Effects of Cooling Rates after Hot Rolling on the Planar Anisotropy of Extremely Low Carbon Niobium Cold Rolled Sheets*

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Synopsis

The effects of the cooling rates after hot rolling on the planar anisotropy of cold rolled Nb steel sheets with extremely low C ($C \sim 0.004\%$, $Nb \sim 0.12\%$) were examined. The specimens were hot rolled to 3.2 mm and cooled variously, i.e., quenching in water, rapid cooling under water spray, air cooling, or furnace cooling, then cold rolled to 0.8 mm, and annealed at 780°C for 5 hr.

The direction of earing formed upon conical cup test varied from diagonal to longitudinal-transverse in accordance with the decrease of cooling rate after hot rolling.

To clarify the phenomenon, specimens were left in air for several different periods of time after hot rolling and quenched in water. It was found then that the specimens quenched from γ state gave rise to earing in diagonal directions and those quenched from α in longitudinal-transverse directions, and that the annealing texture of the sheets earing diagonally was $(554) [22\bar{5}]$ and those earing longitudinal-transversely was $(111) [\bar{1}10]$ plus $(111) [11\bar{2}]$.

It was concluded that the difference of earing direction can be attributed to the difference of texture formation which is, in turn, affected by the particle size of Nb carbonitride precipitated in the hot rolled sheet.

I. Introduction

The effects of Nb on the properties of mild steel have in the main been recognized by the hardening effect due to its minute precipitates or solid solution for rendering the steel of a high strength.

However we find interesting data from old literatures. According to an American patent by F. M. Becket and R. Franks¹⁾ of 1941, for example, an $0.10\% \text{C}$, $0.65\% \text{Nb}$ hot rolled steel shows a yield strength of 15 kg/mm^2 , and according to the data of strain aging by C. A. Edwards, D. L. Phillips and H. N. Jones,²⁾ an 0.025% or less C, $0.36\% \text{Nb}$ steel does not show any strain aging. W. B. Morrison³⁾ has shown that the strain aging at 100°C of an $0.15\% \text{C}$, $0.005\% \text{N}$ and $0.10\% \text{Nb}$ steel decreases considerably in either hot rolled or normalized condition. Judging from these data, we expect that a sheet steel with good deep drawability can be manufactured from a low C and high Nb steel.

Many reports have been published concerning good drawability of sheet steels⁴⁻¹⁵⁾ treated by carbonitride forming elements, and some of them describe the texture of cold rolled and annealed sheets in detail.

For example, R. H. Goodenow and J. F. Held⁷⁾ reported appearance of 6 ears in a Ti stabilized steel sheet upon cup testing, concluding its main texture to be $(111) [12\bar{3}]$ for high cold reductions; this will be discussed later with regard to the Nb stabilized steel sheet.

T. Matsuoka and M. Takahashi⁸⁾ studied the low C, Ti steel and reported the change of annealing textures and of precipitated particle size with soaking temperatures before hot rolling; this also will be discussed with regard to the low C, Nb steel.

O. Akisue and K. Takashina¹²⁾ studied the rolling and recrystallization textures of low C, Nb steel sheets which were hot rolled by hot strip mill of actual works.

In the present investigation, the effects of the hot rolling condition on the earing behaviors of cold rolled and annealed sheets of extremely low C, Nb steel were studied.

II. Specimens and the Preliminary Experiment about Cooling Rates after Hot Rolling

1. Experimental Procedure

Three steels were melted in a high frequency induction furnace, deoxidized with Al, Al-Si, or Si, then cast in air into 100 kg ingots. The chemical compositions are given in Table 1. These ingots were forged to slabs of $15 \times 90 \times 150 \text{ mm}$ at 1200°C , soaked at 1300°C for 15 min and hot rolled to a finishing temperature of 900° , 850° or 800°C in one pass. A 2-high research hot rolling mill of diameter 200 mm, roll barrel 200 mm, rolling speed 10 m/min was used. The finishing temperatures were recorded by a radiation pyrometer appropriately calibrated with Pt-Pt Rh thermocouple. After hot rolling, the specimens were cooled variously, i.e., quenched in water (*W.Q.*), rapidly cooled under water spray (*R.C.*), cooled in air (*A.C.*), and cooled in furnace (*F.C.*); all except *W.Q.* being slowly cooled in a heat insulating sand bath below 500°C . The average cooling rates after respective finishing temperatures

