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Ferrite phase analysis based on low-angle grain boundary density in low carbon linepipe steel

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Abstract: Ferrite phases of low carbon linepipe steels were separated into polygonal ferrite-like (PF) and acicular ferrite-like (AF) in this study. Two kinds of steels were fabricated by different hot rolling and cooling process in hot temperature. Microstructures and mechanical properties of the steels were analysed. PF and AF phases were clarified by using low-angle grain boundary (LAGB) density. LAGB was analysed with electron back-scatter diffraction (EBSD) analysis. Nanoindentation test was also performed to identify the mechanical property of each phase. Nano-hardness of the AF phase, which has higher LAGB density, was higher than that of the PF phase. Low yield strength (YS) was presented in the steel which had higher PF phase fraction and larger grain size of PF phase. Differentiation of ferrite phases using LAGB density is advantageous in that it is not unambiguous in classifying the ferrite phases, unlike the conventional method of identifying phases based on the shape of the ferrite.

1. INTRODUCTION

Low carbon linepipe steels widely used worldwide have various complex phases such as acicular ferrite and polygonal ferrite depending on their chemical compositions and hot processing [1]. Each phase in the steels has a characteristic showing different mechanical properties. Therefore, for the optimum design of the steels, it is very important to classify effectively each constituent phase in the microstructure and to analyse the correlation with the mechanical properties.

Up to now, the phase identification of steels has largely been conducted on the basis of grain shape in optical microscopy (OM) [2]. In conventional method, the grain shape classifying criteria was ambiguous and same microstructure was not classified uniquely and the classifying result was dependent on the researcher. In order to overcome this ambiguity, a new method based on low-angle grain boundary (LAGB) density which can be measured in electron back-scatter diffraction (EBSD) is proposed [3].

The mechanical properties of the identified phases were confirmed by nanoindentation test. Based on the difference in nano-hardness between the phases, the phase fraction and grain size of each phase in two steels were compared with its tensile properties [4,5].

2. EXPERIMENTS

The chemical composition and rolling conditions of the steels used in this study are shown in Tables 1 and 2, respectively. At the temperature below 1000°C, austenitization of the steels was carried out. After the austenitization, hot rolling was performed at the recrystallized region and non-recrystallized

Table 1. Chemical composition of the steels. (wt%)

C	Si	Mn	P	S	Al	Ni+Cu+Cr+Mo	Ti+Nb+V	Fe
0.05~0.07	0.23	1.5~1.65	0.0097	0.0017	0.031	0.25~0.30	0.05~0.07	Bal.

Table 2. Rolling conditions of the steels.

Sample	Reheat temperature(°C)	Rolling temperature(°C)	Start cooling temperature(°C)	Finish cooling temperature(°C)	Cooling rate(°C/s)
Steel A	Below 1000 °C	Above Ar_3	Above Ar_3	360-660	13-24
Steel B			Below Ar_3		

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region of austenite. Cooling down of the steels started from one of two temperatures after hot rolling. One was the temperature at austenite single phase region, which was above Ar_3 temperature and the other was the temperature at two phase region of austenite and ferrite, which was below Ar_3 temperature. For convenience, the steel which has start cooling temperature above Ar_3 temperature is referred to as Steel A and the steel which has start cooling temperature below Ar_3 temperature is referred to as Steel B. Finish cooling temperature and cooling rate were changed from 360°C to 660°C and 13°C/s to 24°C/s, respectively.

The transverse direction (TD) planes of the steels were mechanically polished and etched by a 2% nital solution. Microstructures of etched planes were observed with an optical microscope (OM). EBSD measurements (0.2μm resolution) in TD planes of the steels were performed after electro-polishing with a perchloric 10% acid/ethanol solution.

In the present study, phase classification of two low carbon linepipe steels based on low-angle grain boundary (LAGB) density was conducted. LAGB density is defined as the total length of the LAGB within the grain divided by the total area of the grain. Pixel-based digital data measured by EBSD analysis was used to determine the LAGB. Grain size and aspect ratio of each grain were also measured by EBSD. Polygonal ferrite-like (PF) and acicular ferrite-like (AF) phases were discriminated based on LAGB density in which the average grain shape was sufficiently elongated [2,6].

Also, nanoindentation test was performed on the area where EBSD analysis was performed. For each steel at room temperature, total 225 indentations on a 15*15 grid of the TD planes, 6μm away from each point, were performed. A berkovich tip with a calibrated radius of curvature of approximately 300nm was used for all test at a loading rate of 400μm/s with a maximum load of 2000μN. Flat subsize tensile specimens with a gage length of 25mm and a thickness of 3mm were prepared in the TD, and were tested at room temperature at a crosshead speed of 1mm/min.

3. RESULTS AND DISCUSSION

Fig. 1 shows optical micrographs of TD plane of Steel A and B. Steel A, which has start cooling temperature above Ar_3, mainly consists of acicular ferrite (AF). Steel B, which started cooling below Ar_3 temperature, is composed of polygonal ferrite (PF) primarily.

