The effect of roughing deformation on strain induced precipitation kinetics of Nb(CN) in HSLA steel after finishing at 900° C.

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Abstract

A mathematical model of strain induced precipitation kinetics of Nb(CN) in HSLA steel has been established and the model gives accurate prediction when only a single deformation was given. In this work, the model is evaluated by employing roughing deformation at different strain before finishing at 900°C. The results in this way show that both roughing strain and temperature have a significant influenced on precipitation kinetics after finishing. As a roughing strain increased, start time for 5% fraction precipitation is considerably accelerated and achieved a constant value when roughing strain was performed over 0.3.

I. Introduction

There are limited number of works, which investigate the precipitation kinetics of Nb(CN) in HSLA steel when roughing deformation is applied. Most of work concerned with strain induced precipitation kinetics when only a single deformation at finishing temperature is employed. An attempt has been made, by Janampa[1] using different grade of HSLA steel and reported that the roughing deformation has a significant influenced on the start time for 5% fraction precipitates of Nb(CN) after the finishing rolling. However, no empirical equation has been developed to assess these effects. It has been measured that time for start 5% precipitation was five time faster then a single deformation.

Based on classical nucleation theory, Dutta and Sellars in 1987[2], proposed a model which relates the precipitation start time to strain, strain rate, temperature and steel composition. The model can be rewritten as,

$$t_{0.05} = A[Nb]^{-1}\varepsilon^{-1}Z^{-0.5}\exp\left[\frac{270000}{RT}\right]\exp\left[\frac{2.5x10^{10}}{T^{3}(\ln k_{s})^{2}}\right]\ldots.1$$

Where,
[Nb] = Concentration of Nb in solution (wt%)
ε = Appliedd strain
Q_d = 270 kJ/mol for diffusion of Nb in austenite phase
Z = Zener-Holomon Parameter,
Z = $\varepsilon \exp[Q_{def}/RT_{def}]$
ε = Strain rate in sec^{-1}
Q_{def} = Apparent activation energy for deformation, J/mol
R = Gas constant (8.31 J/mol K)
T = Absolute temperature during holding, K
k_s = Solubility parameter
A = Constant (1.5×10^{-5} single deformation, 3×10^{-6} roughing deformation)

The solubility, k_s, can be described according to Irvine et al[3] as,

$$k_s = \log[Nb][C+12/14N]/10^{2.26-6770/T} \ldots..2$$

Valdes and Sellars [4] invetigated that the effect of reheating/roughing temperature on precipitation kinetics Nb(CN) in HSLA steel. The kinetics of strain induced precipitation is determined using the same

Table 1. Chemical compositions of steels investigated, wt%

Steel	C	Si	Mn	P	S	Al	N	Nb
1	0.012	0.45	1.30	0.005	0.003	0.02	0.0024	0.1
2	0.1	0.37	1.35	0.02	0.019	0.041	0.0042	0.031

principle as for hot rolling,
but the final deformation, during which the effect of flow stress is measured. This technique, however, has been used by Dutta [5] to investigate X-70 HSLA steel. The experimental results obtained by Valdes and Sellars[4] might be seen in figure 1a and 1b respectively. The figure 1a shows that the time to peak strengthening depends on the reheating temperature when no roughing deformation is performed, and on the roughing temperature when sufficient strain is applied by the roughing deformation. In the figure 1b, the time to peak strengthening obtained by different Nb-steel used is plotted as a function of reciprocal temperature. The temperature dependence of the equivalent isothermal holding time for peak strengthening, t_m, according to a single straight line in the figure can be expressed as,

$$t_m = 1.45x10^9 \exp\left[\frac{-200.000}{RT}\right] \ldots..3$$

The equation above leads to a constant of A in the strain induced precipitation kinetics model in equation 1 by recalling the assumption that $t_{0.05} = 0.5\ t_p$ [6], (t_p= time to the peak stress) can be written as,

$$A = 2x10^2 \exp\left[\frac{-200.000}{RT_r}\right] \ldots\ldots4$$

Where T_r is roughing temperature or reheating temperature when no roughing deformation was given. For HSLA steel which subjected to roughing deformations, the estimated value of $A = 3 \times 10^{-6}$ is found in close agreement with strain induced kinetics model in equation 1 when large roughing deformation at a temperature of 1063° C takes place. It is also reported that the multipass roughing deformation at progressively decreasing temperature may lead to minimum precipitation time for the final roughing temperature even when the deformation in each roughing pass is not excessively large. In the present work, the effect of a single roughing strain on precipitation kinetics after finishing at 900°C are investigated and the strain induced precipitation kinetics model is evaluated by using two different grade of Nb-steel.

II. Experimental Procedures

1. Steel use in this experiment

Table 1 shows the chemical composition of steel use in this work. Both HSLA steel (Nb-steel) containing high niobium-low carbon and low niobium-high carbon are used in this investigation. The steel were vacuum-melted and cast into 15 cm diameter ingot, then reheated and extruded into 30 mm wide and 11 mm thick bar. The steel containing low niobium-high carbon is received as conventional hot-rolled plate steel.

Both steel plate and extruded bar were subsequently cut and machined for making compression specimens, as illustrated in figure 2, and then to prevent oxidation and scaling during reheating in the furnace, the specimen were chromium plated to thickness of 15 μm. Micrograph of chromium plating after reheating at a temperature of 1200°C is displayed in figure 3.

2. Testing Procedure.

The precipitation kinetics of Nb(CN) was followed by using servotest machine (plane strain compression testing). This mechanical method has been developed and established by Liu and Jonas [7]. This method was used to detect the start time for precipitation, which based on the analysis of isothermal stress relaxation data showing that relaxation is retarded at the start of precipitation.