Table 1. Chemical compositions of specimens (wt%)

Specimen	Type of deoxidation	C	Si	Mn	P	S	Tot. Nb	sol. Nb	sol. Al	insol. Al	sol. N	insol. N
1	Al	0.0059	0.003	0.41	0.003	0.005	0.12	0.065	<0.002	0.003	0.0036	<0.001
2	Al-Si	0.0041	0.020	0.13	0.009	0.007	0.11	0.051	0.003	0.003	0.0049	<0.001
3	Si	0.0037	0.140	0.22	0.010	0.007	0.14	0.096	—	—	0.0028	0.001

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are shown in Table 2.

The hot rolled sheets were pickled, cold rolled from 3.2 to 0.8 mm, annealed at 780°C for 5 hr at a rate 400°C/hr to 400°C then 25°C/hr to 780°C, and cooled in furnace.

The sheets, both as hot rolled and as cold rolled and annealed (this will be called simply the annealed hereafter), were examined metallographically (optical microscopy, X-ray diffraction for textures) and by hardness, with tensile tests and conical cup tests added for the annealed sheets.

2. Experimental Results

The hardness of hot rolled sheets increased with increasing cooling rate. Although the annealed

sheets did not show any particular hardness change with cooling rate, the total elongation increased as the cooling rate decreased, whereby an interesting change was observed in the r value and the flange shape of conical cup.

In Fig. 1, planar anisotropy of r value and flange shape of conical cup after cup testing are shown in each quadrant of the circle with respect to the cooling rates. The r values in intermediate directions were calculated on the longitudinal, transverse and diagonal direction values using Hill's method.¹⁶⁾

The following facts have become clear with this experiment.

(1) The earing of conical cup changes according to the cooling rates after hot rolling, signifying the change of planar anisotropy in the annealed sheet.

(2) The directions of earing of Specimens 1 and 2 are changed from diagonal to longitudinal-transverse as the cooling rate decreases.

(3) There is no direct correlation between planar anisotropy of r value and flange shape of conical cup.

(4) The flanges having 6 ears (in longitudinal and diagonal directions) are found in Specimen 3. The flange shape and the planar anisotropy of r value do not correspond to each other in this case, as Hill's method fails to apply to complicated cases as will be shown later in Fig. 7.

(5) As is shown in Table 3, specimens finished at 800°C show better $\bar{r}$ values but no clear tendency is obtained concerning the cooling rate.

	Finishing temperature (°C)	Average cooling rate (°C/sec)
Rapid cooling	900	40.2
	850	45.3
	800	53.6
Air cooling	900	10.3
	850	11.4
	800	11.1
Furnace cooling	900	0.34
	850	0.36
	800	0.40

Fig. 1. Changes in r value and flange shape of conical cup of cold-rolled and annealed sheets with the after hot-rolling cooling rates (Specimens were annealed at 780°C for 5 hr)

3. Precipitation of Nb Carbonitride and X-ray Diffraction Intensity

In Fig. 2, the relation between the cooling rate after hot rolling and the quantity of Nb carbonitride as determined by the insoluble Nb (the residue when dissolved in 6N HCl) is shown. The insoluble Nb of

Table 3. Changes in $\bar{r}$ value of cold-rolled and annealed sheets with the after hot-rolling cooling rates

Specimen	Finishing temperature (°C)	$\bar{r}$			
		Water quenched	Rapid cooled	Air cooled	Furnace cooled
1	900	1.68	1.82	1.92	1.94
	850	1.75	1.89	2.08	1.93
	800	1.75	2.44	1.95	2.24
2	900	1.45	2.14	2.36	1.96
	850	1.76	2.34	2.58	1.94
	800	2.58	2.29	2.35	2.36
3	900	2.27	2.28	2.14	2.16
	850	2.01	2.37	2.11	2.09
	800	2.36	2.40	2.21	2.05

