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Effect of Hot Charge Rolling Condition on Mechanical Properties of Nb Bearing Steel Plate

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A large number of steel plates is currently produced by HCR(Hot Charge Rolling) process. In this study, the effect of HCR condition on the mechanical properties of Nb bearing steel is investigated.

If the charging temperature is above A_{r1} , the toughness of Nb bearing steel through HCR process is inferior to conventionally rolled one. This phenomenon comes from the larger amount of Nb in solid solution in the HCR process than in the CCR(Cold Charged Rolling) process. This Nb in solid solution markedly retards the recrystallization of austenite and then it precipitates finely as Nb(CN) after rolling in the HCR process. To improve the balance between the tensile strength and ductile-brittle transition temperature in Nb bearing steel at a charging temperature above A_{r1} , 1100°C is the most favorable for the rolling start temperature, because the finest austenite grain is obtained at this temperature range due to the homogeneous recrystallization.

KEY WORDS: high strength low alloy steel; plate; phase transformation; precipitation; recrystallization; strength; toughness.

1. Introduction

The manufacturing process of steel products has undergone profound changes from the standpoint of saving energy, and recently Hot Charge Rolling process (HCR) has been introduced.¹⁾ HCR process is a new rolling process, connecting the rolling process with the continuous casting process. In this process, continuous cast slabs are directly charged to furnace to roll without cooling until ambient temperature.²⁾

In order to attain an optimum balance between strength and toughness, High Strength Low Alloy (HSLA) steels have been produced by controlling the microalloying and the thermomechanical treatment. If HSLA steels are made through HCR process, then it is reasonable to think that the mechanical properties of these steels may be different from that of cold charged rolling (CCR) processed materials which is conventional process. Because the austenite before rolling shows a larger grain size and contains a larger amount of microalloy in solid solution compared with that of CCR processed materials.³⁻⁵⁾

There are hitherto many papers on hot cracking of continuously cast HSLA steels, but few papers on mechanical properties of hot charge rolled HSLA steels.⁶⁻⁸⁾

In this paper, the effect of hot charge rolling condition on mechanical properties of Nb bearing steel was examined, comparing their recrystallization during rolling.⁹⁾ Then the optimum rolling condition for Nb bearing steel through HCR process are discussed.

2. Experimental Procedure

2.1. Hot Rolling Experiment

Two kinds of steels are used in this experiment whose chemical composition is listed in Table 1, consisting of 0.03 % Nb bearing steel (Nb steel) and plain carbon steel (10C steel). These steels were melt in argon atmosphere and in a vacuum by using 200 and 50 kg induction heating furnaces, and cast into eleven and three metal molds, respectively, whose ingot shape is 100 mm square and 200 mm high. In order to measure the cooling speed and A_{r3} , A_{r1} transformation temperature of these steels after solidification, the thermocouple-R (0.5 mm in diameter) within alumina crucible was set in the center of one of those mold. After solidification, they were immediately removed from the mold, and charge to electric furnace at a various charging temperature which was surface temperature of them. Slabs were heated up to T_h °C and kept for 1 h. Then they were rolled to 15 mm thick plates through 14 passes. In the rolling process, two stage of control rolling were adopted. On the first stage, they were rolled to 45 mm thick plates through 6 passes above 850°C, then

Table 1. Chemical composition. (wt%)

Steel	C	Si	Mn	P	S	Nb	Sol. Al	N
10C1	0.09	0.27	1.32	0.008	0.007	—	0.021	0.007
Nb1	0.09	0.21	1.34	0.003	0.005	0.034	0.025	0.005
10C2	0.10	0.31	1.44	0.003	0.006	—	0.028	0.007
Nb2	0.09	0.29	1.43	0.004	0.003	0.036	0.023	0.007
Nb3	0.11	0.25	1.35	0.002	0.003	0.029	0.020	0.002

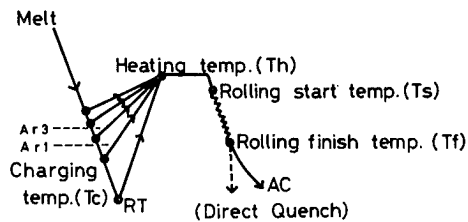


Fig. 1. Laboratory simulation method for hot charge rolling and conventional rolling processes.

rolled to 15 mm thick plates through the next 8 passes from 850 to 750°C (or 780°C) on the second stage. The heat cycle of hot rolled steel plates in this experiment is shown in Fig. 1.

