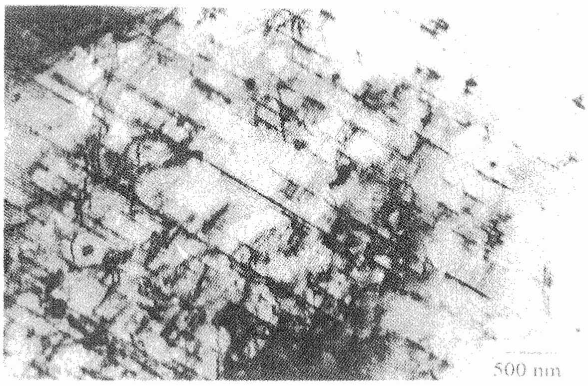

Figure 3 Dislocations in the slip band of the deformed substructure.

The TEM observation of selected static recrystallized specimens after the annealing process was supposed to confirm the progress in the recrystallization process and to console the results with those received from SEM analysis of the recrystallized structure, which shows clearly smooth ECC contours in the grain interior. After short annealing times, the change in the dislocation substructure, which corresponded to a recrystallized structure, had occurred for both different treatments. However, the dislocation structure heterogeneity was still frequently found in the interior of the grain structure. For shorter times of annealing, the lower dislocation density was observed as documented in Figure 4. The dislocation segments and pile ups are arranged on acting slip systems. Subgrain boundaries, due to the recovery process, had formed during the annealing time in 5 minutes, Figure 5. However, the dislocation tangles interactions within grains were still found for a shorter time of annealing in the deformed area, Figure 6. The non-uniformity of the recrystallized process, as manifested in the structure by electron channeling contrast mode by the SEM analysis method for short annealing periods has been also proved by TEM. The dislocation segments in recrystallized grain have been found also for a longer period of annealing as documented in Figure 7. The presence of small precipitates distributed non-uniformly in the structure caused the interaction with dislocation structures persisting in recrystallized grains for short annealed conditions. The dislocation substructures vary from grain to grain, and are distributed more or less heterogeneously.

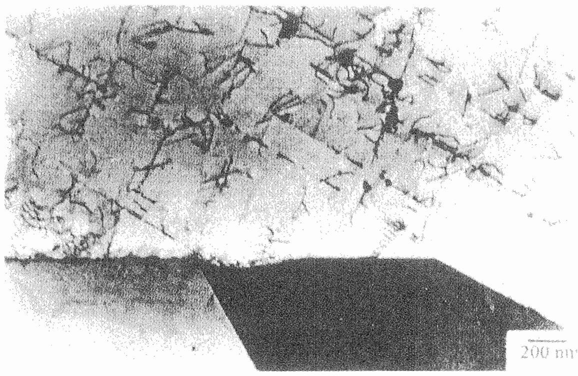

Figure 4 Lower dislocation density in recrystallized grain after 5 minutes annealing.

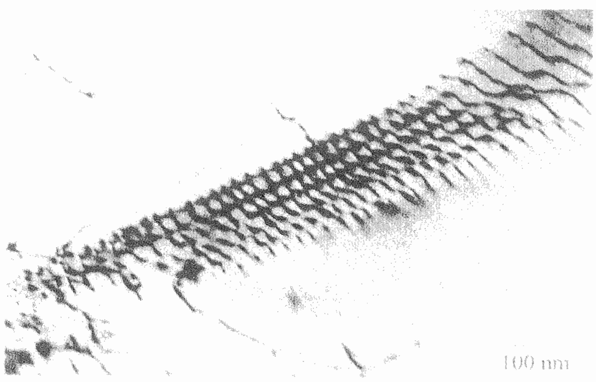

Figure 5 Subgrain boundary after annealing for 5 minutes.

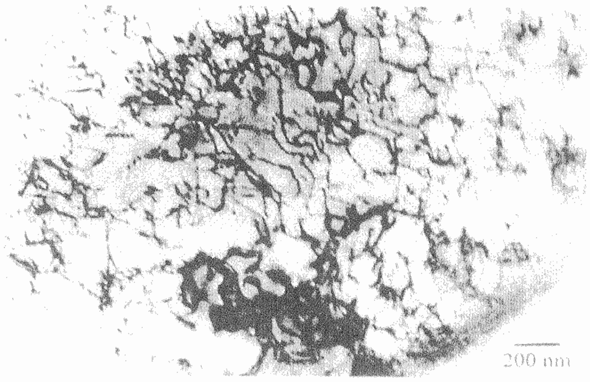

Figure 6 Dislocation substructures after annealing for 5 minutes.

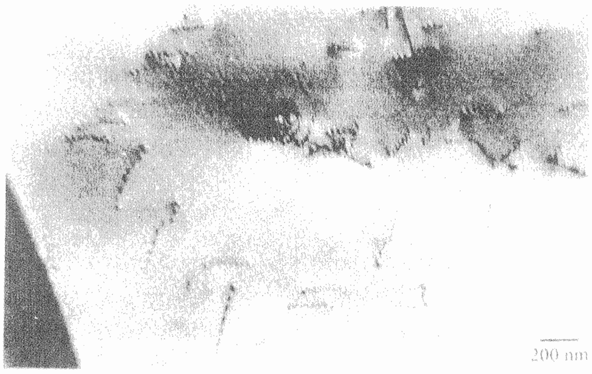

Figure 7 Lower density of dislocation structure in recrystallized grain after 10 minutes.

