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EFFECT OF FORMING PARAMETERS ON HOT WORKING PROCESS IN NICKEL BASE ALLOY

Panyawat WANGYAO [1*], Tibor KVAČKAJ [2],
Jozef ZRNÍK [3], Vladimir VRCHOVINSKÝ [3] and Zbyšek NOVÝ [4]

[1]Metallurgy and Materials Science Research Institute, Chulalongkorn University, Thailand

[2]Department of Metal forming, Faculty of Metallurgy, Technical University,
Košice, Slovakia

[3]Department of Materials Science, Faculty of Metallurgy, Technical University,
Košice, Slovakia

[4]Complete Technological Service-Forming & Heat Treatment Ltd., Plzeň, Czech Rep.

ABSTRACT

NiMoCr alloy, one of the nickel based superalloys, have been frequently developed for use as a material part application in a nuclear reactor where excellent moderately high temperature strength, good resistance to creep and fatigue, good oxidation resistance to molten fluoride salts as well as good formability are required. Thermomechanical processing (TMP) plays a vital role in controlling the microstructure and properties in the alloy. By controlling the quality of the input material (billet grain size and chemistry) and the TMP process (temperature, strain, strain rate etc.), the desired properties can be reached. However, there are very few reports in the previous literature exhibit the most suitable and practicable TMP conditions for the alloy. Therefore, this research work attempts to study and search for the processing parameters such as preheating and reheating temperatures, % reduction during each step forming and number of forming steps influencing the overall forming process for use in the future. Accordingly, this TMP conditions study was also conducted to investigate several combinations of many parameters with the intent of obtaining the most proper processing techniques leading to the optimized microstructure.

Keywords: Thermomechanical processing, Forming parameters, and nickel based alloy

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* To whom correspondence should be addressed:
E-mail: panyawat@hotmail.com Tel: (662) 218 4233 Fax : (662) 611 7586

INTRODUCTION

The initial microstructure of the materials from the casting process, have a typical non-uniform grain structure. However, grain size optimization and control can be achieved by the hot working process, where plastic deforms at temperatures which are high enough for recovery and recrystallization to counteract strain hardening (Jones, *et al.* 1999; and Howson, *et al.* 1989). The main goal for hot working an ingot into a semi-finished product is to refine and uniform the cast ingot structure (Symonds, 1974). The hot working process by which microstructure development is controlled is strongly dependent on the type of the alloy (Jones and Jackman, 1999). Therefore, lots of works have been done to control recrystallization in order to modify grain growth as desired structures for numerous nickel based alloys. However, one of these alloys, Hastelloy N, which was compared to the NiMoCr alloy, has a wide range of hot working temperatures between 870-1180°C (Symonds, 1974). This is because there is no need to consider the effect of secondary phase precipitate such as gamma prime and/or enough carbide, which controlled the recrystallization process during hot working.

It is well known that besides the purpose to break down the casting ingot structure and for shape reduction, thermomechanical processing (TMP) such as hot working can also improve the size and morphology of the grain structure (Howson, *et al.* 1984; Symond, 1974; Jackmon, 1984; and Couts, 1972). TMP operation at 815-1100°C can also promote favorable carbide distribution in alloys. The desired uniform coarsening recrystallized microstructure does not only provide high creep strength and crack growth resistance but also promotes resistance to thermal fatigue, which are needed for the alloy to sustain the complex stress conditions. However, control of the grain size is vital and difficult. A balance must be carefully considered to avoid excessively fine grains, which could be harmful to the tensile, fatigue and yield properties (Decker and Sims, 1972).

Usually, the forming process can be classified into hot and cold working operations. Deformation during the hot working process takes place under conditions of temperature, and strain rate control can generate the recovery process occurring simultaneously in metals. The deformed (distorted) grain structure and strain hardening, which occurs during operation, is rapidly eliminated by the recrystallization process at high temperature because of the formation of new strain-free grains. A very high degree of deformation is possible to carry out in this operation by the recovery process (Tamura, 1989).

Since the flow stress decreases with increasing temperature, the energy required for deformation is generally much less for hot working than cold working. In the hot working process, strain hardening is eliminated by the effect of temperature unlike in the cold working process where flow stress increases with percentage of deformation (Courtney, 1990). Therefore, the total degree of deformation without causing fracture is less for cold working than for hot working, unless the effect of cold working is relieved by the annealing process before the next step cold working to reach the same or higher total amount of deformation (reduction).