Mechanical properties were determined from transverse tensile specimen (8.5 mm in diameter) and longitudinal Charpy specimen (2 mm V notch) machined from the center of plates. The microstructures were characterized by optical metallography of longitudinal section, and ferrite grain size was measured by means of linear analysis method. The distribution of precipitates was examined by transmission electron microscopy (TEM).

Nb compound was analyzed by using electrolysis method employing 10 % acetylene acetone-1 % tetramethyl ammonium chloride-methanol solution. Soluble Nb was analyzed by measuring Nb content in the solution filtered through 0.2 µm meshed filter.¹⁰⁾

And further, in order to study a change in recrystallization process with hot rolling in HCR process, Nb6 and 10C4 steels were directly quenched after rolling with the rolling finish temperature of 800°C, and the cross section of them was cut, polished, and etched by 20 % nitric acid solution for 2 min, then the ratio of recrystallized region was measured.

2.2. Interrupted Compression Test

The chemical composition of steels used is listed in Table 2. Though carbon and nitrogen contents of Nb5 are extremely lower than that of Nb4, it was prepared to study the effect of Nb in solid solution on the recrystallization process. As the solution temperature of Nb(CN) for Nb5 is calculated to be 744°C from the solubility product,¹¹⁾ the precipitation of Nb(CN) cannot occur at the temperature range above 750°C.

The interrupted compression test which is illustrated in Fig. 2 was carried out by using a hot deformation simulator (THERMECMASER-Z: Fuji Electric Ind. Co., Ltd.) to compare the recrystallization process of these steels by measuring the softening ratio which is represented by Eq. (1).^{12,13)}

$$X_s = (\sigma_m - \sigma_2) / (\sigma_m - \sigma_1) \quad \dots\dots\dots (1)$$

where, σ_m : the maximum stress before unloading
 σ_1, σ_2 : the initial yield stresses during pre-straining and reloading, respectively.

In this testing, the cylindrical specimens, 8 mm in diameter and 12 mm high, were used, and the loading were carried out with strain rate of 1/s.

Table 2. Chemical composition. (wt%)

Steel	C	Si	Mn	P	S	Nb	Sol. Al	N
10C3	0.10	0.29	1.35	0.003	0.003	—	0.028	0.002
Nb4	0.10	0.23	1.34	0.002	0.003	0.030	0.015	0.005
Nb5	0.0006	0.22	1.33	0.001	0.003	0.026	0.016	0.001

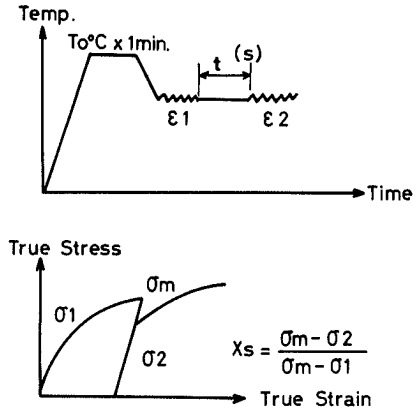


Fig. 2. Schematic representation of interrupted compression testing method.

3. Experimental Results

3.1. Effect of Charging Temperature (T_c) on Strength and Toughness

The effect of charging temperature (T_c) on tensile strength (TS) and brittle-ductile transition temperature (vTs) of Nb1 steel is shown in Fig. 3, comparing with that of 10C1 steel. The vTs for Nb1 and 10C1 steels increases when the charging temperature is raised from 600 to 650°C. In the case of $T_c = 600^\circ\text{C}$, the γ - α transformation has been finished since the Ar_1 temperature of Nb and 10C1 steels were measured to be 609 and 601°C, respectively.

Meanwhile, when the charging temperature is raised from below 600°C to above 650°C, the TS of Nb1 steel increases by 3–4 kgf/mm² than that in case of T_c below 600°C. In case of T_c above 650°C, the γ - α transformation of Nb1 steel has not occurred or completed since the Ar_3 temperature was measured to be 671°C. But TS of 10C1 steel shows constant with no dependency of T_c temperature. Fig. 4 shows the change in microstructure for Nb1 and 10C1 steels with T_c temperature. Ferrite grain size of both steels in case of $T_c = 1000^\circ\text{C}$ is larger than that in case of ambient T_c temperature. And further Nb1 steel shows inhomogeneous microstructure but 10C1 steel shows homogeneous one.

In this way, the change in mechanical property and microstructure of HCR processed steels is greatly depended upon the charging temperature T_c , and the dependency is most steep with T_c around Ar_1 .