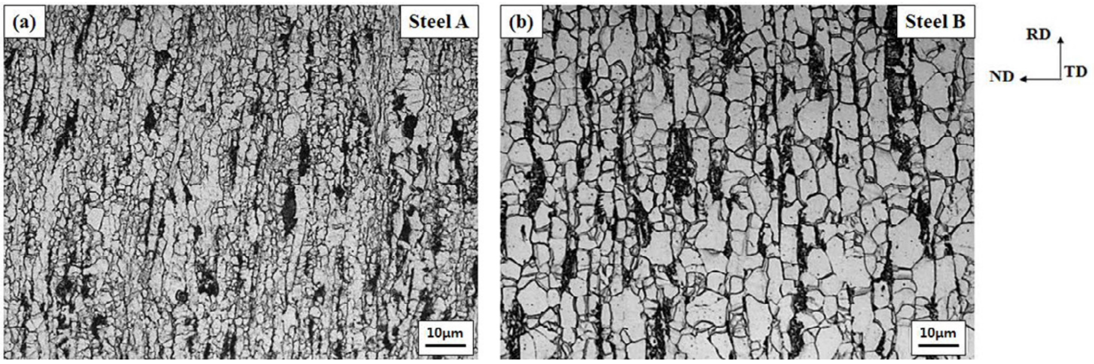

Fig. 1. Optical micrographs of Steel (a) A and (b) B showing their TD plane. Nital etched.

Figs. 2(a) and 2(b) shows misorientation angle maps of TD plane of Steel A and B analysed by EBSD. Misorientation angle below 15° was counted as LAGB, and the angle above 15° was counted as high angle grain boundary (HAGB). For the quantitative comparison of the LAGB, LAGB density of each steel was calculated. Average LAGB density of Steel A was more than twice as large as Steel B. The difference in LAGB density between Steel A and B means that LAGB density is changed with the phases in those steels. To investigate the behavior of LAGB density for each phase in both steels, LAGB density was calculated for grain by grain. Area fraction, aspect ratio, and grain size of the grains according to LAGB density were analysed to confirm the change of properties for each grain by LAGB density value.

Area fraction versus LAGB density of each grain in Steel A and B is presented in Fig. 3(a). Aspect ratio versus LAGB density of each grain in Steel A and B is also presented in Fig. 3(b). At this time, aspect ratio was defined as the grain length of minor axis over major axis. The conditions in which the area fraction decreased under 2% was excluded in analysis of aspect ratio since the fraction was too small to represent the characteristic of the grains. As shown in Fig. 3(b), aspect ratio of the grains tended to decrease as LAGB density increased. Also, aspect ratio of both steels converged under 0.35 when the LAGB density reached 0.3/μm. Since similar trends in grain shape occurs at LAGB density of 0.3/μm, the grains of Steel A and B were classified into two phases, PF phase and AF phase, based on LAGB density of 0.3/μm. Grain which have LAGB density below 0.3/μm and relatively rounded shape were classified as PF phase and the other grains which have LAGB density above 0.3/μm and relatively elongated shape were classified as AF phase. Since the AF phase has a various aspect ratio, the criterion for classifying the phases only by aspect ratio is ambiguous [6]. Thus, both LAGB density and aspect ratio was used to classify the phases more clearly. Table 3 shows area fraction and grain size of each phase of Steel A and B and the phase fraction map of each steel is presented in Fig. 2(c) and 2(d). Grain boundary was measured based on misorientation angle above 15°.

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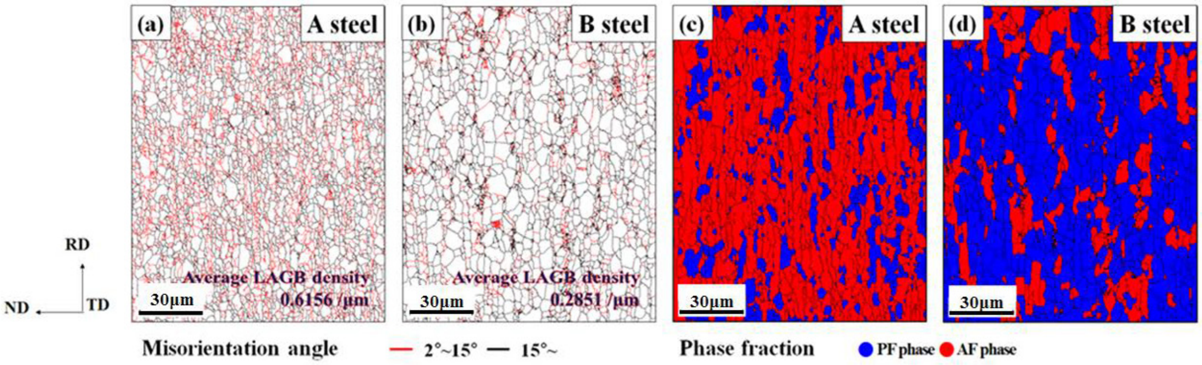

Fig. 2. EBSD misorientation angle map of Steel (a) A and (b) B and the identified phase fraction map of Steel (c) A and (d) B showing their TD plane.