For most commercial alloys, the hot working operation must be performed in the range of high temperatures in order to produce a rapid enough rate of recrystallization. However, it is important to consider that the temperature of the work piece in metalworking also depends on 1) the initial temperature of the tools and metal, 2) heat generation due to plastic deformation 3) heat generated by friction at the tool / metal interface, and 4) heat transfer between the deformed metal and the tools as well as the surrounding environment (Courtney, 1990).

The minimum limit temperature for hot working of metals is the lowest temperature at which the rate of dynamic recrystallization is rapid enough to reduce the strain-hardening effect. The minimum operating temperature depends on the amount of deformation and the time, which the metal is at the temperature. The limit (recrystallization) temperature will decrease for the larger deformation. The upper limit for hot working temperatures is considered by the temperature either melting or excessive oxidation occurs. Generally, the maximum operating temperature is limited to 50°C below the melting point (Dieter, 1988).

Most hot working operations are done in a number of multi steps. The working temperatures for the intermediate steps are controlled to be above the minimum working temperature for the lower flow stress. It is possible that during these intermediate temperatures grain growth can occur after the recrystallization. In generals, a fine grain structure is usually required. Thus it is necessary to control the lower working temperature. To ensure a fine recrystallized grain size the amount of deformation in the last step should be relatively large. Hot working of metals and alloys is usually and generally carried out at temperatures higher than 0.5-0.6 T_m and at high strain rates in the range of 0.5-500 s^{-1} (Courtney, 1990)

MATERIALS AND EXPERIMENTAL PROCEDURES

From the deformation procedure of previous works (Novy, 1999), the parameters of hot working procedures to investigate the formability of the NiMoCr alloy in terms of reheating temperature level, amount of % reduction during hot rolling, and number of hot rolling steps (reduction passes) applied were investigated. The chemical composition of the alloy in wt% is stated in Table 1.

In order to investigate the deformation ability of alloy in dependence of reheating temperature and successive rolling step reductions of different extents, the various multiple-step rolling procedures were carried out. The diagrams of these rolling procedures with detailed rolling conditions are presented in Figures 1-5, for specimens X1-X5 respectively. Note that all rolling experiments have rolling rate: v = 0.8 m/s.