3.2. Effect of Heating Temperature (T_h) on Strength and Toughness

It is observed that the mechanical properties and microstructure of HCR processed Nb steel change steeply with T_c . It is reasonable to think that this change is due to the precipitation as well as the solu-

tion of Nb during the cooling process after solidification and the heating process before rolling. Therefore, the effect of heating temperature (T_h) on the relation between TS and vTs of Nb steel was examined. The result is shown in Fig. 5 with the reference of carbon steel 10C. 10C steel does not show much change in the relation between TS and vTs with heating temperature (T_h), but Nb steel shows a change in the relation with the change of T_c . As a result, the following description focuses on Nb steel. In case of T_c , ambient temperature, TS of Nb steel increases and the vTs shifts to higher temperature with a rise in T_h . In case of T_c , 900°C, the TS increases but the vTs does not show much change with a rise in T_h . And in case of T_c , 1 000°C, the

TS shows high independently of T_h but the vTs shifts to lower temperature with a rise in T_h . Moreover, even when T_h is 1 200°C where Nb precipitates are considered to dissolve completely in austenite according to the calculation of solubility products,¹¹⁾ TS of HCR processed material with the charging temperature above Ar_1 keeps still higher by about 2 kgf/mm² than that of conventional one.

Fig. 6 shows the change in microstructure with T_h , where T_c is 1 000°C. These microstructures consist of mix-sized grains of ferrite and pearlite, but the microstructures change more homogeneous and finer with a rise in T_h . This change well explains the improvement in vTs .

3.3. Effect of Rolling Start Temperature (T_s) on Strength and Toughness

In case of T_c above Ar_3 , the relation between TS

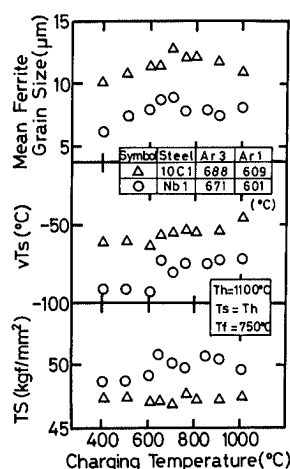


Fig. 3. Effect of charging temperature (T_c) on mechanical properties of 10C1 and Nb1 steel.

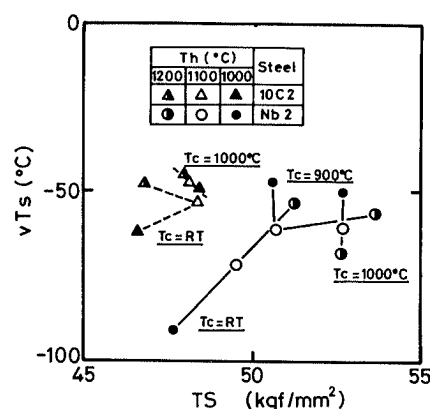
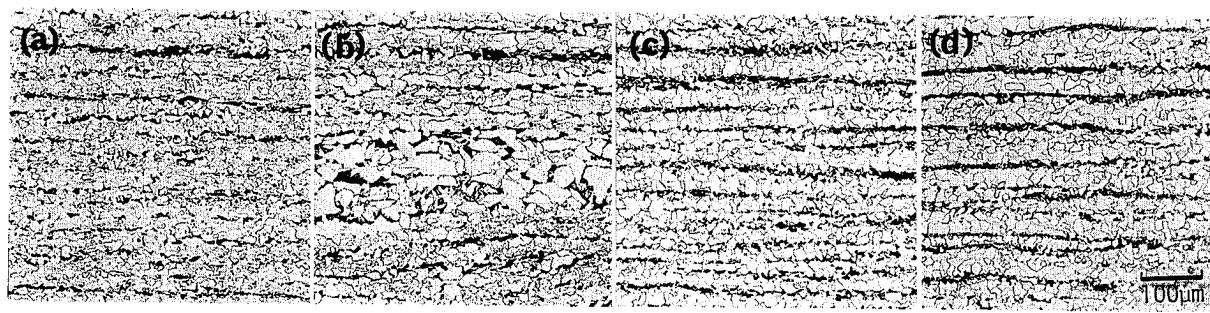
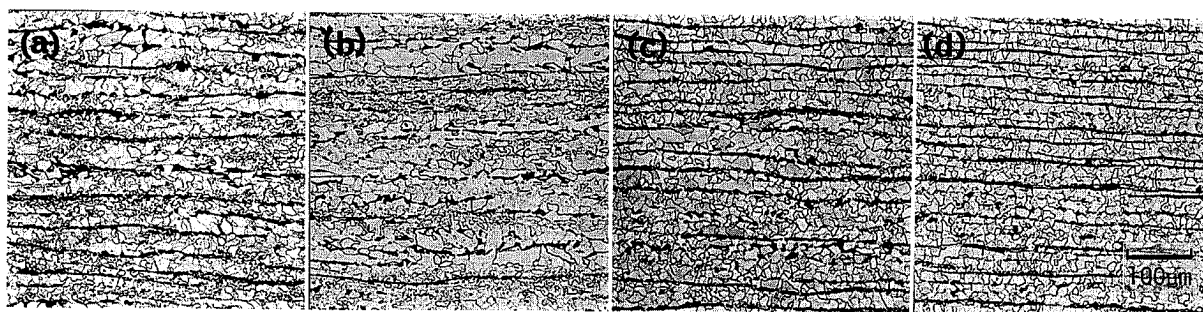


