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Improvement of toughness of low carbon steels containing nitrogen by fine microstructures

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An attempt has been made to improve the toughness of low carbon steels containing total nitrogen from 58 to 150 mass ppm by hot rolling at temperatures lower than the A_1 point (control-rolling). The control-rolled steels had fine microstructures and Charpy impact values higher than 100 J at 273 K. Strain aged steels had Charpy impact values higher than those as control-rolled. The cause is discussed. © *2002 Kluwer Academic Publishers*

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1. Introduction

It is said that nitrogen makes low carbon steels brittle. Charpy impact values of the steels decrease with the increase in total nitrogen contents [1, 2]. The maximum solid solubility of nitrogen in αFe is reported about 0.095 mass% at 858 K [3]. The interstitial nitrogen atoms gather to dislocations, and they cause the strain aging embrittlement of the steels. Otherwise, more nitrogen than the solid solubility limit in αFe forms the iron nitride Fe_4N which supplies the interstitial nitrogen in solid solution during heating. We call nitrogen in Fe_4N soluble nitrogen. If the low carbon steels contain such as titanium, vanadium, aluminum and so on, which form stable nitrides, the steels have no strain aging for the absence of interstitial nitrogen atoms.

In this research work, the low carbon steels which contain total nitrogen contents from 58 to 150 mass ppm, were prepared in a practical direct electric current arc-melting furnace, and hot-rolled after continuous casting, then finally rolled at temperatures lower than the A_1 point, and immediately cooled by water spray. The toughness of the steels was investigated and compared to microstructures, and an attempt was made to improve the toughness of the steels containing high nitrogen contents by fine microstructures.

2. Experimental

The chemical compositions of the low carbon steels as rolled are shown in Table I. Carbon concentrations in these steels are from 0.14 to 0.21 mass% and carbon equivalents Ceq are in a range of 0.38 to 0.48. These carbon equivalents are calculated by Ceq = (C mass%) + (Si mass%)/24 + (Mn mass%)/6 + (Ni mass%)/40 + (Cr mass%)/4 + (Mo mass%)/4 + (V mass%)/14 [4]. These steels were melted in the practical 130,000 kg direct arc-melting furnace and continuously cast in 200 mm thickness × 400 mm width. The ingots were finally rolled at a temperature range of 873–923 K lower than the A_1 point from 24 to 13 mm in thickness, and immediately cooled by water spray. This rolling is called “control-rolling”. The conventional steel was finally rolled at 1073 K from 24 to 13 mm in thickness. Total nitrogen contents and soluble nitrogen in iron nitrides contents were measured by chemical analysis, and those of interstitial nitrogen and carbon were obtained from Snoek peak heights of internal friction obtained by the free decay of a longitudinal resonance vibration [5]. A specimen for the internal friction measurement was 1 mm thickness × 10 mm width × 100 mm length, and its length direction was parallel to a rolling direction. The steel plates were cut 60 mm width × 500 mm × 13 mm thickness for tensile plastic deformation. The length of the plates was parallel to the rolling direction. The steel plates were plastically deformed 1, 2, 3 and 5% respectively, in elongation at room temperature by a tensile machine, and Charpy impact test pieces were cut in an electric spark machine. These Charpy impact test pieces were cut 10 mm height × 10 mm width × 55 mm length with a 45° V-notch 2 mm in depth [6]. The root radius of the V-notch was 0.25 mm. The impact test piece length was parallel to the rolling direction, and the notch was in the thickness direction of the steel plate. The Charpy impact test pieces were strain aged at 523 K for 3.6 ks.

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TABLE I Chemical compositions of test low carbon steels as rolled

Sample number	C	Si	Mn	P	S	Cu	Ni	Cr	Mo	Al	Ti	C_{eq}	O	Total N	Soluble N	N (mass ppm)	C (mass ppm)
	Chemical compositions (mass%)												O,N (mass%)			Interstitial	
	$\times10^{-2}$			$\times10^{-3}$		$\times10^{-2}$				$\times10^{-3}$	$\times10^{-3}$	$\times10^{-2}$		N			
No Ti addition																	
1	14	19	136	19	6	26	13	19	4	15	1	43	12	58	28	0	0
2	15	22	128	20	6	28	7	12	1	1	3	40	44	79	78	18	2.8
H	14	20	131	20	3	31	9	15	1	1	3	40	38	98	95	17	2.7
3	14	21	128	17	5	28	8	11	1	0	1	39	53	117	105	8.9	3.6
4	14	21	124	16	9	32	8	9	1	1	1	38	83	132	126	16	19
Ti addition																	
5	14	20	132	23	7	30	14	20	4	11	13	42	16	62	58	0	0
6	12	25	132	28	8	28	7	29	1	6	20	41	23	73	72	10	3.0
D	13	19	137	16	4	34	8	8	1	2	21	39	33	95	93	0	0
7	14	23	125	19	10	30	8	9	1	5	15	38	38	119	108	20	5.8
S	15	25	130	19	6	28	9	12	1	2	8	41	29	150	129	4.9	3.7
Conventional	21	19	85	20	7	30	9	23	1	1	0	41	102	119	115	22	15