Fig. 2. Changes in insoluble Nb with the after hot-rolling cooling rates

hot rolled sheets increases as the cooling rate decreases, and in the case of R.C. it decreases as the finishing temperature becomes lower. The insoluble Nb increases by the precipitation of Nb carbonitride during annealing, becoming almost constant irrespective of the hot rolling condition.

The X-ray diffraction intensities of the annealed sheets of Specimen 2 are shown in Fig. 3. The (222) intensities become maximum and (200) intensities minimum for A.C.

The size of Nb carbonitride as well as of grains of the hot rolled sheets change with the hot rolling condition. In Photo. 1, electron micrographs of Nb

Fig. 3. Changes in X-ray diffraction intensities of cold-rolled and annealed sheets with the after hot-rolling cooling rates (Specimen 2)

Photo. 1. Electron photomicrographs of Nb carbonitrides in hot-rolled sheets (Specimen 2, carbon extraction replica) ($\times 60\,000$) ($\times 5/6$)

carbonitride of Specimen 2 are shown for various hot rolling conditions. It will be seen that, although the particles are fine and uniformly distributed in *W.Q.* specimens, they get coagulated more as the cooling rate becomes slower and hot rolling finishing temperature higher. Thus, the size of particles appears to depend on the time in which the specimen is held through high temperatures.

Transmission electron micrographs of Nb carbonitride of annealed specimens are shown in Photo. 2. The shape of precipitates for the finishing temperature of 800°C is somewhat different from that for 850°C and above, where angular precipitates are found. This is especially clear with the *W.Q.* or *F.C.* specimens.

In Fig. 4, the recrystallization curves by hardness measurement are shown (heating rate 25°C/hr). Very little change is found among these curves for the same cooling rate, but some change is found in the recovery stage for specimens cooled differently from the same finishing temperature, especially for 900° and 850°C: the recovery and softening proceeds more as the cooling rate becomes slower. This suggests that fine Nb carbonitrides in hot rolled sheets can coagulate and grow during annealing after cold rolling to influence the texture at recovery and recrystallization.

From those experiments, it was concluded that the planar anisotropy in r value and flange shape of conical cup of annealed sheet change with the finishing temperature of hot rolling, presumably being affected by the quantity and distribution of Nb carbonitride precipitates.

III. Change in Planar Anisotropy of Annealed Sheets with the Finishing Temperature of Hot Rolling and the Subsequent Cooling Time before Water Quench

1. Experimental Procedure

To see the change of earing with the quantity and distribution of Nb carbonitride, Specimen 2 (Al-Si deoxidized) was hot rolled with the finishing temperatures of 900°, 850° and 800°C, and left in air for several different periods of time before quenched in water. The sheets were cold rolled and annealed as before.

2. Test Results

The change in planar anisotropy in r value as calculated by Hill's method and in flange shape of conical cup of annealed sheets are shown in Fig. 5. The earing upon conical cup test changes from diagonal to longitudinal-transverse as the time before water quench increases, but no clear correlations are found between planar anisotropy of r value and flange shape of conical cup as before.

In Fig. 6, planar anisotropies of r values, one as calculated by Hill's method and the other as measured actually, are shown with flange shape of conical cup (Specimen 3, hot rolled to 850°C, water quenched immediately, cold rolled, annealed as

Photo. 2. Transmission electron photomicrographs of Nb carbonitrides in cold-rolled and annealed sheets (Specimen 2) ($\times 15\,000$) ($\times 4/5$)

Fig. 4. Changes in recrystallization (hardness reduction) curves on annealing with finishing temperature and the after hot-rolling cooling rates (Heating rate, 25°C/hr)

usual). It will be noted that there is a definite correlation between the flange shape and the planar anisotropy of r value as measured but not with the calculated r values. Therefore it is concluded that