It was observed that introduction of longer annealing times over 10 minutes should reasonably provide more uniform recrystallized structures with only residual dislocations in recrystallized grains. Any dislocation tangle has been observed in the structure. Frequently, only the dislocation clusters or dislocations pile up at precipitates were still found even after the longest annealing time (50 minutes), as it can be seen in Figures 8 and 9. The grain structure with high angle boundaries proves the progress in recrystallization for both programs. The characteristic recrystallized structure corresponding to 10 minutes annealing time is documented in Figure 10. The final recrystallized structure corresponding to the longest annealing time of 50 minutes is presented in Figure 11. Despite of the long time of annealing, in recrystallized grains the stable dislocation arrangements of pile-ups still persist close to grain boundaries. Anyhow, the character of high angle grain boundaries is fully recrystallized.

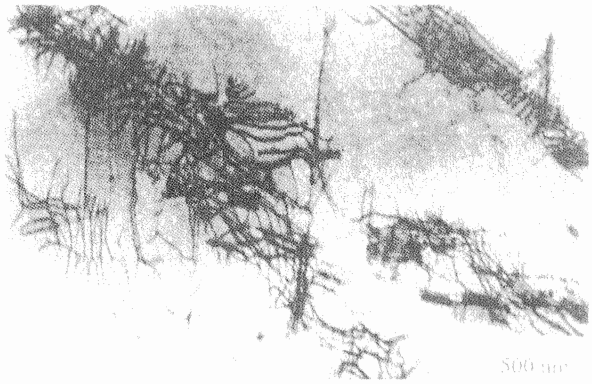

Figure 8 Dislocation clusters after 50 minutes annealing.

Figure 9 Dislocation pile-ups at precipitate particle after 50 minutes annealing.

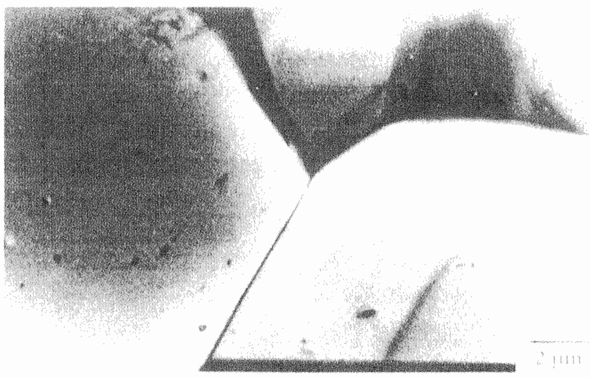

Figure 10 The high angle grain boundaries after 10 minutes annealing.

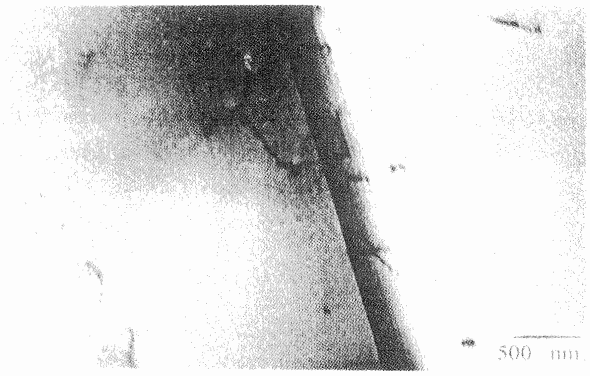

Figure 11 Final substructure after 50 minutes annealing.

To follow the results of all programs, it was found that after annealing within the frame time of experiments, all observed annealed microstructures were almost completely recrystallized (which were close to 100% static recrystallization), see previous work of Wangyao, *et al.* (2002) . However, such nearly complete recrystallization behavior should be explained by the heterogeneous nature of previous dynamic substructures according to (Sakai, 1995). When hot deformation was completed, the dynamic recrystallization (DRX) nuclei might continue to grow leading to metadynamic recrystallization (MDRX) in their localities. The growing DRX grains should contain few dislocations near their boundaries, and so classical nucleation was not possible to occur within them. Accordingly, they could only soften by metadynamic recovery (MDRV) in their interiors.

Finally, the full work hardened DRX grains underwent static recovery (SRV) and nucleation under the annealing process leading to classical static recrystallization (SRX). However, the incomplete softening could be caused by the stable existence of growing DRX grains having many-sided irregular shapes with high density dislocations. Therefore, with further annealing of such mixed structure (i.e., MDRX or SRX new grains and MDRV grains containing the moderate dislocation densities), the growth rate of new recrystallized grains should decrease because of a small difference in dislocation densities across their boundaries, (Xu and Sakai, 1991).

Furthermore, the other reasons of incomplete static recrystallization process, even, after the longest annealing time for 50 minutes, could also be possibly due to the following explanations. First was due to the precipitate particles, which blocked the movement of statically recrystallized growth. The long term annealing could not annihilate completely the interactions between dislocations and precipitates. Second was the effect of a high diffusion co-efficient of alloying elements in the matrix causing a low diffusion rate, which caused dislocations moving more difficultly and slowly. Therefore, they could not annihilate each other properly. Thus, the recrystallization process needs more time to be completed.

It should be also noted that when the alloy band specimen was deformed, most of the energy was used in changing the shape and in generating adiabatic heat. However, a small amount of energy still remained and stored in the metal. The stored energy was mainly in the form of elastic energy in the strain fields of dislocation arrangement and point defects, which had the concentrations order of magnitude higher than in annealed alloy. The distribution of dislocations was, in addition, quite different to that in the annealed state, i.e., there were pile-ups against barriers or obstacles, dislocation tangles and diffuse sub-boundary arrays.

CONCLUSIONS

TEM investigation confirmed the presence of not completed recrystallized grains, yet the volume portion is less with the prolonged annealing time. To obtain identical recrystallization behavior from the only hot working in program A and B is quite difficult. The obtained results on substructure would suggest to modify the next step processing in a way of higher deformations should be introduced during hot working or more multi-steps of successive individual hot passes where particularly lower reductions will be carried out, and/or the additional cold working would be applied after the hot working process.

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