Carbon equivalent $C_{eq} = (C\ mass\%) + (Si\ mass\%)/24 + (Mn\ mass\%)/6 + (Ni\ mass\%)/40 + (Cr\ mass\%)/4 + (Mo\ mass\%)/4 + (V\ mass\%)/14$ [4]. Conventional: Finally rolled at 1073 K.

3. Experimental results

3.1. Total nitrogen

Figs 1 and 2 show relations between Charpy impact values at 273 K and total nitrogen contents in steels. Charpy impact values linearly decrease with total nitrogen contents, and Charpy impact values of the steels with no addition of titanium at the same total nitrogen contents, increase by strain aging, and there is no difference in the aging-induced changing tendency of Charpy impact values of titanium addition steels between as control-rolled and strain aged steels. The 5% strained and unaged steels approach to those as control-rolled. But, compared to the conventional steel, which is finally rolled at 1073 K and has very low impact values, the control rolling improves the toughness of the steels. Figs 3 and 4 show Charpy impact values at 273 K of the as control-rolled, strain aged and strained 5% steels. The Charpy impact values of the steels with no addition of titanium increase with strain aging. This is abnormal in low carbon steels. The reason is discussed later. Otherwise, Charpy impact values of titanium addition steels show small changes with strain aging. This means that there is only small amount of interstitial nitrogen atoms as seen in Table I.

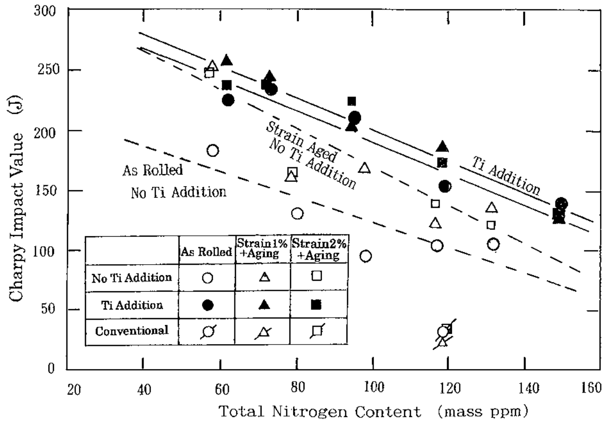


Figure 1 Relation between V-notch Charpy impact values at 273 K and total nitrogen contents in control-rolled, or conventionally rolled steels. Conventional: Final rolling temperature at 1073 K. Strain aging: at 523 K for 3.6 ks.

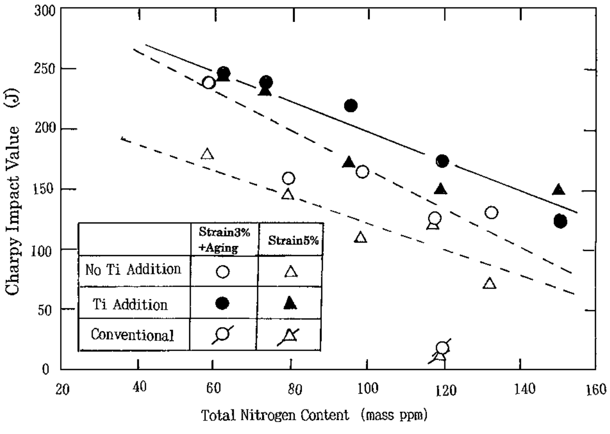


Figure 2 Changes of V-notch Charpy impact values at 273 K with total nitrogen contents in control-rolled, and conventionally rolled steels. Conventional: Final rolling temperature at 1073 K. Strain aging: at 523 K for 3.6 Ks.