The condition of all of the test should be the same, so that the specimens were first heated to an austenizing temperature to dissolve Nb(CN) precipitates. The solution temperature for Nb(CN) precipitation in HSLA steel can be calculated by using an equation developed by Irvine et al [3],see equation 2, as,

$$Log[Nb][C+\frac{12}{14}N]=2.26-\frac{6770}{T} \quad .....5$$

Where [Nb] and [C+12/14N] are the equilibrium of Nb, C and N in solution (wt%) at the absolute temperature, T. Reheating has been done approximately 50 – 100°C above temperature obtained by calculating in equation 5. In steel 1, high niobium- low carbon, reheating temperature is determined at a temperature of 1150°C, while for steel 2, low niobium-high carbon, is 1200°C. To ensure that the dissolution is complete, the specimen is heated up to the austenising temperature and held for 15 minutes to stabilize the temperature. The characteristic of these steel can be clearly seen in stoichiometric line composition in figure 4.

3. Thermomechanical treatment

For steel 1, after reheating at a temperature of 1200°C, the specimen are air cooled to different roughing temperatures of 1150, 1100, 1050 and 1000°C. A thermomechanical schedule adopted for this steel is illustrated in figure 5. After roughing at strains of 0.1, 0.3 and 0.7, the deformed specimen is furnace cooled to 900°C. After a finishing at strain of 0.5, relaxation is begun for 400 sec, then the specimen quenched.

For steel 2, after reheating at a temperature of 1200°C, the specimen is air cooled to the roughing temperature. The thermomechanical treatment for this steel had two thermal cycles. Figure 6 and 7 respectively show the thermal cycle applied to test specimens.

Before finishing at 900°C, two roughing deformations are applied. The first roughing at 1150°C, then furnace cooling for about 30 sec before second deformation is performed at 1050°C. The first roughing and finishing strain were kept constant at 0.2 and 0.5, while the second roughing deformation was performed at different strain of 0.1, 0.3 and 0.5. After finishing, the relaxation was started.

After roughing at temperature of 1050°C with strain of 0.4, the deformed specimen is air cooling to 900°C and then finishing deformation were performed at strains of 0.1, 0.3 and 0.5 and strain rate of 1, 5 and 10 sec^{-1} for each finishing strain followed relaxation. Typical temperature-time recorded from the embedded thermocouple for steel 1 is shown in figure 8.

4. Optical Metallography

Etching to reveal austenite grain boundaries in both undeformed and deformed specimen was carried out by using aqueous picric acid containing a small amount of HCl [8] and a wetting agent teepol for steel 1. For steel2, Japanese etchan addition of small amount of HCl is used. After polishing, specimen is etched at a room temperature for 30 seconds. Austenite grain size is measured by using linear intercept method.

III. The experimental results

1. Austenite Grain.

The reheated austenite grain structure of steel 1 was examined for thr reheating Temperatures used in this experiments, 1200 and 1150°C. In general the reheated austenite grains have homogeneous grains and the grain size is measured in the range of 150 ± 5µm to 115 ± 8µm in diameter. Typical structures of austenite grain is illustrated in figure 9. While for steel 2, prior austenite grain size is measured as 60 and 116 µm after reheating at temperature of 1150 and 1200°C. Small austenite grain sizes are observed after reheating temperature of 1000 to 1050°C in both steels. The austenite grain size of 20± 2µm is measured. At this temperature Nb(CN) precipitates, have strongly retardad the austenite grain growth. Inhomogeneous elongated austenite grains are clearly observed in steel 2 after a single deformation and relaxation. Small elongated grains are formed in the middle of deformed specimen compared with that are near the surface (dead zone). Figure 10 shows elongated austenite grains after single deformation at temperature of 900°C.

2. Nb(CN) precipitates

Representative Nb(CN) solubility data according to the equation 5 which proposed by Irvine et al[3] indicates that all Nb(CN) in both steel 1 and steel 2 should be in solution at the reheating temperature. To observed Nb(CN) precipitates, carbon coated method (carbon replication) was used. In figure 11 shows, carbon extraction replica micrograph of insoluble Nb(CN) at that reheating temperature.

It is interesting to observe Nb(CN) particles after roughing at higher temperature. Nb(CN) particles around 20 nm in diameter are observed in steel 2 after two roughing deformations. The micrograph of carbon replica of these precipitase is shows in figure 12 after air cooled to 900^{0}C then quenched after holdng at that temperature for 100 seconds.

3. Strain Induced Precipitation Kinetics of Nb(CN)

In general, as roughing temperature decreases, time for precipitation also decreases. At a roughing temperature of 1050°C, start time for 5% precipitation is predicted shorter than at roughing temperature of 1150°C. To predict start time for 5% Nb(CN) precipitates, mechanical method of stress relaxation technique is used. The stress relaxation curve of both roughing temperature are displayed in figure 13a and 13b respectively. All results of the effect roughing temperature on precipitation kinetics Nb(CN) for both steel1 and 2 are displayed in figure 14. The figure shows that strains up to 0.3 in steel 1 have a significant effect on precipitation kinetics of Nb(CN). As roughing strain increases from 0.3 to 0.7 at a constant roughing temperature, precipitation kinetics of Nb(CN) appears to reach a minimum time. The minimum time at around 1.8, 2.2 and 3.5 seconds are reached at roughing temperature of 1000, 1050 and 1150°C respectively.

IV. Discussion

Microstructure evaluation

For steel 1 and 2, the microstructure of reheated austenite grains appear to be fully martensite with no evidence of ferrite formation along prior austenite grain boundaries. The overall coarsening behaviour is illustrated in figure 15 as a function of reheating temperature. A mixed austenite grain in steel 2 is observed at a temperature lower than solution temperature of 1050°C. It is reported that the grain coarsening temperature is found to be about 40 to 100°C[9] less than the solution temperature. The grains coarsening temperature for this steel (steel 2) is assumed to be in close agreement with predicted[10].

It has been reported [10] that the steel which containing low wt% phosphorous, difficult in revealing the austenite grain boundaries after reheating at the solution temperature and the grain coarsening temperature cannot be identified. As shown in figure 15, at the same reheating temperature, steel 1 gives the austenite grain size larger than steel 2. It is interesting to note that at a solution temperature of 1150°C, in steel 2, highly-serrated austenitic boundaries are observed, see figure 16, which may signal some of precipitation Nb(CN) still affective to prevent the austenite grain growth. The fully recrystallised austenite grain after two roughing deformations is observed. The recrystallised austenite grain size of 27 ± 7μm is measured and it is found to be in agreement with the value expected from relationship of 28 μm [6] suggested as,

$$d_{rex} = 0.9 d_o^{0.67} \varepsilon^{-0.67} \ldots\ldots\ldots\ldots\ldots 6$$

Where d_o is the reheated grain size (μm) and ε is the equivalent true strain of roughing deformation. Ferrite phase was observed along austenite grain boundaries appears to be form during cooling (quenching).