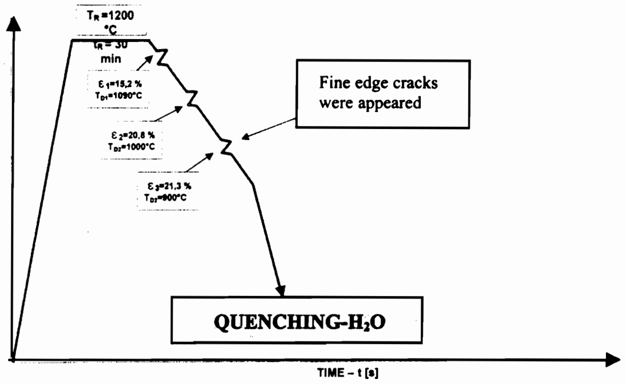

Figure 1 Diagram of rolling experiment X1

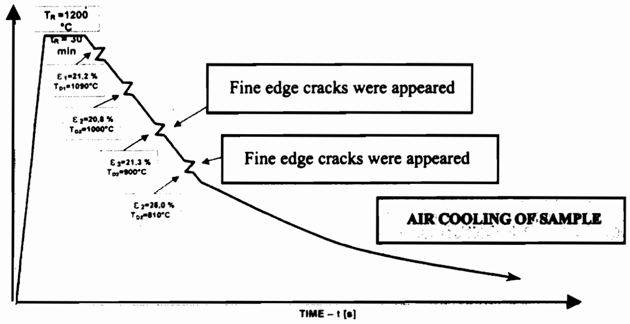

Figure 2 Diagram of rolling experiment X2

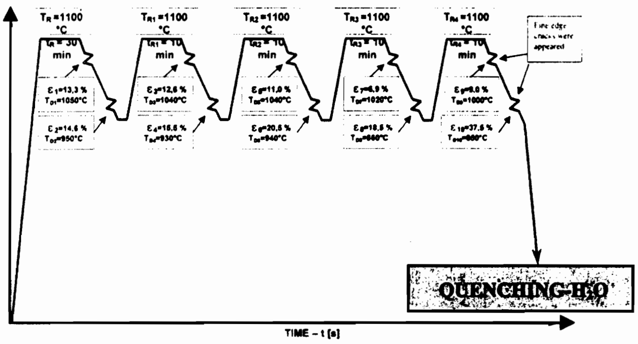

Figure 3 Diagram of rolling experiment X3

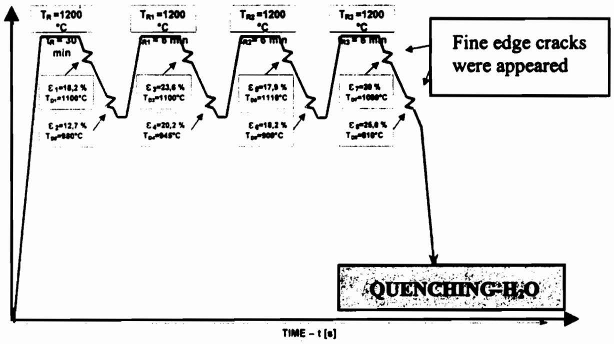

Figure 4 Diagram of rolling experiment X4

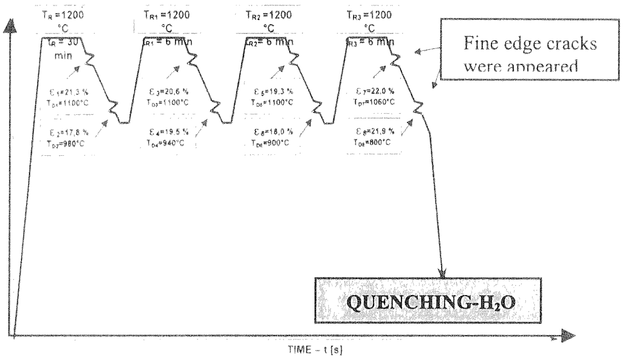

Figure 5 Diagram of rolling experiment X5

The aim of these tests was to investigate and evaluate the effect of reheating temperature level on multiple-step hot rolling, and the effect of the repeated reheating on deformation ability of the alloy when different rolling reductions were introduced for the next rolling pass.

Receiving the respond on the alloy deformation ability to various applied working conditions Deeker and Sims (1972) and on microstructure evaluation from preliminary structure analysis the hot working conditions have been set to carry out the experiment to optimize the alloy structure for high temperature properties and further advanced forming. The details of the working procedures selected for structure and properties optimization, involving the deformation and recrystallization process are stated in Table 2 (See in page 18). The wedge cracked test was performed to check the fine edge cracks which determine the limit of formability.

The samples of 15 x 20 x 100 mm were prepared for rolling experiments.

For program A, the details of the work conditions for those specimens are as follows:

- Reheating to 1200°C for 30 minutes and then followed by hot working passes for size reduction in the range of 17-18% (actual rolling temperature range is in the range of 1025-1075°C.
- For program B the details of the work condition for those specimens were as follows:
- Reheating to 1100°C for 30 minutes and then followed by hot working passes for size reduction in the range of 11-14% (actual rolling temperature range is in the range of 875-975°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 Tensile test results at various temperatures.

Specimen	Yield strength (MPa)	Ultimate tensile strength (MPa)	Testing temperature (°C)
1	197	484	25
2	129	409	400
3	111	294	600
4	103	207	800
5	101	203	1000

Table 3 The specimen dimension before and after tensile tests.

Testing temperature (°C)	Before testing			After testing		
	Specimen diameter (mm)	Cross section area of specimen (mm^2)	Specimen gauge length (mm)	Specimen diameter (mm)	Cross section area of specimen (mm^2)	Specimen gauge length (mm)
20	10.01	78.7	50	6.24	30.58	77.06
400	10.01	78.7	50	5.10	20.43	85.10
600	9.99	78.38	50	7.65	45.96	71.60
800	10.01	78.7	50	9.75	74.66	53.10
1000	9.99	78.38	50	9.86	76.33	52.00

RESULTS AND DISCUSSION

Tensile tests

The tensile test results carried out at different high temperatures are shown in Tables 2 and 3. It should be noted that the tensile testing rate was 2 mm/min; maximum load of tensile testing machine was 250 kN.