Fig. 5. Changes in flange shape of conical cup and r value of cold-rolled and annealed sheets with the time of air cooling (sec) before water quench after hot rolling (Specimen 2)

Fig. 6. Flange shape of conical cup and planar anisotropies of r value (Specimen 3, hot rolling finishing temperature 900°C, quenched in water, cold-rolled and annealed at 780°C for 5 hr)

complex planar anisotropy of r value like this case cannot be calculated by Hill's method. The earing changes from diagonal to longitudinal-transverse after 10, 5 and 0 sec of air cooling time for the specimens finished at 900°, 850° and 800°C, respectively.

In Fig. 7, the cooling curves after hot rolling are shown. There are horizontal parts near 800° to 815°C. The specimens that gave rise to diagonal earing were those that were quenched above this temperature, which was ascertained to be the α - γ

Fig. 7. Cooling curves after hot rolling (Specimens were hot-rolled, air cooled and water quenched.)

transformation point by dilatometry test using a 2.0 mm ϕ specimen prepared from hot rolled sheet. It is concluded therefore that the planar anisotropy changes according to whether the transformation point is passed during the air cooling after hot rolling or not.

3. Precipitation of Nb carbonitride

In the experiment described above, it was made

Photo. 3. Electron photomicrographs of Nb carbonitrides in hot-rolled sheets as quenched in water after air cooled for various periods of time (Specimen 2, carbon extraction replica) ($\times 60\,000$) ($\times 4/5$)

Fig. 8. Relation between insoluble Nb in hot-rolled sheets and quenching temperature after hot rolling

clear that the diagonal earing should be ascribed to Nb carbonitride precipitation occurring only in γ state after hot rolling, whereas the longitudinal-transverse earing to precipitation in both γ and α . This has a close relation with the preliminary experiment: in the specimens cooled rapidly after hot rolling, precipitation proceeded mainly in γ to become fine and uniformly distributed, whereas in slowly cooled ones, precipitation proceeded in a nearly equilibrium, the precipitates growing and coagulating.

The electron photomicrographs of Photo. 3 as well as the relationship between insoluble Nb in hot rolled sheets and quenching temperature after hot rolling, shown in Fig. 8, confirm the above conclusion.

4. Change in Planar Anisotropy of Annealed Sheets with Cooling Time before Water Quench after Hot Rolling

The change of planar anisotropy with differences in prior hot rolling may be ascribed to either of the following two reasons.

(1) On passing the A_3 transformation point during rolling, the texture of hot rolled sheet changes, and so does the texture of annealed sheet.

(2) As the solubility products of Nb carbonitride

in α is different from that in γ , the state of precipitation changes, causing the recrystallization texture to change.

In Fig. 9, the diffraction intensities as hot rolled, as cold rolled and as cold rolled and annealed are shown for the water quenching temperature after hot rolling. It will be seen that generally the intensities are all very weak, and no particular changes are found at around 800°C. Further, as will be shown later, pole figures of hot rolled sheets do not show any fundamental change whether finished above the A_3 point or below it.

With regard to the second factor, the distribution state of Nb carbonitride (the average numbers per unit area and the average particle diameter) was determined by the method of M. F. Ashby and R. Ebeling.¹⁷⁾ In Fig. 10, the average particle diameter and the average number per unit area of each specimen are shown for the finishing temperature. On passing the A_3 transformation point, the average particle size increases while the average number decreases; this is because, as the solubility product of Nb carbonitride in α iron is much smaller than in γ iron, some of the particles are allowed to grow at their previously precipitated sites.