Precipitation kinetics of Nb(CN) after Roughing and finishing.

The effect of strain induced precipitation of similar Nb-steel after single deformation at 900°C has been discussed by Siradj [11,12] and it is found that as strain increased, start time for 5% fraction precipitation is considerably accelerated. It seems that in close agreement with previously reported [13].

The effect of roughing strain on the start time for 5% precipitation Nb(CN) after finishing at 900°C obtained in this work is displayed in figure 17. The figure shows that the strain required to achieved the minimum time increases significantly with increasing reheating and roughing temperature. In general, the results obtained are consistent with that reported [4].

On the basis of figure 14, the lowest observed values of start time after strain of 0.3 to 0.7 are plotted in figure 17 as a function of reciprocal temperature. In this figure, T_r is defined as a roughing temperature in K. The present results for both steel 1 and steel2 can be represented by a single straight line which appears to be identical slope. Compared with the Valdes and Sellars results, the slope of present results is less steep.

The relationship between start time precipitation and roughing temperature in the present results can be written as,

$$t_{0.05} = 9.4x10^3 \exp\left[\frac{-87000}{RT_r}\right] \ldots\ldots\ldots 7$$

Here T_r is a roughing temperature in K. By using the strain induced precipitation kinetics in equation 1, leads to the constant A as follows,

$$A = 2.95x10^{-4} \exp\left[\frac{-87000}{RT_r}\right] \ldots\ldots\ldots 8$$

Considering the effect of a roughing temperature modifies in the equation 7 and 8 above, the strain induced precipitation kinetics in equation 1 can be modified as equation 9.

$$t_{0.05} = 2.95x10^{-4}[Nb]^{-1}\varepsilon^{-1}Z^{-0.5}\exp\left[\frac{-87000}{RT_r}\right]\exp\left[\frac{270000}{RT}\right]\exp\left[\frac{2.5x10^{10}}{T^3(\ln k_s)^2}\right] \ldots\ldots\ldots\ldots 9$$

It is of interest to compare the predictions of the present model with those experimentally reported by the worker [1]. The comparison is made in term of the precipitation-time-temperature diagram. As displayed in figure 18, the present model appears in close agreement with experimental results reported.

Conclusion

The minimum precipitation time attained appears to be increase significantly with increasing of roughing temperature. In general, the results obtained are consistent with that reported [4].

The modified model of strain induced precipitation kinetics of Nb(CN) by considering the effect of roughing temperature seems to be in closer agreement with experimental resulted obtained by Janampa[1]

The strain required to achieved the minimum time increases significantly with increasing reheating and roughing temperature.

As the roughing strain increases over 0.3, the minimum time seems to be constant at start time for 5% precipitation about 2 seconds after roughing 1050°C.

Reference.

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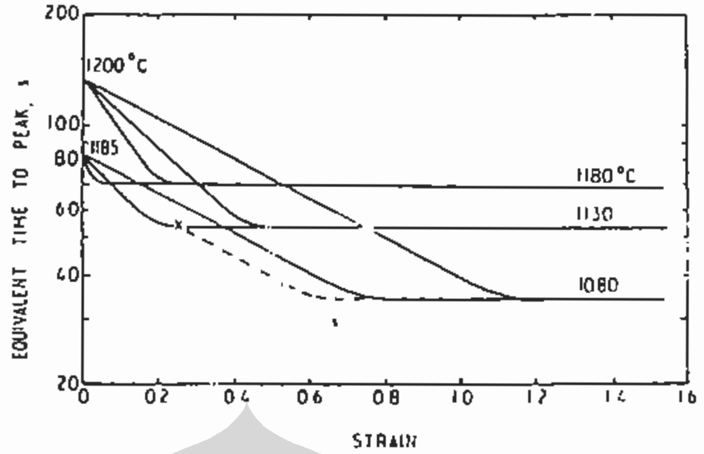
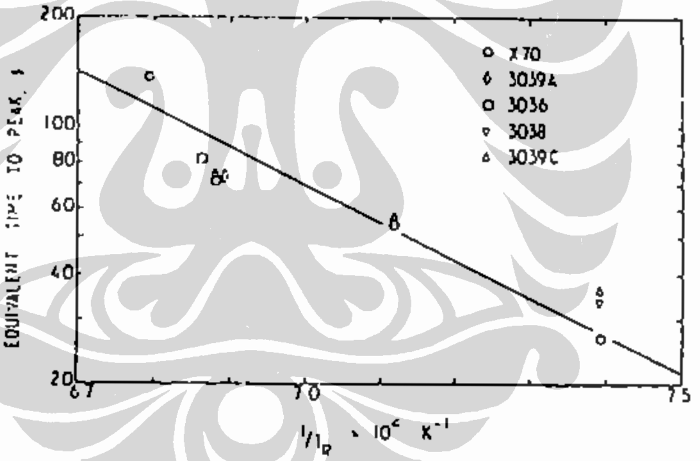
(a)



Summary of influence of reheating/roughing temperature and strain in roughing deformation on equivalent isothermal holding time at 900°C after 15% reduction at 950/955°C to attain peak strengthening in final finishing deformation

(b)



Dependence on reheating/roughing temperature of observed minimum equivalent isothermal holding time at 900°C for peak strengthening, after 15% reduction at 950/955°C.

