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The impact of Nb on dynamic microstructure evolution of an Nb-Ti microalloyed steel

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ABSTRACT

Dynamic recrystallization (DRX) grain size of a Nb-Ti microalloyed steel is investigated at various strain rates and temperatures. Electron microscopy reveals that Nb preferably precipitates on TiN particles at temperature range of $T < 1100\,^{\circ}C$. As a result, considerable data scatterings are observed for the power law relationship between the normalized DRXed grain size (D_{DRX}/b) and the Zener-Hollomon parameter (Z), the peak strain (ε_p) and the normalized steady state stress (σ_{ss}/E). Much better fittings are achieved, however, when distinct analyzes are carried out within the two temperature ranges of $T < 1100\,^{\circ}C$ and $T \geq 1100\,^{\circ}C$. It is found that the well-known universal relationship suggested earlier for D_{DRX}/b does not agree with the present results when they are analyzed separately at the two ranges of temperatures. Through this separation, an approach is developed for quantitative analysis of DRX retardation by Nb solute atoms. Moreover, the results show that separate analyses at the two temperature ranges result in almost similar dependencies of D_{DRX}/b, ε_p and σ_{ss}/E as a power function of Z with indexes in the range of 0.11–0.15.

1. Introduction

High Strength Low Alloy (HSLA) steels have received increasing interest in numerous industrial applications owing to their high strength and toughness, good weldability and yet relatively low cost [1,2]. Superior performance of these steels are achieved by a proper design of chemical composition along with an optimized thermo-mechanical processing. The key factor is to develop a fine grained austenite during hot deformation which leads to form fine grained ferrite from the austenite phase transformation during cooling [3,4]. Therefore, it is of great importance to evaluate the austenite grain size during the thermomechanical processing. Generally, the austenite grain size can be affected by static and dynamic recrystallization (DRX) during and between hot deformation passes [5]. In addition to the hot deformation parameters, i.e. strain, strain rate and temperature, the microalloying elements, particularly Nb and Ti, play an important role in this way. They constitute various types of precipitates mainly in the forms of carbides, nitrides and carbonitrides [6,7]. These precipitates are very effective on the development of final strength and toughness of the microalloyed steels through both grain refinement and precipitation hardening [8]. Ti-rich precipitates, in particular TiN, have been shown to be effective on preventing austenite grains growth at very high temperatures [9]. Nb, in both forms of solute atoms and precipitates, in particular NbC, plays an important role in microstructural evolutions of microalloyed steels during hot deformation. It can retard the occurrence of DRX by its pinning effect on the austenite grain boundaries during hot deformation [10]. Thus, the strain can be accumulated up to higher levels during hot deformation leading to enhanced grain refinement during phase transformation [11,12].

According to the mentioned role of microalloying elements, one can find that a proper design of thermomechanical processing requires a comprehensive knowledge of their mechanism of precipitation and interactions with the hot deformation parameters. Strain induced precipitation is considered as the most effective mechanism of NbC formation during hot deformation [13]. However, depending on the employed thermomechanical process, Nb precipitates can form in austenite during hot deformation, in ferrite after the phase transformation or during the austenite to ferrite phase transformation [14]. Increase in

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the deformation temperature would restrict the Nb precipitation in austenite and enhance its precipitation in ferrite [15]. It has been shown that in a Nb-Ti microalloyed steel, this can result in higher strength due to finer Nb-rich precipitates, although the grain refinement contribution is decreased by increase of the deformation temperature [16]. Therefore, an optimized design of thermomechanical processing is required to get advantages of both precipitation hardening and grain refinement.

As explained above, not only the hot deformation conditions, but also the state of the microalloying elements, Nb in particular, are effective on the austenite grain size before the phase transformation. Accordingly, it is of great importance to investigate and compare the austenite grain sizes in presence of Nb solute atoms versus Nb-rich precipitates during high temperature deformation. Therefore, the present study aims to investigate the austenite DRX grain size of a Nb-Ti microalloyed steel as a function of the deformation conditions in both states of Nb microalloying element: solute atoms and fine precipitates.