The tensile properties at different high temperature are given in Figure 6. The NiMoCr alloy exhibited a continuous decrease in ultimate tensile strength and yield strength (at lower rate) when the temperature was increased. The difference between both curves decreased as temperature increased. At the temperature range of 800-1000°C, both tensile strength and yield strength were quite stable and show a very narrow difference of both final strength levels. The temperature should mainly affect the strength of the alloy by means of the difference in diffusion rate (atom and dislocation movement), different slip patterns and the change in grain boundary strength.

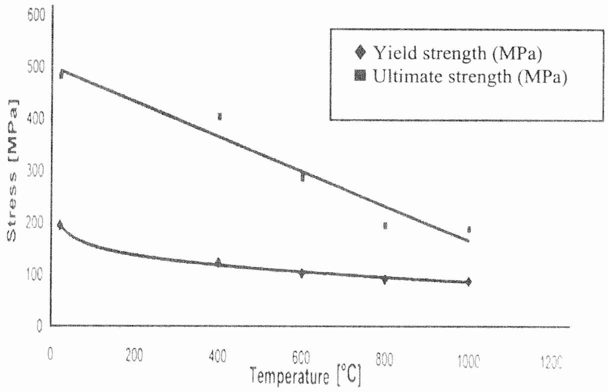

Figure 6 Tensile tests at different temperatures.

Ductility and reduction in area generally increased with a rise of temperature from room temperature until 400°C, as illustrated in Figure 7. This might be due to the fact that over ambient temperature up to 400°C, the movement of atoms became easier. However, at temperatures over 400°C, both characteristics vastly decrease with the increase of temperature. It might be noted that over this temperature, the velocity of diffusion of solute atoms became as high as the velocity of the dislocation movement. Serration should then occur and then ductility should decrease. This caused the hardening of the grains and strain concentration at grain boundaries at which the fracture occurred in an intergranular manner. Both curves in the figure are quite similar and stable at temperatures over 800°C.

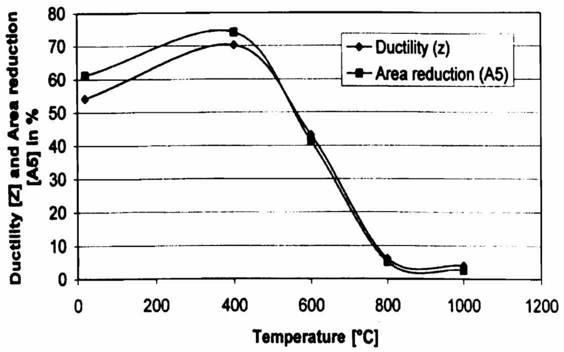

Figure 7 Dependence pf the formability parameters on temperature

HOT WORKING CHARACTERISTICS

High Temperature Wedge Crack Test for One Pass Rolling

From high temperature wedge crack tests (see Figure 8), fine edge cracks were observed at surfaces of worked specimens when the amount of hot reduction along the Y-axis reached 30.8% at a deformation temperature of 900°C (after heating at 1000°C for 30 minutes) and at 33.6% at a deformation temperature of 1000°C (after heating at 1100°C for 30 minutes). The obtained results show the limit of the hot formability values of the first pass of hot rolling at the minimum actual operated deformation temperature in case of 900°C. From the results, it might be suggested that higher deformation temperature provides the higher critical amount of hot reduction. This also can be explained that at a higher temperature the higher rate of dynamic recovery and/or dynamic recrystallization could occur, which should suppress the effect of work hardening. However, the edges of worked specimens, where usually were the lower deformation temperature than specimen interior, could cause the higher effect of work hardening resulting in lower metal flow or ductility. In such conditions, too high strain concentration preferentially appeared at grain boundaries, in which the first edge crack occurred.