Figure 1. The effect of reheating/roughing temperature and strain in roughing deformation by Valdes and Sellars [4]

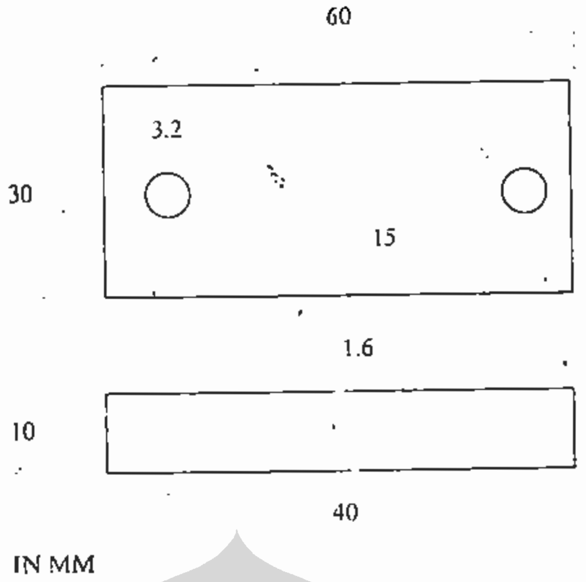


Figure 2. Illustration of plane strain compression test specimen

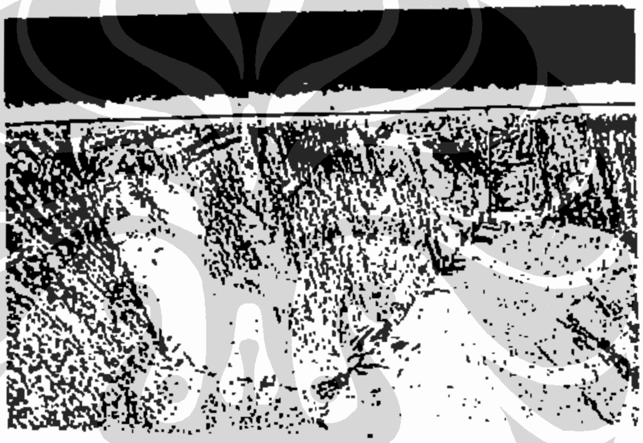


Figure 3. Micrograph of chromium plated

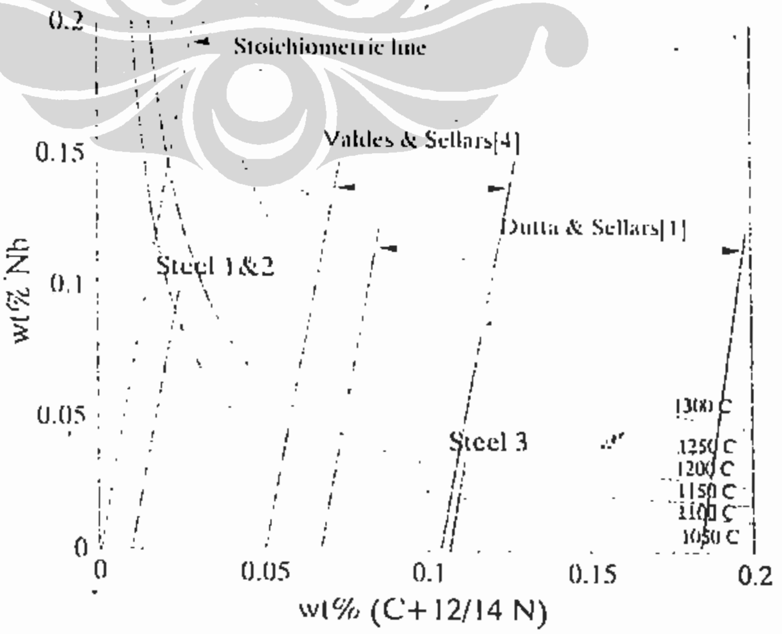


Figure 4. Stoichiometric line composition of Nb-HSLA steel

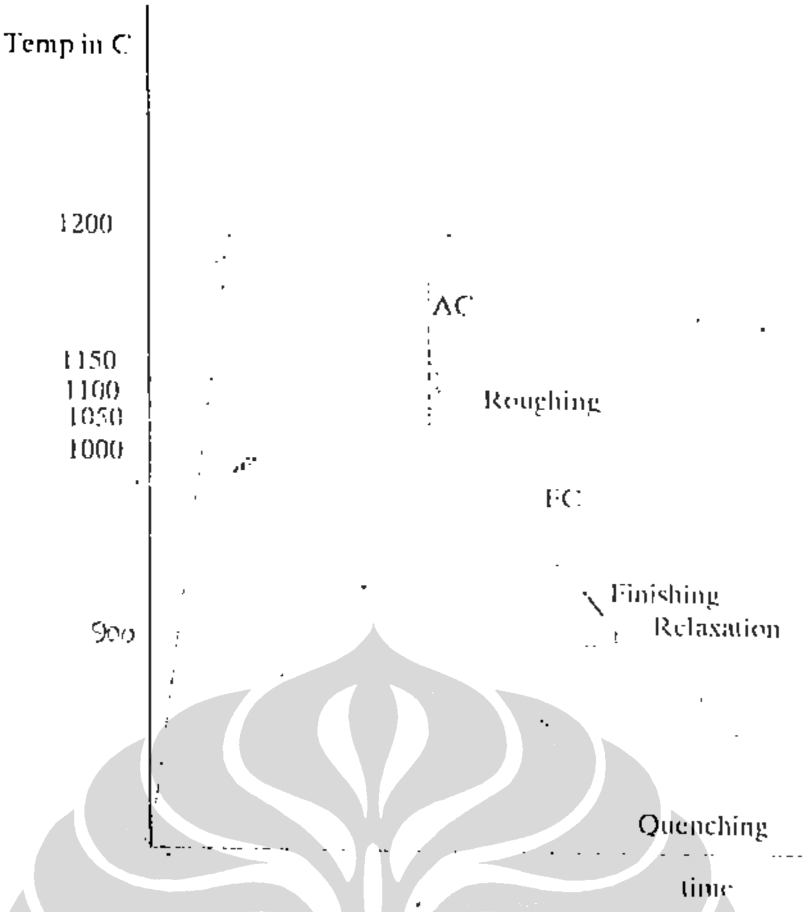