Fig. 5. Effect of heating temperature (T_h) on TS - vTs balance of 10C2 and Nb2 steels.



(a) $T_c = RT$: Nb1, (b) $T_c = 1\ 000^\circ\text{C}$: Nb1, (c) $T_c = RT$: 10C1, (d) $T_c = 1\ 000^\circ\text{C}$: 10C1
 $T_h = 1\ 100^\circ\text{C}$, $T_s = T_h$, $T_f = 750^\circ\text{C}$

Fig. 4. Change of optical microstructures of 10C1 and Nb1 steels with T_c .



(a) $T_h = 1\ 200^\circ\text{C}$: Nb2, (b) $T_h = 1\ 000^\circ\text{C}$: Nb2, (c) $T_h = 1\ 200^\circ\text{C}$: 10C2, (d) $T_h = 1\ 000^\circ\text{C}$: 10C2
 $T_c = 1\ 000^\circ\text{C}$, $T_s = T_h$, $T_f = 750^\circ\text{C}$

Fig. 6. Change of optical microstructures of 10C2 and Nb2 steels with T_h .

and vTs of HCR processed Nb steel is improved with a rise in T_h . This suggests that hot working at higher temperature region has good influence on the microstructure. Fig. 7 shows the effect of rolling start temperature (T_s) on TS and vTs for Nb steel. In this experiment, the rolling finish temperature is raised to 780°C to deform only austenite phase, the meaning of which is mentioned later.

In CCR process, TS increases and vTs shifts to higher temperature with a rise in T_h due to the increase in the amount of Nb in solid solution, vTs is improved with a decrease in T_s .

On the other hand, in HCR process, there is no change in TS with T_h , but vTs is improved with a rise in T_s until 1 100°C.

Fig. 8 shows the change in microstructure for HCR processed Nb steel with various rolling start temperature, where T_c is 1 000°C. They show mixed size of ferrite and pearlite grains, and the area of mix-sized grains is increased with a decrease in T_s . Especially in case of rolling start temperature below 1 000°C, the microstructure shows mixed size of ferrite and pearlite grains and it seems that the inhomogeneous structure has been transformed from a large austenite grain.^{14,15)}

As mentioned above, TS and vTs changes depending upon the rolling start temperature in spite of the same austenite grain size before rolling. So, it is suggested that the inhomogeneity in microstructure and the deterioration of vTs might be originated from not only γ - α transformation process but also recrystallization process due to hot rolling condition.

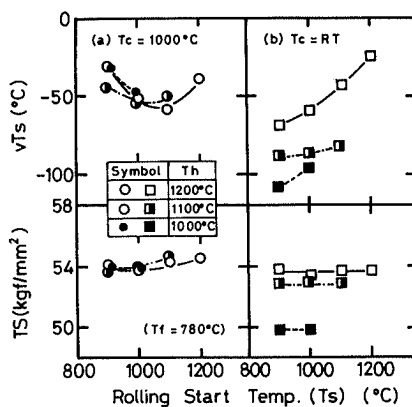


Fig. 7. Effect of rolling start temperature (T_s) on mechanical properties of Nb3 steel.

3.4. Difference in Recrystallization Process between HCR and CCR

Fig. 9 shows the effect of rolling start temperature and rolling reduction on the ratio of recrystallized region for HCR processed Nb6 and 10C4 steels, where T_c 1 000°C, T_h 1 100°C, and T_f 800°C.