In Fig. 11, changes of earing on conical cup test and in planar anisotropy with average particle size are shown. It will be seen in Fig. 11(a) that the diameter differential $[X-L]$ (X and L being the diameters (mm) of a conical cup measured in diagonal and longitudinal directions, respectively) decreases with the increase of average particle size, becoming zero at the size of ca. 140 Å. In Fig. 11(b), further, the relation of Δr and average particle size is shown ($\Delta r = 1/2 \times (r_0 + r_{90}) - r_{45}$; r_0 , r_{45} and r_{90} are the r

Fig. 9. Changes in X-ray diffraction intensities with quenching temperature after hot rolling (Specimen 2). The designated temperatures denote the finishing temperature, ○ as hot-rolled; ● as cold-rolled; and × as cold-rolled.

Fig. 10. Relations between average particle size and particle number per unit area in hot-rolled sheets and quenching temperature after hot rolling

(a) Variation in the diametric anisotropy of a conical cup (X: the diagonal diameter L: the longitudinal diameter)
(b) Variation in the planar anisotropy ($\Delta r = 1/2 \times (r_0 + r_{90}) - r_{45}$)

Fig. 11. Relations between plastic anisotropies of cold-rolled and annealed sheets and average particle size in hot-rolled sheets

values of longitudinal, diagonal and transverse directions, respectively), where a similar tendency is to be observed. In Fig. 12, the relation between the diffraction intensities and average particle size is shown. There are maxima for (222) diffraction intensity and for the intensity ratio of (222) to (200), and a minimum for (200) diffraction intensity, all at an average particle size of 180 to 200 Å.

5. Textures

In Fig. 13, (200) pole figures of hot rolled, cold rolled, or annealed sheets of Specimens 2 and 3 are shown. Pole figures of series (a) are for the specimens quenched from the finishing temperature of nominally 800°C, the cases which show maximum in the $\bar{r}$ value, whereas those of series (b) and (c) are for those of water quenched after 10 sec and 20 sec of air cooling, respectively, after hot rolling to a finish-

Fig. 12. Relations between X-ray diffraction intensity and average particle size

Fig. 13. Changes in (200) pole figures with the hot rolling conditions

ing temperature of nominally 800°C. As previously noticed, the change in direction of earing is found between (b) and (c).

It is clear from these figures that the textures of hot rolled sheets of specimens (b) and (c) are not much different, and no clear change occurs on passing the A_3 transformation point. The same result was obtained about the cold rolling textures.

The recrystallization textures, however, show a remarkable change; that is, the main texture of (a) and (b) is (554) [225], whereas that of (c) is (111) [110] plus (111) [112]. The main texture of series (d) specimen, which was deoxidized with Si and gave rise to 6 ears on conical cup test, is (554) [225].

IV. Discussions

The precipitation phenomena of Nb carbonitride

are explained generally by the isothermal precipitation curves proposed by J. F. Grey and R. B. G. Yeo,¹⁸⁾ but when stress is imposed during the sheet-making as in the case of hot rolling, the curves would suffer some alterations. Namely, as previously discussed, the fine and numerous particles of Nb carbonitride precipitated in γ state coagulate and grow as the temperature decreases. But after passing the A_3 transformation point, its nominal average particle size becomes smaller, and the number of particle increases as new fine precipitates make their appearances in α . And as the temperature decreases further, they coagulate and grow again, until finally a small number of large particles are remained. These phenomena are explained qualitatively in Photo. 3 and quantitatively in Fig. 10.

The explanation for the change of planar anisot-

ropy of r value with the difference of particle size discussed above is given for the change of particle size by cooling rates of the preliminary experiment.

In Fig. 14, (200) pole figures of an annealed specimen (0.011% C, 0.14% Nb) as quenched from either γ or α state after hot rolling are shown. It will be seen that, when water quenched in γ , the main recrystallization texture is (554) [225], whereas when quenched in α , it is (111) [110] plus (111) [112].

According to H. Abe,¹⁹⁾ the formation of annealing texture of near (111) [112] (that is, (554) [225]) is attributed to the rotation of (112) [110] around $\langle 110 \rangle$ axis and that of (111) [110] is attributed to the rotation of (111) [112] around $\langle 111 \rangle$ axis. In the present study, the annealing texture of specimen quenched in γ after hot rolling appears to correspond to the former (near (111) [112]), and that quenched in α to the latter ((111) [110]), though the details are to be studied further.