Figure 5. Thermomechanical cycle applied to test steel 1

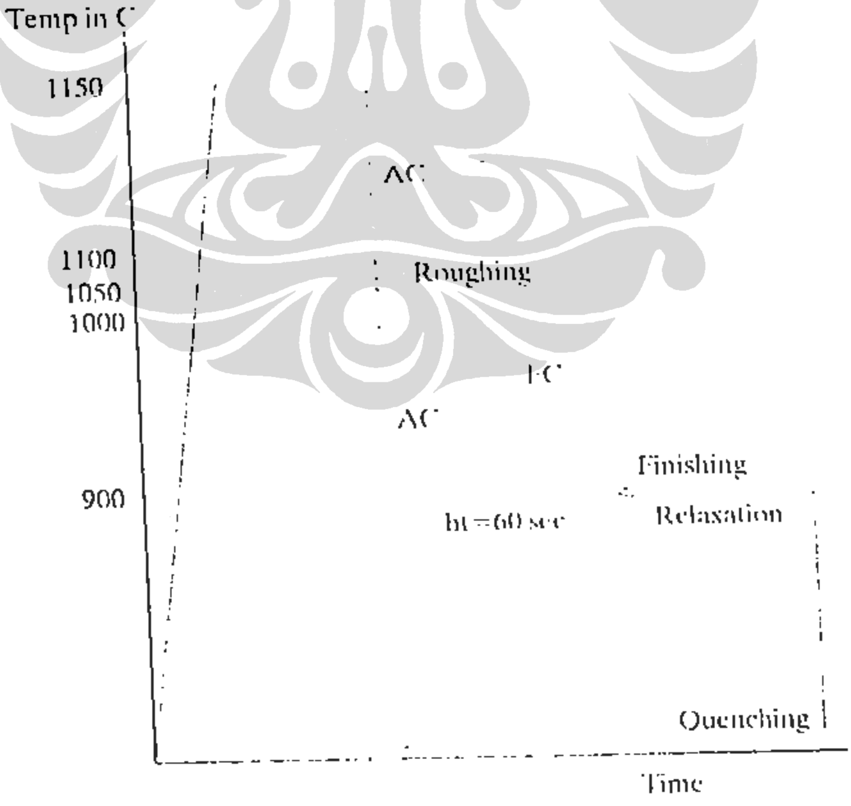


Figure 6. Thermomechanical cycle applied to test steel 2

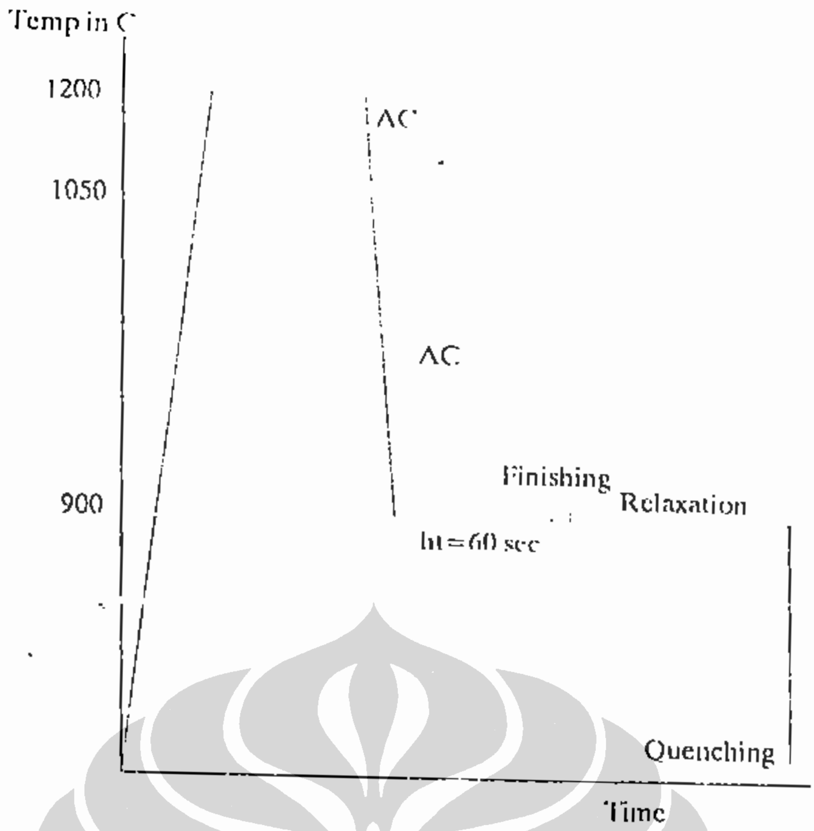


Figure 7. Thermomechanical cycle applied to test steel 2

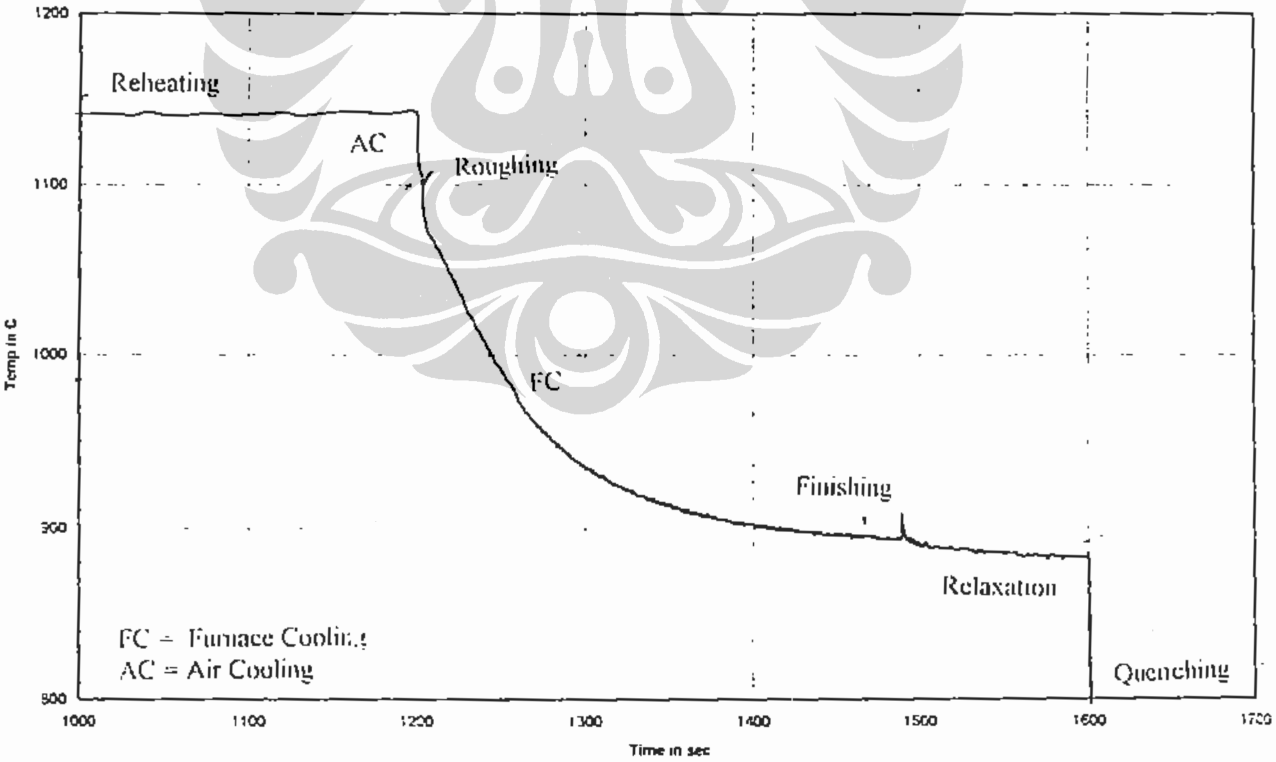


Figure 8. Typically temperature-time recorders from embedded thermocouples

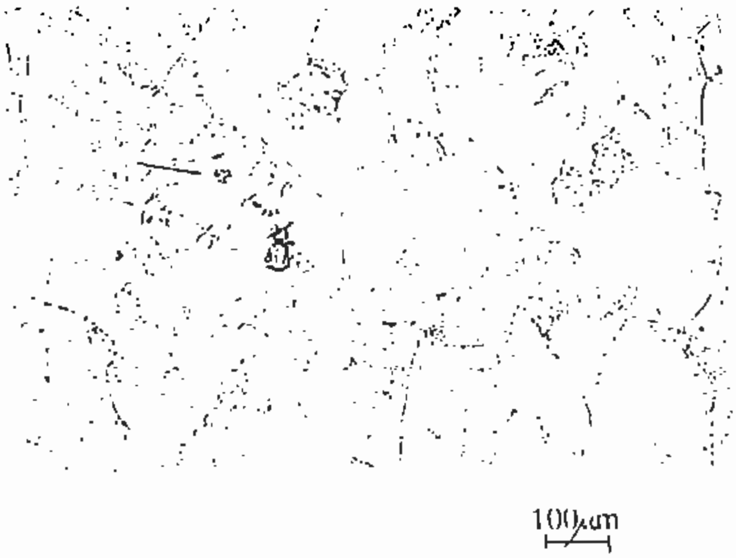


Figure 9. Micrograph of reheated austenite grains