The ratio of recrystallized region for 10C4 steel increases with a rise in rolling start temperature and with increase in rolling reduction. On the other hand, the recrystallization in Nb6 steel scarcely occurs in case of the rolling start temperature below 1 000°C, but it occurs in the rolling start temperature of 1 100°C. It is shown that the increase in rolling reduction promotes the recrystallization. Fig. 10 shows some example of the etched cross section for Nb6 steel. The initiation site of recrystallization greatly depends upon the solidification structure. The initiation has turned out to be at the region of equiaxial structure in the center of ingot, and then it propagates outwards to the region of columnar structure as the rolling temperature is raised, or the rolling reduction is increased. In the outer columnar structure, the recrystallization initiates at the grain boundary of columnar structure. This indicates that the strain is concentrated in the vicinity of grain boundary when large austenite grain is hot worked.

In order to compare the recrystallization process in HCR process with that in CCR process, the interrupted compression testing is carried out by using a hot deformation simulator. As shown in Fig. 2, the specimen heated up to 1 350°C for 1 min and 1 100°C

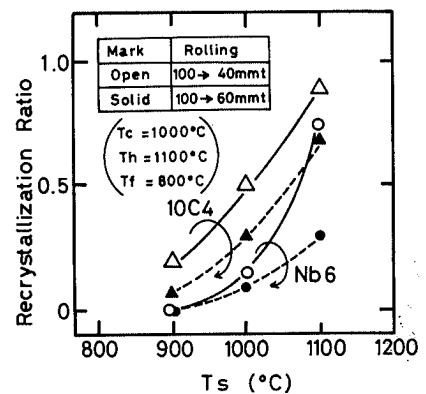


Fig. 9. Effect of rolling start temperature and rolling reduction on recrystallization ratio of 10C4 and Nb6 steels in hot charge rolling.

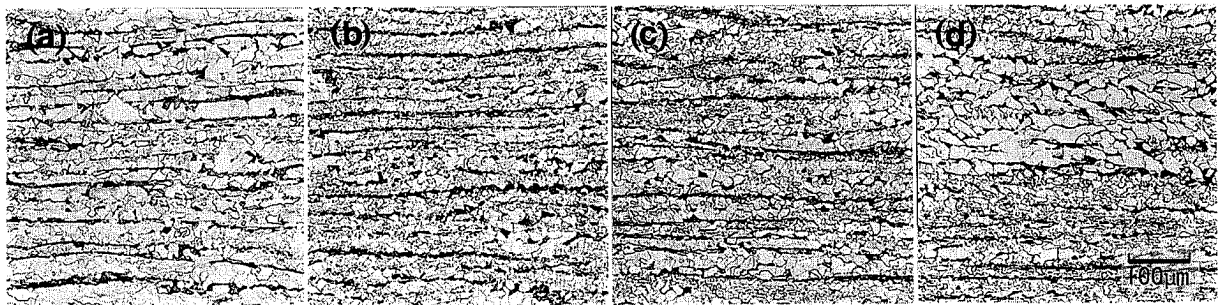
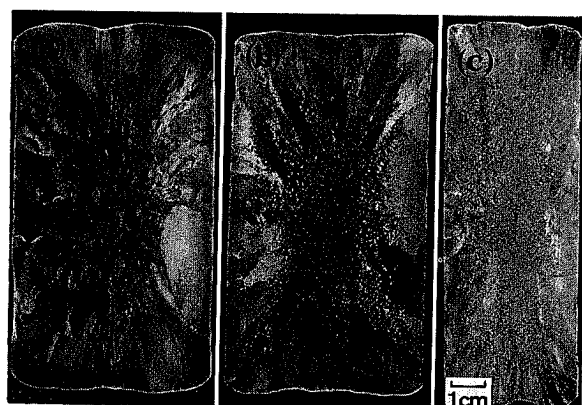


Fig. 8. Change of optical microstructures of hot charge rolling processed Nb3 steel with T_s .
 $T_c = 1\,000^\circ\text{C}$, $T_h = 1\,200^\circ\text{C}$, $T_f = 780^\circ\text{C}$



(a) $T_s=900^\circ\text{C}$
 (b) $T_s=1100^\circ\text{C}$: 100 → 60 mm
 (c) $T_s=1100^\circ\text{C}$: 100 → 40 mm
 $T_c=1000^\circ\text{C}$, $T_h=1100^\circ\text{C}$, $T_f=800^\circ\text{C}$

Fig. 10. Change in recrystallization ratio of Nb6 steel with rolling start temperature and rolling reduction.