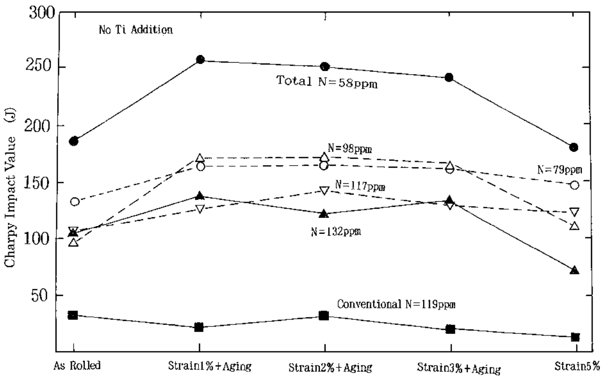


Figure 3 V-notch Charpy impact values of no titanium addition steels as control-rolled, 1% strain + aged, 2% strain + aged, 3% strain + aged and 5% strained. Strain aging: at 523 K for 3.6 ks. Nitrogen content: Total mass ppm. Conventional: finally rolled at 1073 K.

3.2. Soluble nitrogen

Figs 5 and 6 indicate the relations between Charpy impact values at 273 K and soluble nitrogen contents (Fe_4N) in steels. The changing tendencies of Charpy impact values are similar to those in Figs 1 and 2. The

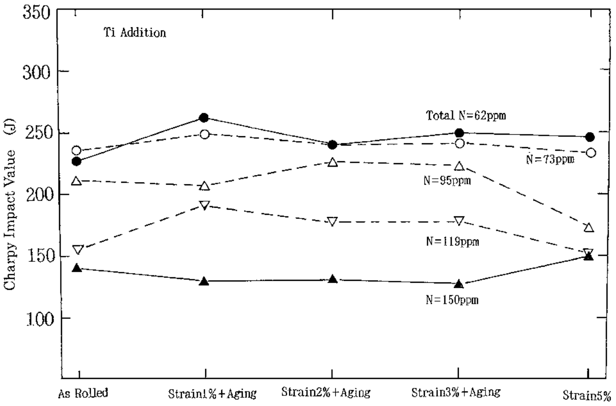


Figure 4 V-notch Charpy impact values in titanium addition steels as rolled and strain aged. Strain aging: at 523 K for 3.6 ks. Nitrogen content: Total nitrogen mass ppm.

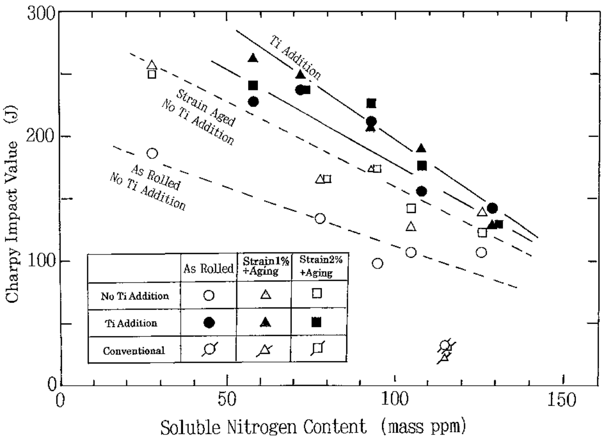


Figure 5 Relation between V-notch Charpy impact values at 273 K and soluble nitrogen contents in as rolled steels. Conventional: Finally rolled at 1073 K.

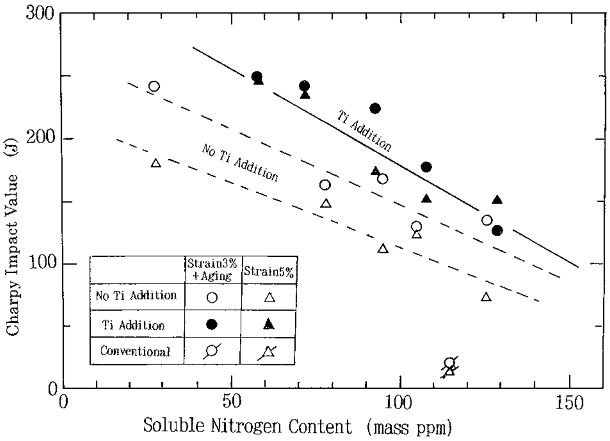


Figure 6 Changes of V-notch Charpy impact values at 273 K with soluble nitrogen contents in as rolled steels. Conventional: Finally rolled at 1073 K.

influence of total nitrogen contents to Charpy impact values is essentially similar to that of soluble nitrogen contents.