P. R. Mould and S. M. Gray¹⁴⁾ studied a low C steel containing Nb and reported that isothermal transformation at a high temperature, or in a simulated high temperature coiling followed by slow cooling, favors the development of higher r value in the cold rolled and annealed sheets, and that the effectiveness of high (simulated) coiling temperatures in promoting high r values is apparently related to the formation of Nb carbonitrides within the size range of 40 to 500 Å. I. F. Hughes and R. C. Hudd¹⁰⁾ applied for a patent to obtain high r values in cold rolled and annealed sheets of Nb-containing steels by such particular hot rolling practices or heat treatments that produce Nb carbides within a preferred size range of 80 to 500 Å. H. Abe and K. Takagi²⁰⁾

showed that the size and distribution of Fe_3C particles in hot rolled sheet influence the annealing texture of an 0.06% C rimmed steel: the hot rolled sheet solution-treated at 920°C for 1 hr and water quenched developed a main texture of (554) [225] after cold rolling and annealing. This tendency was strong when the water quenched hot rolled sheet was tempered at 200°C for 3 hr. They believed that the Fe_3C was dissolved when solution treated, reprecipitating after quenching and tempering as fine Fe_3C precipitates to affect the annealing texture.

In the light of the present results and the previous report, it is suggested that different size and distribution of Nb carbonitride in hot rolled sheet has different effects on the texture (plastic anisotropy) of the annealed sheet, be it of Nb steel, of rimmed steel, or of steels containing Ti or other carbonitride forming elements.

On the other hand, though R. Gillanders and C. Dasarathy²¹⁾ reported that soluble Nb affected hot rolling texture in that (112) [110] became sharper and responsible for the near (111) [112] of annealed texture, no such phenomenon was found in the present study as shown in Fig. 13.

V. Summary and Conclusions

(1) The 100 kg ingots (C ~ 0.004%, Nb ~ 0.12%, deoxidized with Al, Al-Si, or Si) were forged and hot rolled to 3.2 mm with a finishing temperature of 900°, 850° or 800°C. After hot rolling they were cooled variously; water quenched, rapid cooled under water spray, cooled in air, or cooled in furnace. The hot rolled sheets were cold rolled to 0.8 mm and annealed at 780°C for 5 hr. The specimens quenched

Fig. 14. Changes in (200) pole figures of cold-rolled and annealed sheets with hot rolling conditions

after hot rolling showed earing in diagonal directions upon conical cup test. As the cooling rates decreases, the earing showed the tendency to occur in longitudinal-transverse directions.

(2) The specimens that were water quenched from temperatures higher than about 810°C showed earing in diagonal directions, whereas those from temperatures lower than that showed earing in longitudinal-transverse directions. The border temperature was the A_3 transformation point.

(3) Of the two possible reasons to account for the change of earing, *i.e.*, (i) the change of hot rolling texture on passing the A_3 transformation temperature, and (ii) the change of Nb carbonitride particle size due to the change of solubility products from γ to α , the (i) was denied as no fundamental change was found in the texture either as hot rolled or as cold rolled. It was found, however, for the cold rolled and annealed sheets, in those that showed earing in diagonal directions the main texture was (554) [225], whereas in ones that showed earing in longitudinal-transverse directions it was (111) $[\bar{1}10]$ plus (111) [112].

(4) On the other hand, however, electron microscopy study of hot rolled sheets revealed that there was an abrupt growth in the average particle size when the specimen was transformed from γ to α , and that when the average particle size was smaller than *ca.* 140 Å, the earing occurred in diagonal directions whereas when it was larger than 140 Å, the earing was in longitudinal-transverse direction.

(5) It was further found that the diffraction intensity ratio of (222) to (200) of the cold rolled and annealed sheet became the greatest when the particle size in the hot rolled sheet was 180 to 200 Å.

(6) Thus, it was concluded that the difference of earing can be explained by (ii), namely, by the dif-

ference of Nb carbonitride particle size.

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