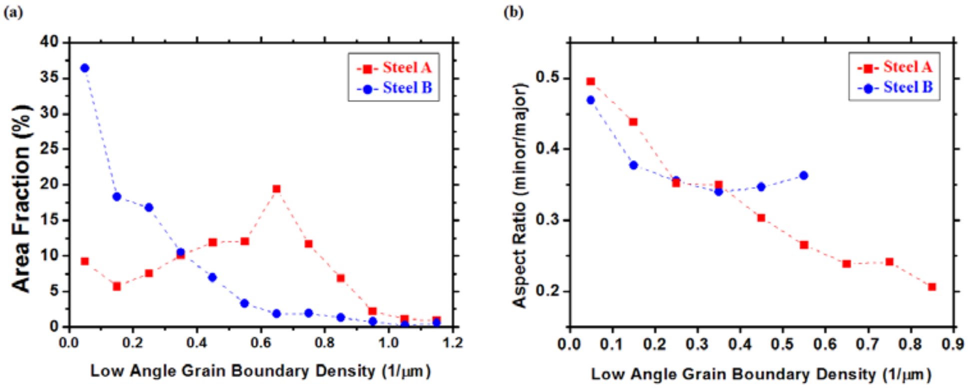

Fig. 3. (a) Area fraction vs. LAGB density and (b) aspect ratio vs. LAGB density of Steel A and B.

Table 3. Area fraction and grain size of the PF and AF phases in A, B steels.

Sample	PF Fraction (%)	PF Grain size (μm)	AF Fraction (%)	AF Grain size (μm)
Steel A	22.4	4.58	77.6	10.1
Steel B	71.5	7.23	28.5	7.77

Nanoindentation analysis was also performed to compare the mechanical properties of each phase. Fig.4 shows the load-depth curve of the PF and AF phases in Steel A and B. The average nano-hardness of the AF phase was 2.95GPa and that of the PF phase was 2.67GPa. It can be considered that the higher LAGB density, the higher average hardness of the grains.

Fig. 5 shows the room temperature stress-strain curves of Steel A and B and the tensile properties obtained from the curves are presented on Table 4. Although there was little difference in tensile strength (TS) and elongtation (El) in both steels, yield strength (YS) of Steel B was lower than Steel A. Since yielding occurs first in PF phase which is relatively soft, fraction and grain size of PF phase primarily affects yield strength of those steels [6]. Therefore, low YS in Steel B is caused by higher fraction and larger grain size of PF phase.

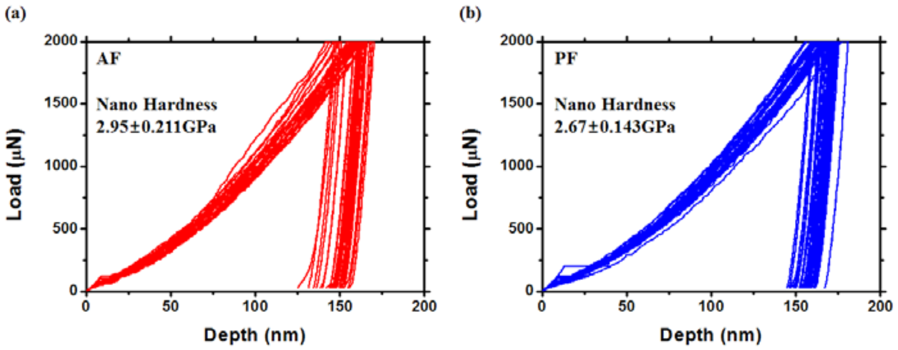

Fig. 4. Load-depth curve and nano-hardness of the (a) AF and (b) PF phases.

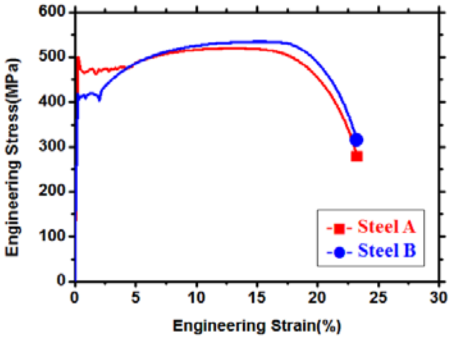

Fig. 5. Stress-strain curve obtained from the room-temperature tensile test of Steel A and B.

Table 4. Room-temperature tensile test results of A and B steels.

Sample	Yield strength (MPa)	Tensile strength (MPa)	Elongation (%)	Yield ratio (%)
Steel A	471	520	23.2	90.6
Steel B	414	534	23.2	77.5

4. CONCLUSION

In this study, the ferrite phase classification of two low carbon linepipe steels was conducted with using LAGB density. LAGB density, aspect ratio, and grain size of each grain were examined by EBSD analysis on both steels. Based on the change of grain morphology according to LAGB density, grains in the steels were classified into PF phase and AF phase on the basis of LAGB density 0.3/μm where aspect ratio was 0.35. The nano-hardness of each phase was measured by nanoindentation and the hardness of AF phase was higher than that of PF phase. By the difference in nano-hardness of the two phases, PF phase have a greater influence on the initial yielding behavior than AF phase. Since Steel B had higher fraction and larger grain size of PF phase than Steel A, Steel B showed lower YS than A steel.

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