3.3. Interstitial nitrogen

Fig. 7 shows changes of Charpy impact values at 273 K with interstitial nitrogen contents. Interstitial nitrogen makes a remarkable decrease in Charpy impact values until 5 ppm, and then reaches a constant load. The fact shows that only small amount of interstitial nitrogen is needed to influence the steel toughness.

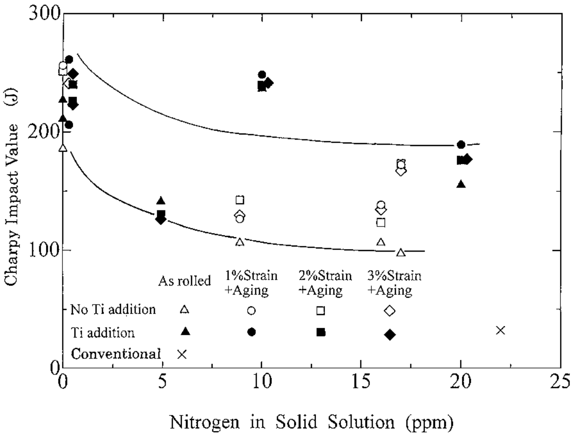


Figure 7 Relation between V-notch impact values at 273 K and interstitial nitrogen contents.

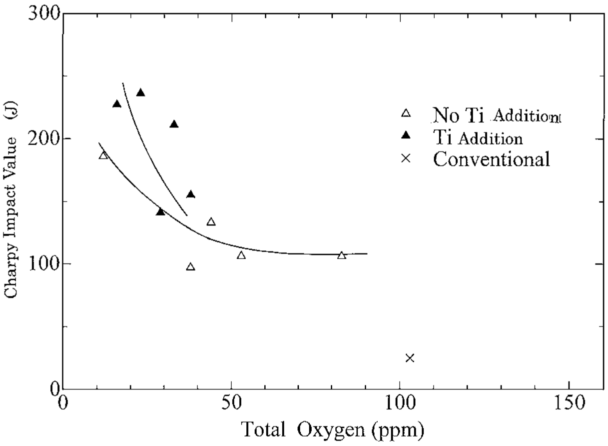


Figure 8 Relation between V-notch Charpy impact values at 273 K and total oxygen contents in as rolled steels.

3.4. Total oxygen

Fig. 8 indicates the decrease in Charpy impact values with total oxygen contents. Until 50 mass ppm total oxygen contents, Charpy impact values decrease remarkably. Of course, high total oxygen content steels contain high total nitrogen contents. But, there is no linear decrease of Charpy impact values with total oxygen contents, so that it is said that the influence on the toughness of steels is different to that of total nitrogen.

3.5. Microstructures

Figs 9 and 10 show optical microstructure and scanning electron micrographs of no titanium addition steel sample number 4 as control-rolled and its total nitrogen content is 132 mass ppm. Figs 11 and 12 show those of the conventional steel of which total nitrogen content is 119 mass ppm. The control-rolled steel has fine microstructures where pearlite is called degenerated pearlite [7, 8], but the conventional

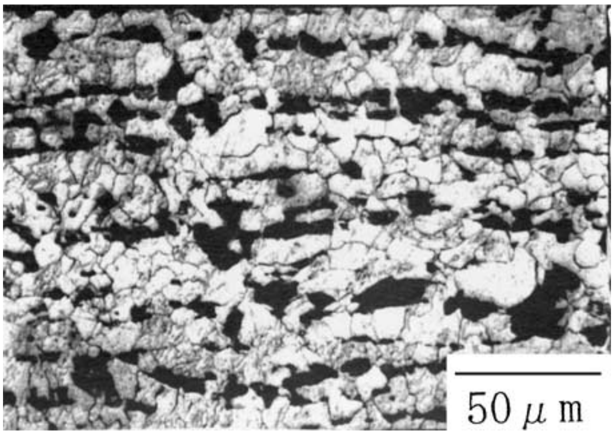


Figure 9 Optical microstructure of no titanium addition steel of sample number 4 as control-rolled. Total nitrogen content: 132 mass ppm.

