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Characterization of nano-sized precipitates of low carbon steels by neutron scattering techniques

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1. Introduction

Hot-rolled sheet steels are normally supplied to satisfy such customer-defined specifications as yield strength, tensile strength and total elongation. Typically, as yield strength (or yield-to-tensile ratio) increases, relative ductility decreases. Boron-added low carbon steel is recommended for the automotive parts because of its excellent ductility. While the effect of boron on tensile strength is known through its influence on the hardenability, its influence on the evolution of ferrite morphology and elongation is still controversial(1). In this study, the effect of nano-sized precipitates and addition of boron on the mechanical properties of low carbon steels was investigated to make clear the precipitation behavior with rolling temperature. In order to quantitatively investigate the behavior of the precipitates in boron-added (BA) and boron-free (BF) steels during rolling process, a small angle neutron scattering (SANS) and a neutron powder diffraction technique were used. In addition, we describe the influence of hot rolling temperature on the mechanical properties of the boron-added low carbon steels.

2. Experimental Procedures

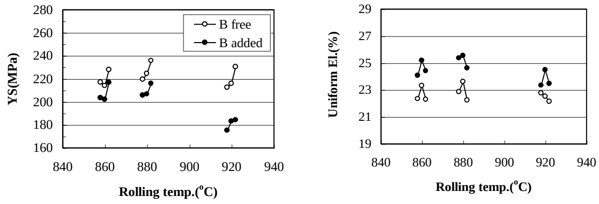
The chemical composition of a steels used are 0.02C, 0.2Mn, 0.012P, 0.025 Sol-Al and 0.002 B in wt% produce by the POSCO steel company, Korea. The ingots were vacuum melted in the laboratory and hot rolled to small slab with thickness of 30 mm after heating to 1200°C. The slabs were reheated to 1150°C for 120 minutes. To confirm the dependence of rolling temperature, the pilot mill tests were performed at different final pass temperature such as 860, 880 and 920°C. After hot rolling, the hot rolled sheets were cooled to 650°C corresponding coiling temperature.

An analysis of microstructure was performed by the optical microscopy. The characteristics of precipitates such as MnS, BN and AlN were investigated by transmission electron microscopy (TEM, Philips CM1200). TEM analysis used a standard replica method. Also to investigate quantitatively the total volume fraction of cementite precipitates, a neutron powder diffractometer in the HANARO, KAERI was used. A SANS instrument was used to investigate the behaviors of the nano-sized precipitates (size < 60 nm in diameter) in the samples.

3. Results and Discussion

Fig.1 represents the effect of the hot rolling temperature on the mechanical properties of both BA and BF low carbon steels. The yield strength and uniform elongation of boron-free steels maintain nearly constant values with rolling temperature. However, the strength, especially the yield strength, of boron-added steels decreases with increasing rolling temperature. This dependence of mechanical properties on rolling temperature for boron-added steel is suggested to be due to the different microstructural evolution and precipitation behavior of that steel.

Fig. 2 shows the relationship between total volume fraction of the cementites and rolling temperature calculated from neutron diffraction patterns by Rietveld method(2). In boron-free steels, the volume fraction of cementites decreased with increasing rolling temperature. On the other hand, in boron-added steels, the volume fraction of cementites increased drastically at higher rolling temperature.



(a) yield strength (b) uniform elongation

Fig. 1 Effects of rolling temperature on the mechanical properties of low carbon steel:

TEM observation showed that most of precipitates in ferrite matrix were identified as BN precipitates which have nucleus of MnS or CuS by EDS analysis. The nucleus size was mainly in the range of 10 to 50 nm, although some contained a very large precipitates, up to 500 nm. Thus, in boron added steels, their shapes are either core-shell structured spherical or ellipsoidal such as MnS precipitate surrounded by BN layers. Otherwise, in BF steels, precipitate shapes are either spherical or ellipsoidal.

In order to investigate precipitates quantitatively, SANS experiments were carried out for boron-free and boron-added low carbon steels. In the case of boron-free steels, the intensities of SANS spectra are contributed by MnS precipitates in the range of 10 to 50nm and by very fine MnS or/and CuS precipitates less than 5 nm.