1. *Heating at 1000°C for 30 minutes, Deformation temperature at 880°C, Deformation rate at* $0.8s^{-1}$*. The critical amount of deformation was found to appear when the amount of hot reduction in y axis reached at 30.8%*
2. *Heating at 1100°C for 30 minutes, Deformation temperature at 1000°C, Deformation rate at* $0.8s^{-1}$*. The critical amount of deformation was found to appear when the amount of hot reduction in y axis reached at 33.6%*

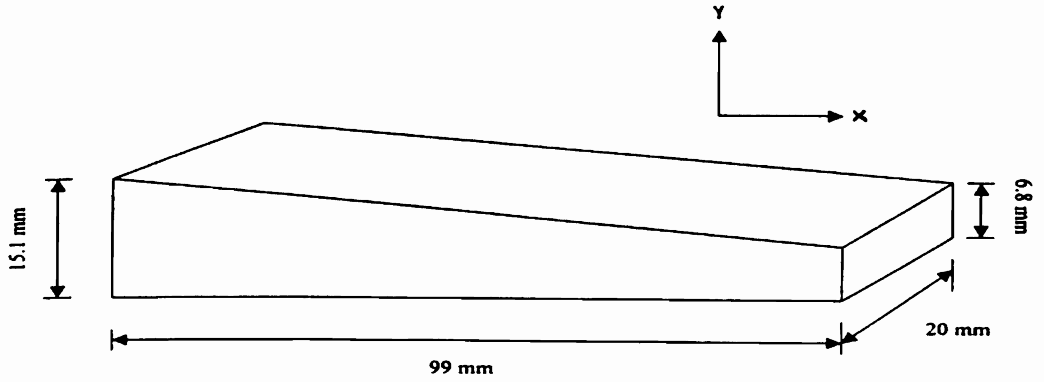

Figure 8 Wedge crack tests.

Multi Passes Hot Rolling Ability of The Alloy

As also found in previous works Hosnedl, *et al.* (1998), the only one-step hot rolling after only one reheating at 1100°C and at about 12% reduction could not provide the desired microstructure after annealing. For two-steps hot rolling with one reheating of 1100°C for 12% + 14% reductions could result in a more uniform microstructure (Navy, *et al.* 1999; and Navy, 1998). However, for multi steps hot rolling with one reheating (such as at one reheating temperature at 1200°C for 15% + 20% + 20% or 20% + 20% + 30% reductions from the tests, as shown in Figures 1 and 2, could cause many fine edge cracks to appear in worked samples.

In multi passes (3-4 steps) hot working after only one preheating, the fine edge cracks were found in both cases, after the third pass. This implies that the remained working temperature could not eliminate absolutely work hardening, which originated during the first and second pass, by dynamic recovery and/or recrystallization during hot deformation and by static recovery and/or recrystallization during specimen transportation from the furnace. The ductility of the alloy tended to decrease in each step due to the decrease of the deformation temperature. When the reduction was introduced to the worked specimen in the third pass, this excessive load caused a high strain concentration to build up in the samples and resulted in the appearance of fine edge cracks at the specimen surface, which was the most deformed area during hot rolling. These results investigating the limit formability at multi passes hot rolling and high temperature wedge crack tests were considered and utilized to design the hot working procedure of program A and B.

In another experimental multi passes of hot rolling (2 passes in each cycle and each cycle followed by reheating according to schedule presented in Figures 3-5), the results show that the reheating following each hot rolling cycle provided good ductility for the next step of double passes hot rolling successively. Each next reheating assisted to reduce the effect of previous work hardening. However, the selected reheating conditions (temperature and time) could not generate the full recrystallization in the microstructure as it can be seen in recrystallization behavior, Diagram 1 of previous work, (Wangyao, *et al.* 2003). The recrystallization diagram shows that the annealing conditions at 1100°C with a hold of 10minute-annealing could provide about a 90% recrystallized structure. It can be seen that after the fifth cycle of sample X3 and after the fourth cycle of samples X4 and X5, the edge cracks appeared already. The crack appearance can be explained by a similar idea in the above paragraph. However, in this case, the main reason to cause the cracking was mainly from the continuous increasing of the remained work hardening after each reheating (imperfect annealing); which deformation temperatures were quite constant. It can be noted that the samples X4 and X5 have the similar hot working characteristics even though they had the small detailed difference in amount of % reduction in each cycle. The total amount of % reduction in the first passes and second passes were quite similar (sample X4: 1st pass 79.7% & 2nd pass 51.1% and X5: 1st 83.2% & 2nd pass 55.3%).