Figure 10. Micrograph of elongated austenite grains for steel 2 after deformation at temperature of 900 C, with strain of 0.5 then relaxation for 60 seconds and quenched

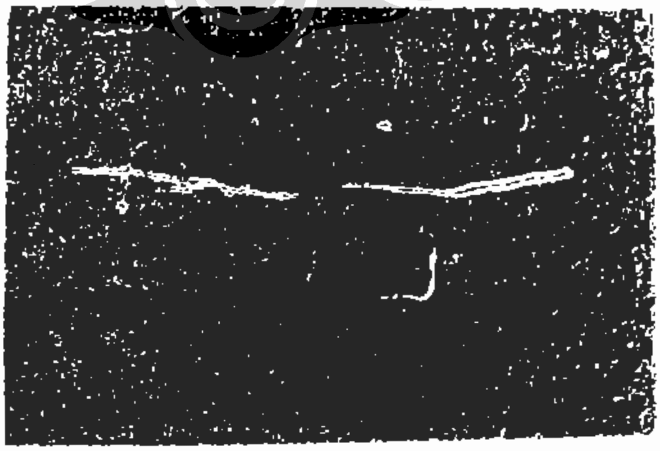


Figure 11. Carbon extraction replica micrograph showing insolube Nb(CN) precipitates at 1200 C for steel 2.

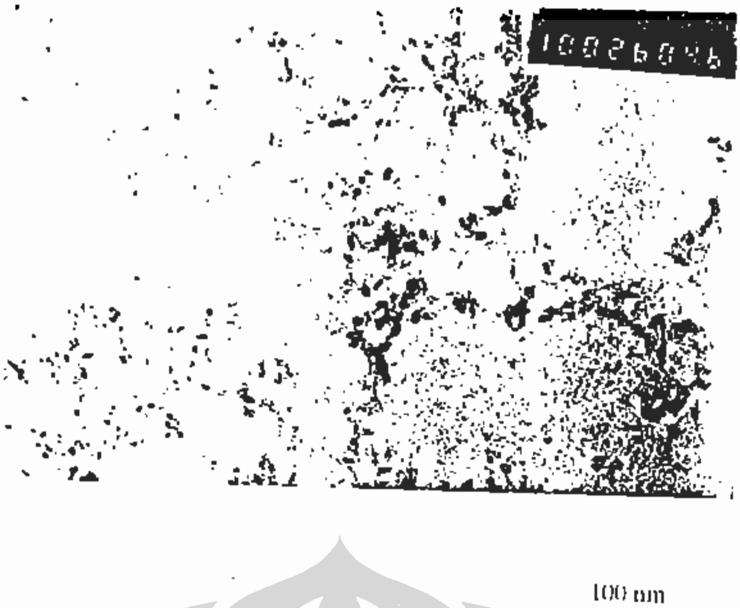


Figure 12. Carbon extraction replica micrograph showing Nb(CN) precipitates after finishing at 900 C and holding for 100 seconds.