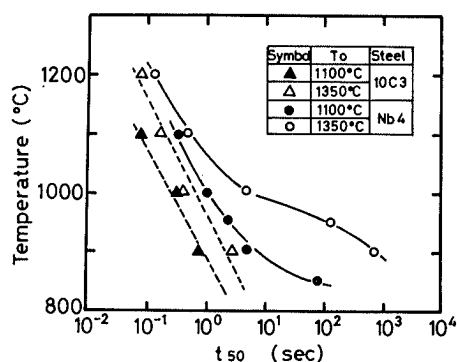


Fig. 11. Relation of half softening time, t_{50} , vs. deformation temperature for 10C3 and Nb4 steels.

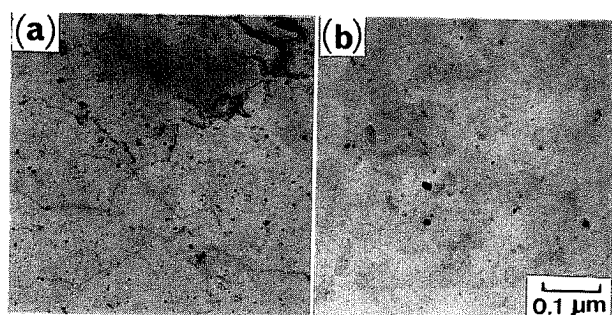
for 1 min to simulate HCR process and CCR process, respectively. Then the austenite grain shows about $900\text{ }\mu\text{m}$ in diameter for 1350°C heating and about $100\text{ }\mu\text{m}$ in diameter for 1100°C heating. In Nb4 steel, it is calculated from solubility products¹¹ that Nb(CN) precipitates dissolves completely in austenite at 1350°C and the 70 % of them dissolves at 1100°C . The deformation is carried out at the temperature from 850 to 1200°C . Fig. 11 shows the relation between t_{50} and deformation temperature for these steels in the case of the prestrain 0.69. Here, t_{50} is the interrupted time when the softening ratio reaches 0.5.

The t_{50} curves against deformation temperature for both steels are retarded with a rise in heating temperature. Especially in the case of Nb4 steel, the softening is retarded remarkably at the temperature range below 1000°C . This method is an indirect method to estimate the recrystallization process. But the result well corresponds with the result in Fig. 6.

4. Discussion

4.1. Strength of HCR Processed Nb Steel

Solution temperature of Nb(CN) in Nb2 steel is calculated to be 1160°C according to Irvine's solubility products,¹¹ but TS of HCR processed Nb steel



(a) $T_c=1000^\circ\text{C}$ (b) $T_c=RT$
 $T_h=1200^\circ\text{C}$, $T_f=750^\circ\text{C}$

Fig. 12. Comparison of Nb(CN) particles in hot charge rolling processed and conventional rolling processed Nb2 steel.

keeps higher by 2 kgf/mm^2 than CCR processed one even in case of T_h is 1200°C . Fig. 12 is TEM micrographs showing precipitates in HCR processed steel compared with CCR processed one. It is observed that row precipitation together with homogeneous precipitation occurred in HCR process, and the mean diameter of them is measured to be $27\text{ }\text{\AA}$. The diffraction pattern from these precipitates is shown in Fig. 13. The pattern is only one pair of spots, but correspond to interplanar spacing of Nb(CN).

In CCR process, fine particles precipitate in addition to coarse rounded particles whose diameter is about $100\text{ }\text{\AA}$. The total number of them are less than that in HCR process. The mean diameter of the precipitates in CCR processed steel is $40\text{ }\text{\AA}$. According to Kelly and Nicholson,¹⁶ it is calculated that the internal stress due to a misfit in $\alpha\text{-Fe}$ matrix reaches maximum with the size ranging from 20 to $30\text{ }\text{\AA}$.¹⁷ This calculation suggests that increase in finely precipitated particles results in the rise in TS of HCR processed Nb steel in such a manner as HCR processed Ti bearing steel.⁵ Fig. 14 shows the relation between TS and $\text{Sol.Nb}/\text{Total Nb}$ for Nb3 steel shown in Fig. 9, but the data points are shown only in case of $T_s=T_h$ in order to eliminate the effect of rolling condition on TS .