The reheating conditions (1100°C for 10 minutes) of sample X3 should provide the finer recrystallized grain structure (and/or lower amount / rate of grain growth) after each reheating cycle (compared to the coarser recrystallized structure or amount of grain growth of sample X4 and X5) resulting in better ductility or lower resistance for hot deformation. However, when considering all results of samples X3, X4 and X5, the total amounts of % reduction of both first and second passes from every cycle after the first fine edge crack appearing had a very similar limitation of % reduction, which were 73.285%, 76.609%, and 78.658%, respectively. From the obtained multi passes hot working results, the procedure parameters such as % reduction in each pass, preheating, reheating, and actual deformation temperatures, and number of hot working cycles can be applied and utilized in the production line to produce sheets and plates as a final product.

Hot rolling Ability of the Alloy for Program A and B

The nickel base solid solution NiMoCr alloy was designed to provide high strength at elevated temperature together with good corrosion resistance in a specific nuclear environment. Therefore, the fabrication of this alloy requires a more complicated procedure compared with either high strength stainless steels or high strength alloyed steels. The results received with the hot rolling process condition testing of the alloy are shown in Figures 9-12.

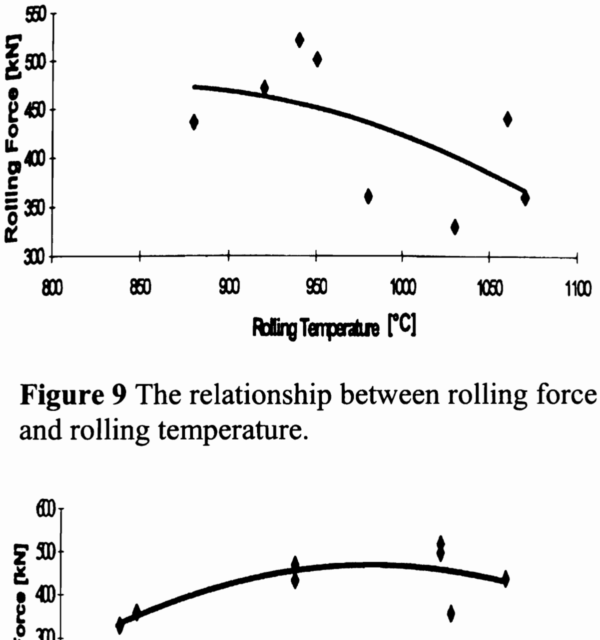

Figure 9 The relationship between rolling force and rolling temperature.

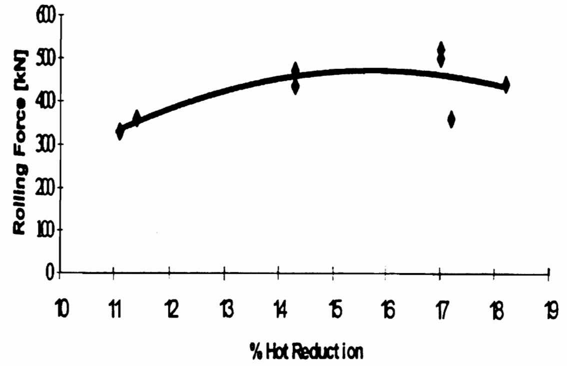

Figure 10 The relationship between rolling force and percentage reduction during hot rolling.

The rolling force during hot rolling in each specimen was measured and then calculated for technological deformation resistance TDR (σ_{Ds}) of the alloy. Figure 9 shows the relationship between rolling force and rolling temperature during two-step hot rolling in program A and B. In the selected temperature region, the rolling force is influenced by the rolling temperature.

The results imply that when the rolling temperature increases the rolling force required for the alloy deformation decreases as the resistance to slip decreases with the increase in temperature. Furthermore, at higher rolling temperatures, the working conditions usually should allow a greater amount of simultaneous dynamic recovery and/or recrystallization as well. As regards to the effect of reduction introduced, in Figure 10, the rolling force increases with an increase of % deformation during hot rolling. The higher % reduction requires more rolling force to encounter with the higher amount of strain hardening. Therefore, it can be summarized that the rolling force for the alloy should increase more rapidly with a higher amount of % deformation and/or with faster cooling of the specimen rolled down (lower rolling temperature).