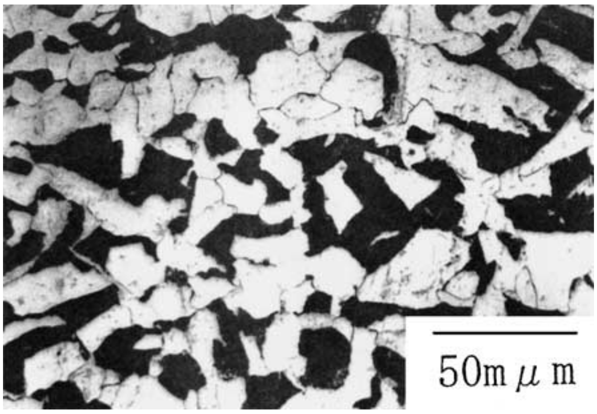


Figure 11 Optical microstructure of conventionally rolled steel of sample number Conventional. Total nitrogen content: 119 mass ppm.

one has coarse microstructures where pearlite is standard.

4. Discussion

The strain aging temperature in this research is 523 K where the nitrogen solid solubility in ferrite in equilibrium with Fe_4N is 41 mass ppm [3]. As seen in Table 1, all as rolled steels used in this research contain interstitial nitrogen less than 41 mass ppm. The solid solubility of nitrogen in ferrite in equilibrium with $Fe_{16}N_2$ at 523 K is 2400 mass ppm [3]. We have no evidence of iron nitrides whether Fe_4N or $Fe_{16}N_2$. But, the nitrogen contents in solid solution are less than 41 mass ppm as shown in Table I; it may be right to be Fe_4N.

The average ferrite grain size of the conventionally rolled steel is about 30 μm and that of the control-rolled steels is about 10 μm. The ferrite grain boundary area of the control-rolled steels is about ten times of that of the conventional material. As seen in Fig. 10, cementite in control-rolled steel is degenerated [7, 8], so that the interface area between ferrite and cementite is much wider than that of conventional pearlite. The grain boundaries and interfaces between ferrite and cementite are favorable for nitrogen precipitation. Fig. 13 shows the intensities of N K_α and C K_α in an electron probe microanalyser of which the electron beam diameter is 0.1 μm. The nitrogen concentration is high on the interface between ferrite and cementite, especially after strain aging at 523 K. As seen in Fig. 10, there is a possibility to produce degenerate pearlite [7, 8], which makes the toughness of steels increase. Nitrogen precipitates present after strain aging on the interfaces between ferrite and cementite, and ferrite boundaries are considered to contribute to the increase of the steel toughness. In this research work, we have no experimental datum on nitrogen precipitation on grain boundaries. But, a previous work reported [9] that the Fe-N system has the segregation of high nitrogen concentration to grain boundaries.

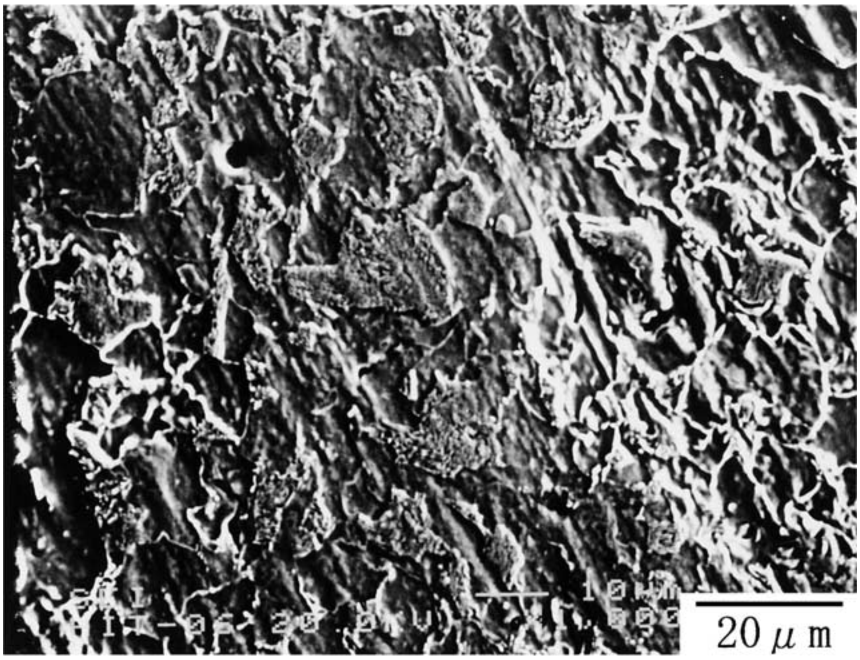


Figure 10 Scanning electron microstructure of no titanium addition steel of sample number 4 as control rolled. Total nitrogen content: 132 mass ppm.