It is required that the precipitation hardening caused from Nb(CN) precipitation after rolling is taken into consideration since these steels were air cooled after rolling. But in HCR process TS and Sol.Nb do not change so much even when the heating temperature was raised from 1000 to 1200°C , nevertheless, in CCR process they increase with a rise in heating temperature. And when the heating temperature is raised up to 1200°C , the difference in TS between HCR and CCR becomes to be negligible. On the contrary, it is illustrated in Fig. 5 that there is still a difference in TS between HCR and CCR even when the heating temperature was raised up to 1200°C . It is reasonable to think that this difference may originate from the shortage of heating time for the precipitates to dissolve, because the heating time is only 1 h, and the Nb content of Nb2 steel shown in Fig. 5 was higher than that of Nb3 steel in Fig. 7. The difference in TS will be negligible if it is heated

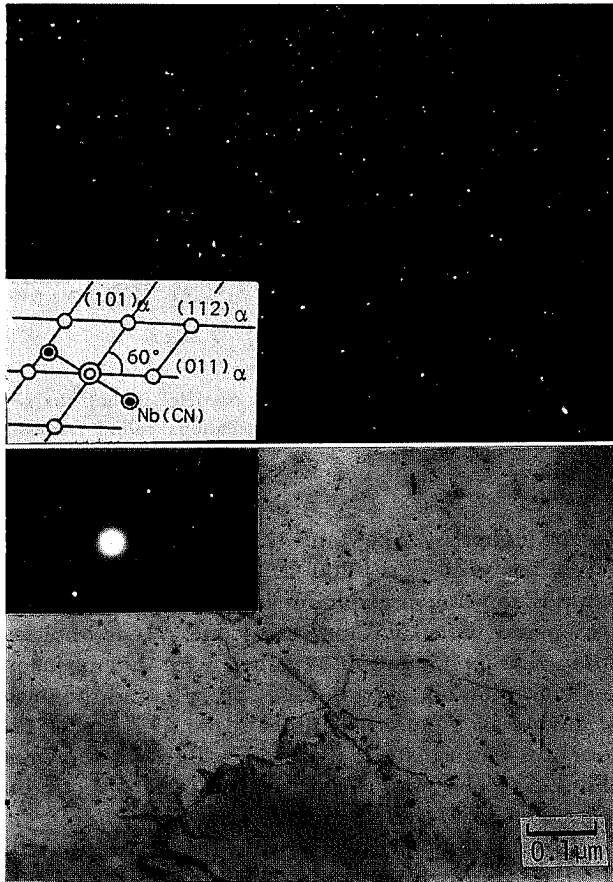


Fig. 13. Nb(CN) particles in hot charge rolling processed Nb3 steel

for enough long time and at enough high temperature for Nb(CN) precipitates to dissolve. Fig. 14 suggests that even when Nb steel is rolled in CCR process, the similar precipitation to that in HCR must be attained. The increase in TS of HCR processed Nb steel with T_c above A_{r1} appears to come from the precipitation hardening in ferrite matrix after γ - α transformation due to the excess amount of Nb in solid solution than that with T_c below A_{r1} . On the contrary, it was reported that TS of HCR processed Nb steel with T_c above A_{r1} decreased with increase in heating time, but this phenomenon is consistent with the result shown above. Because the decrease in TS with increase in heating time came from the precipitation of Nb(CN) in austenite which have a dull effect on the hardening. So, in order to utilize the increase in TS of Nb steel through HCR process, it is required that the cast slabs must be charged to furnace with the charging temperature above A_{r1} . If the heating before rolling is carried out for so long time like plate rolling, then it is required that they must be charged to a furnace with the charging temperature above 900°C and heated at quite high temperature even in HCR process in order to keep the amount of Nb in solid solution.

4.2. Toughness of HCR Processed Nb Steel

Fig. 15 shows the Arrhenius plot of the reciprocal of half softening time, $1/t_{50}$, vs. deformation temperature in Kelvin, T , assuming that the recrystallization

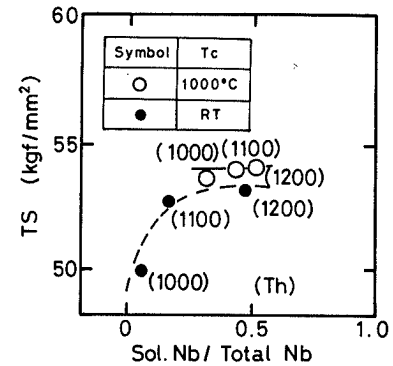


Fig. 14. Relation between TS and Sol. Nb/Total Nb of Nb3 steel in hot charge rolling and conventional rolling processes.