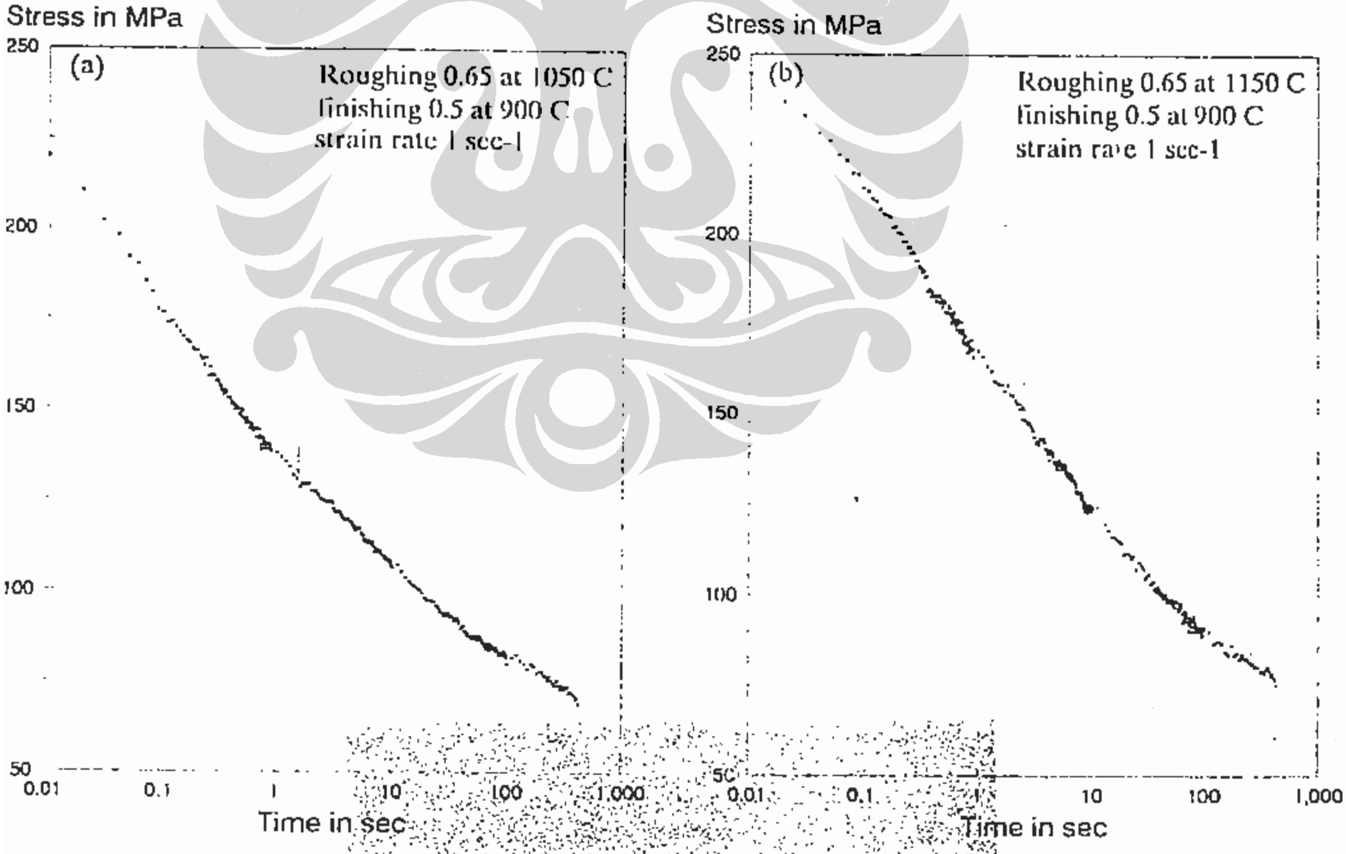


Figure 13. Stress relaxation curve for steel 1 at 900 C

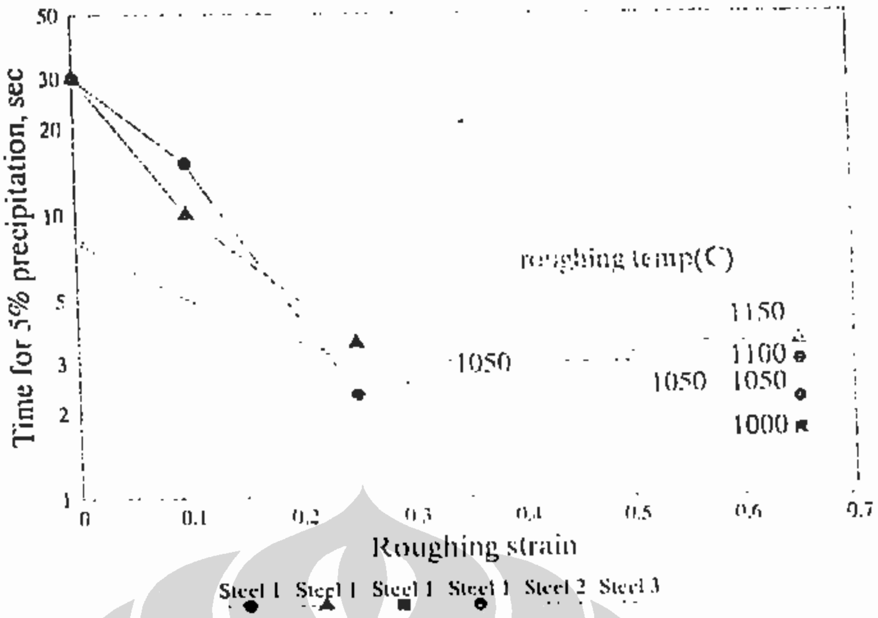


Figure 14. The influence of roughing temperature and strain in roughing deformationon time for 5% precipitation after finishing strain of 0.5 at 900 C.