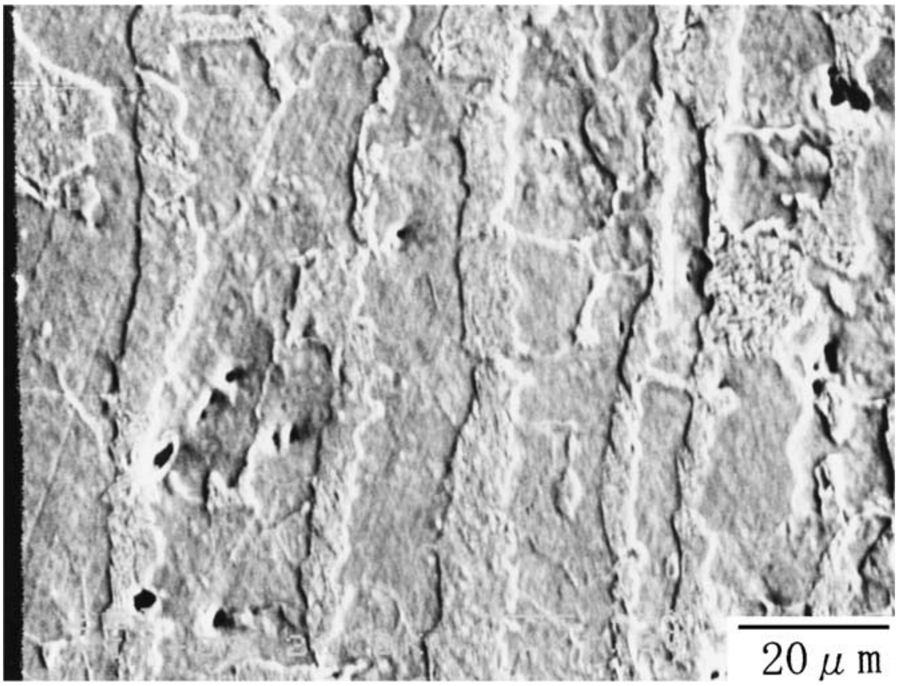
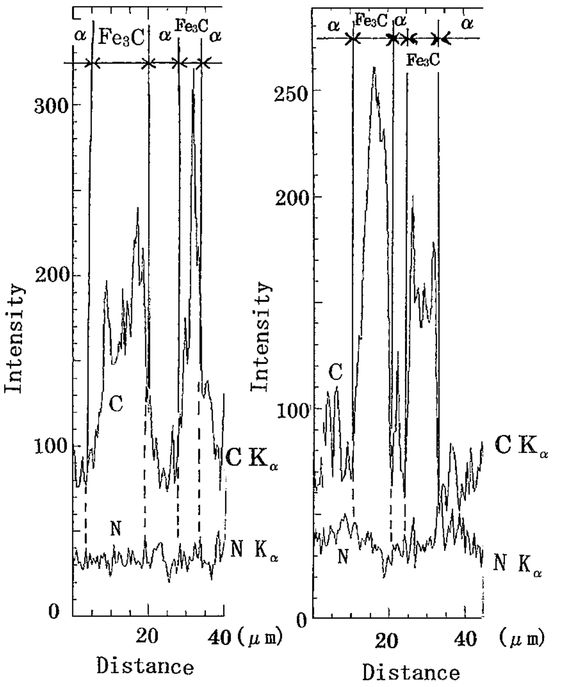


Figure 12 Scanning electron microstructure of conventionally rolled steel of sample number Conventional. Total nitrogen content: 119 mass ppm.



(A) As control-rolled (B) Strain aged

Figure 13 Line analysis of intensities of C K α and N K α in electron probe microanalyses. (A) As control-rolled sample number 4, (B) Sample number 4, Strain 5% + Aging at 523 K for 3.6 ks. Electron beam diameter: 0.1 μm, α: αFe.

5. Summary

The control-rolling of which rolling temperatures are lower than the A_1 point, is effective to improve the toughness of the low carbon steels which contain total nitrogen from 58 to 150 mass ppm. The control-rolled steels have higher Charpy impact values than 100 J at 273 K. The cause is considered to be due to fine microstructures.

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