The core-shell structured precipitates, which are MnS surrounded by BN layer in the range of 10 to 50 nm and spherical MnS precipitates less than 10 nm were dispersed in BA steels. In BF steels, MnS precipitates with wider size range were dispersed. The scattering length densities of MnS and BN precipitates in iron matrix are $(\Delta\eta)_{MnS} = 8.258\times10^{-6}$ cm^{-2} and $(\Delta\eta)_{BN} = 4.402\times10^{-6}$ cm^{-2}, respectively.

The model fitting of the real-space size distribution to the scattering patterns was performed using the non-linear least-square fitting method$^{(3), (4)}$. A simple real-space model consists of one distribution of precipitates per curve. These log-normal distributions were fitted to the Q range between 0.14 and 2.4 nm^{-1} for BF and BA steels.

Fig. 3 shows the volume fraction of the precipitates calculated by a direct model fitting of the SANS spectra as a function of the hot rolling temperature for the BA and BF steels, respectively. The results revealed that the volume fraction of the precipitates in the BA steels was larger than that of the ones in the BF steels. Fine spherical core-shell structured precipitates, with an average size of ~ 5 nm in radius, existed in the BA samples, whereas fine spherical precipitates, with an average size of ~ 4.8 nm, existed in the BF steels. When boron is added to the low carbon steels, the precipitates were coarsened with the growing BN layers on the MnS and CuS precipitates. However, the average size of fine precipitates less than 20 nm in radius in both the BA and the BF steels had no significant changes with decreasing rolling temperature.

It was also found that when boron was added the volume fraction of the fine precipitates in the low carbon steels increased. There are two reasons as follows; the BN layers grew rapidly on the MnS or CuS precipitates and the weight fraction of boron-cementite phases like $Fe_3(C, B)$ larger than 20 nm in radius increased with an increasing rolling temperature because of decreasing the weight fraction of the cementite phases. Number of boron-precipitates such as BN, MnS surrounded by BN, $Fe_3(C, B)$, and so on, also increased at higher rolling temperature. However, in the BF steels, MnS and CuS precipitates were observed and the volume fraction of the precipitates less than 20 nm in radius did not change significantly. This suggests that the precipitation of the boron-precipitates was activated at higher rolling temperature. Thus, the excess boron reduced and the excess carbon/nitrogen increased a little in the matrix.

It is clear that the boron addition to the low carbon steels increased the uniform elongation and decreased the yield strength as shown in Fig. 1, and that it increased the volume fraction of both the spherical core-shell structured precipitates and the cementites as shown in Fig. 2 and 3.. In the BA steels, it seems that the coarsening of the BN precipitates having fine nuclei of MnS or CuS and the increased volume fraction of the cementites reduced the solute nitrogen and carbon contents. As a result, the BA steels exhibited lower strength and higher elongation values than the BF steels.

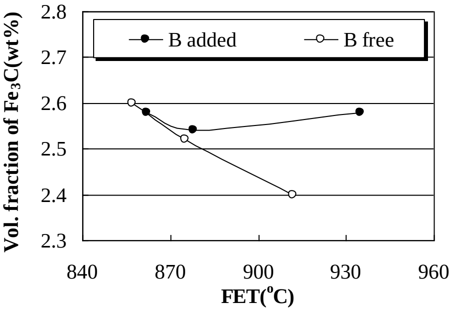


Fig. 2 Total volume fraction of cementite obtained from Rietveld analysis at various rolling temperature in low carbon steels.

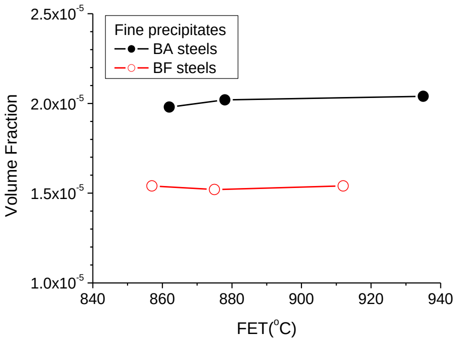


Fig. 3 Relationship between volume fraction of fine precipitates and rolling temperature.

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