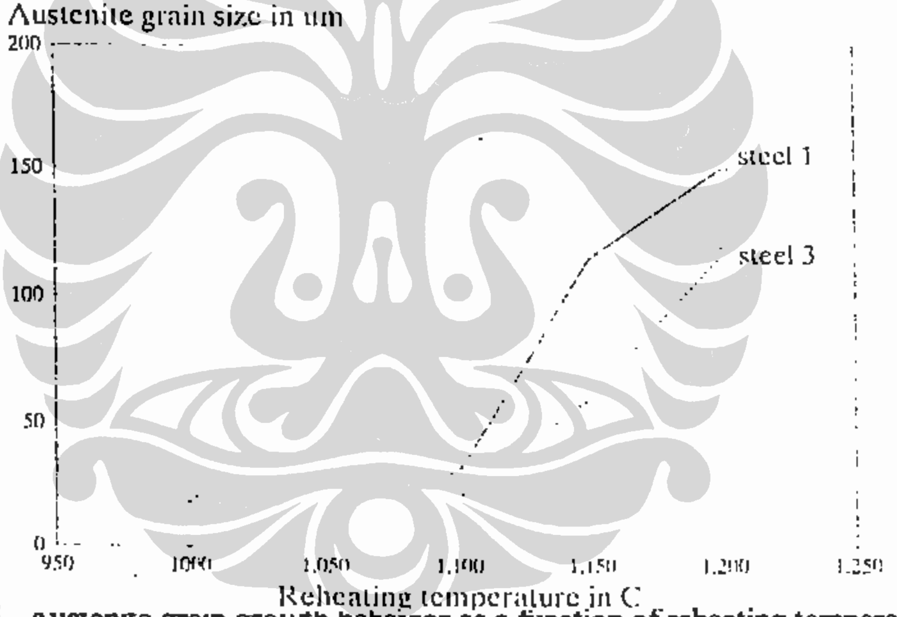


Figure 15. Austenite grain growth behavior as a function of reheating temperature for steel 1 and 2.

Figure 16. Highly-serrated austenitic boundaries in steel 2 after reheating at1200 C

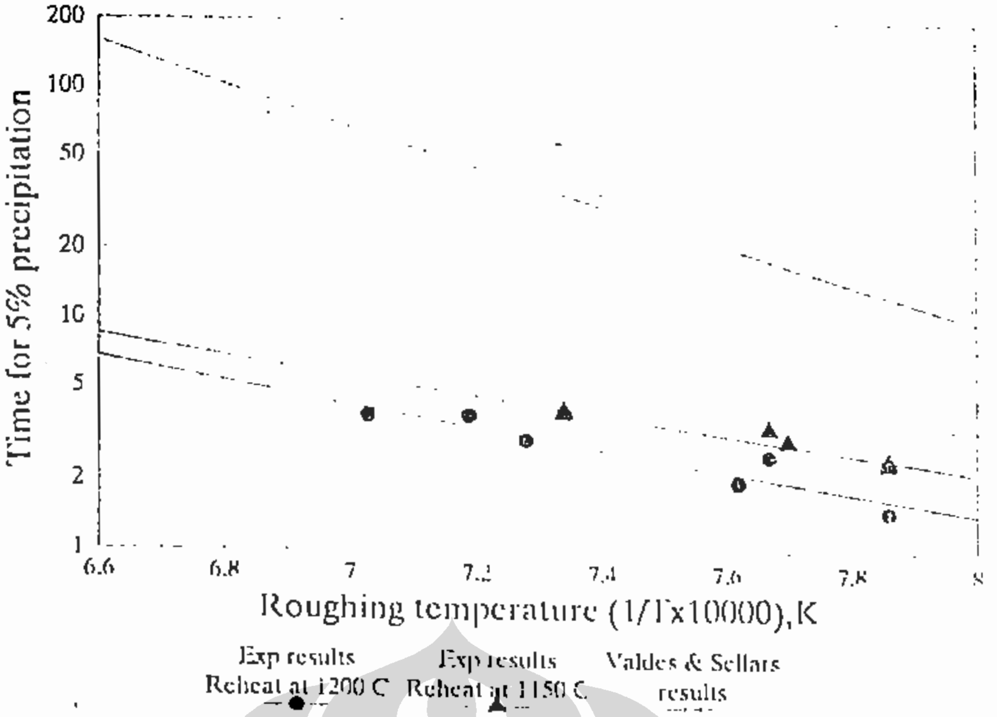


Figure 17. Relationship between start time for 5% precipitation and roughing temperatureafter finishing at 900 C at strain 0.5 and strain rate 1 sec-1, compared with experimental results by Valdes and Sellars[4].

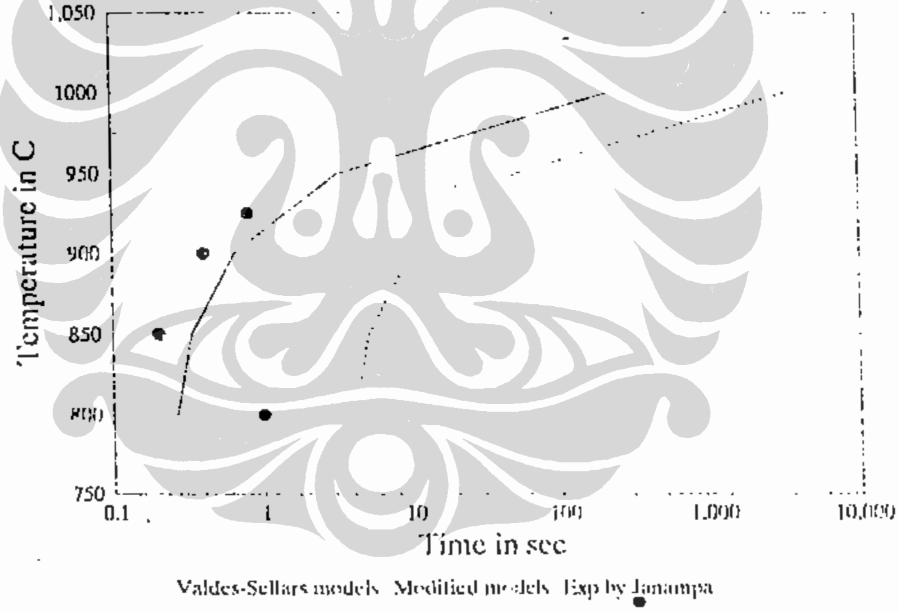


Figure 18. Nb(CN) precipitation start times in Janampa steel[1] containing 0.084% C, 0.0061 % N and 0.03% Nb predicted using the present and the Valdes & Sellars model(4).