The results on TDR (σ_{Ds}) in dependence of rolling temperature in the range of applied reductions are presented in Figures 11 and 12. Both curves show that results received on TDR have the same tendency as for the rolling force and, therefore, can be explained by the same idea. However, after the peak point of curve in Figure 12, the technological deformation resistance tends to slightly decrease. This was due to the more pronounced effect of higher deformation temperature. The TDR was calculated using the following Formula:

$$\text{TDR}\ (\sigma_{Ds}) = \text{Rolling force} / S \qquad (1)$$

Where $S = b_s (R.\Delta h)^{0.5}$ and $b_s = 0.5\ (b_0 + b_1)$

Δh is the difference in sample thickness before and after hot rolling, b_0 and b_1 represent the initial specimen width before hot rolling and the width of specimen after hot rolling respectively.

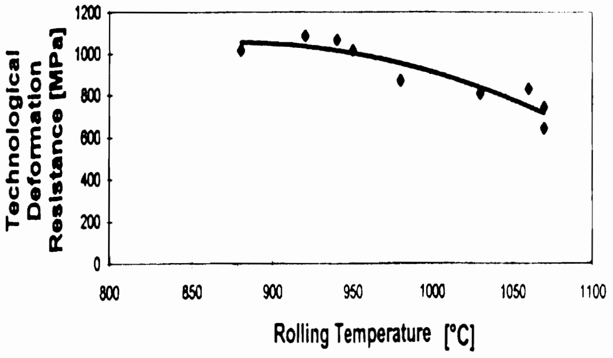

Figure 11 The relationship between Technological Deformation Resistance (TDR) and Rolling Temperature.

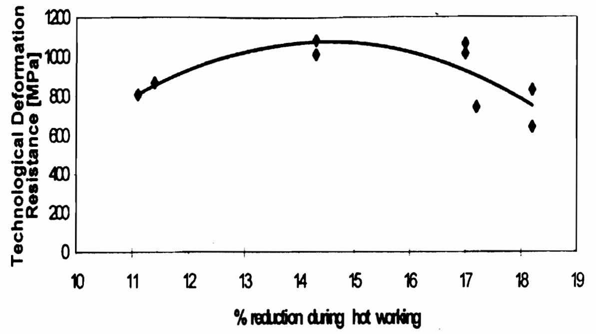

Figure 12 The relationship between TDR and % reduction during hot rolling.

However, from the previous work of (Wangyao, *et al.* 2001), it should be noted that the slip by dislocation movement was considered to be the major deformation mechanism in this NiMoCr alloy hot deformation according to the strain rate levels during hot working in range of 3-4.8s^{-1}. This range level was classified as a high strain rate (> 0.1 s^{-1}). The partial fine grain structure should be produced by dynamic recrystallization caused by an additional deformation mechanism such as grain boundary sliding to occur. It had been observed that the grain size (after full hot deformation and then quenching right away) became smaller than the initial grain size for program A and B; see in previous work of (Wangyao, *et al.* 2002).

CONCLUSIONS

1. For multi-step hot rolling with one reheating, (such as at one reheating, temperature at 1,200°C, for 15% + 20% + 20% or 20% + 20% + 20% + 30% reduction) this could already cause many fine edge cracks in worked samples after the third pass. This implies that the remained working temperature could not eliminate absolutely work hardening, which originated during the first and second pass, by dynamic recovery and/or recrystallization during sample transportation. The ductility of the NiMoCr alloy tended to decrease in each step due to the decrease of deformation temperature. When the reduction was introduced to worked specimen in the third pass, this excessive load can cause high strain concentration built up in the samples, resulting in an appearance of fine edge cracks at the sample surface, which was the most deformed zone during hot rolling.

2. Another experimental program of multi passes of hot rolling (2 passes in each cycle and each cycle followed by reheating), the results show that the reheating following each hot rolling cycle provided good ductility for the next step of double passes hot rolling successively. Each next reheating assisted to reduce the effect of previous work hardening by recrystallization. However, the selected reheating conditions (temperature and time) could not generate the full recrystallization in the microstructure. In this case, the main cause of cracking was mainly from the continuous increasing of the remained work hardening after each reheating (imperfect annealing), during which deformation temperatures were quite constant.

3. Hot working parameters obtained from all programs showed that rolling force and technological deformation resistance were very dependent on rolling temperature and amount of % reduction during hot working. Rolling

force and technological deformation resistance decreased with an increase of rolling temperature and/or decrease of amount of % reduction. However, in this study it can be noted that the amount of % reduction in the range of about 7.5% provided its effect on the rolling force and technological deformation resistance. Which was more than that obtained from the effect of rolling temperature in the range of about 45°C.

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