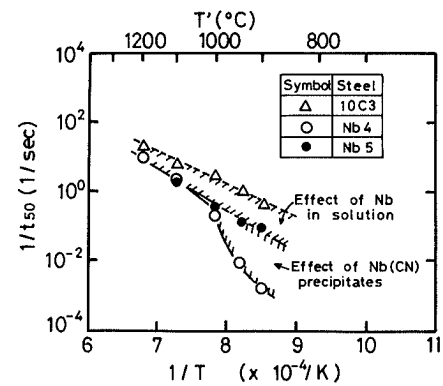


Fig. 15. Arrhenius plot of the reciprocal of half softening time, $1/t_{50}$, vs. deformation temperature in Kelvin, T .

process is thermal activation process. By using this result, an apparent activation energy, Q , is measured according to the following equation (2).

$$1/t_{50} = A \exp(-Q/RT) \quad \dots\dots\dots(2)$$

Here, R : Gass constant

A : a constant.

The activation energy for Nb and 10C3 steels is 23.8 and 20.7 kcal/mol, respectively. The increase in activation energy due to niobium addition is considered to be caused from Nb in solid solution. The activation energy for Nb4 steel is 86.7 kcal/mol with the deformation temperature below 1000°C. This value is nearly equal to that of Nb to diffuse in γ -Fe which is 82.3 kcal/mol. This result indicates that the recrystallization process is governed by the precipitation of Nb(CN) on dislocations and lattice defects introduced by hot deformation in such a manner as CCR process.^{9,14,18)} That is to say, the difference in recrystallization process between Nb4 and Nb5 below 1000°C may originate from the difference in precipitation process of Nb(CN), and the difference between Nb5 and 10C3 may originate from the difference in the presence of Nb in solid solution. In HCR processed Nb steel with T_c above A_{r1} , it is shown that the recrystallization temperature is raised upward above 1000°C due to the great amount of Nb in solid solution and large austenite grain size succeeded from solidification. It was shown in Fig. 5 that the tough-

ness was not improved by rolling with the T_s below 1 000°C since the recrystallization did not start at this deformation temperature range and the refinement of austenite grain due to recrystallization did not occur. But when the hot rolling on the first stage was operated sufficiently at the temperature range above 1 000°C in order to refine the large austenite grain and then done the low temperature range of austenite on the second stage, the toughness would be improved. But, when the hot rolling at too high temperature range above 1 000°C was done, the toughness was deteriorated in opposition. Because the recrystallized austenite grain size increase with a rise in the deformation temperature. Thus, a rolling start temperature of 1 100°C is the lower limit of recrystallization, the vT_s becomes optimum. But this favorable temperature range of deformation depends upon the amount of deformation reduction, and the temperature range is shifted to lower temperature with increase in deformation reduction as shown in Fig. 9. When such a large austenite grain is deformed below its recrystallization temperature, ferrite grain grows large since the number of ferrite grains nucleated within prior austenite grains is not sufficient enough for the refinement, and it results in the deterioration of toughness.

5. Summary

The factor influencing on the strength and the toughness of HCR processed Nb steel with the charging temperature above A_{r1} is investigated, making clear the metallurgy during HCR process. The main results obtained are as follows.

(1) The change in strength and toughness of HCR processed steels greatly depends upon whether γ - α transformation has been completed or not. In case of charging temperature above A_{r1} , the toughness is inferior to that of CCR processed one, and Nb steel shows an increase in strength.

(2) The ratio of Sol. Nb/Total Nb ranges from 0.3 to 0.2 in HCR process with the charging temperature above A_{r1} . The ratio is similar to that in CCR process at high heating temperature for example 1 200°C, and the increase in the ratio results in the increase in strength.

(3) The deterioration in toughness in HCR processed Nb steel with the charging temperature above A_{r1} is attributed to the grain coarsening due to the suppression of recrystallization which is caused by a large amount of fresh precipitation of Nb.

(4) In order to improve the balance between strength and toughness, the first stage rolling should refine the large austenite grain succeeded from solidifi-

cation. It becomes clear that the favorable rolling temperature range is around 1 100°C.

(5) The recrystallization process of large austenite succeeded from solidification during hot rolling depends upon the solidification structure, and the recrystallization is initiated at the region of equiaxial structure at first, and then it propagates outwards to the region of columnar structure as the rolling temperature is raised, or the rolling reduction is increased.

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