2. Experimental procedure

A billet of Nb-Ti microalloyed steel with the chemical composition given in Table 1 was prepared by vacuum induction melting and homogenization at 1200 °C. The billet was hot rolled to a plate with thickness of 16 mm as the primary material. Hot compression specimens with length of 12 mm and diameter of 8 mm were prepared from center of the plate at rolling direction. Isothermal compression tests were implemented at temperatures of 850–1200 °C and strain rates of 0.01–1 s^{-1} up to high strain of 1.2 s^{-1} to ensure that DRX is occurred completely. In order to freeze the microstructure of hot compressed specimens, they were immediately water quenched after the hot compression test.

The microstructures were investigated by light microscopy. Longitudinal sections of the specimens were polished and etched in a saturated aqueous picric acid solution to detect the primary austenite grain boundaries. Grain boundaries were highlighted for accurate detection by the image processing program, ImageJ [17]. Through this approach, the grain size was calculated as average of diameters of circles with equal area of the detected grain.

Scanning Electron Microscopy (SEM) along with Energy Dispersive X-ray Spectroscopy (EDS) were implemented to analyze precipitates in specimens deformed at temperatures of 1000 and 1200 °C with strain rate of 0.1 s^{-1}. A Schotky field emission SEM (JEOL JSM-7600F) equipped with an Energy-Dispersive Spectroscopy (EDS) X-ray analyzer (Oxford Instruments X-max 50 mm2) was employed at 15 kV for this purpose. Furthermore, nanometer-sized precipitates in specimen compressed at 1000 °C with strain rate of 0.1 s^{-1} were analyzed by Transmission Electron Microscopy (TEM). The 3-mm diameter TEM sample was punched out from a thin foil. Subsequently, it was subjected to Precision Ion Polishing System (PIPS) to prepare the thin electron transparent sample. TEM analyses were performed by a Jeol TEM 1200EX at 200 kV operating voltage.

3. Results

3.1. Flow curves

Flow curves obtained by hot compression tests at various strain rates and temperatures are depicted in Fig. 1. Effect of friction and strain rate variation was corrected by a mathematical approach [18].

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

C	Si	Mn	Mo	Nb	Ti	N	Bal.
0.055	0.25	0.7	0.17	0.018	0.023	0.003	Fe

The flow curves show a plateau at large strains where a steady state deformation is expected. The classic features of dynamic recrystallization is seen in the curves, so that the flow stress increases by the strain up to the peak stress (σ_p) corresponding to the peak strain (ε_p) followed by a decrease until the steady state stress (σ_{ss}). Under the conditions of high temperatures and low strain rates, i.e. low Zener-Hollomon parameter, multiple peaks are distinguished as the characteristic of DRX [19,20]. High temperature flow parameters of the present steel were analyzed and a phenomenological constitutive model of the classical hyperbolic sine equation was presented in the previously published paper [21]. The model was developed based on the theoretical activation energy of $Q = 270$ kJ/mol attributing to the austenite self-diffusion and the theoretical exponent of $n = 5$. The same value of Q is implemented here to calculate the Zener-Hollomon parameter (Z) at various deformation conditions:

$$Z = \dot{\varepsilon} \exp\left(\frac{Q}{RT'}\right) \tag{1}$$

in which $\dot{\varepsilon}$ is the strain rate, T' is the absolute temperature and R is the ideal gas constant. Based on investigations of Cabrera et al. [22,23], in order to successfully apply the activation energy of self-diffusion and the theoretical exponent of $n = 5$, temperature dependence of the Young's modulus (E) should be taken into account. Therefore, σ_{ss} is normalized by E in the present work. The Young's modulus temperature dependency within the applied range of temperatures is expressed as [24]:

$$E = 233.7 - 100.36\ T\,(\text{GPa}) \tag{2}$$

3.2. Grain size evolution

The microstructures along with maps of the grain boundaries after hot compression are illustrated in Figs. 2, 3 and 4 for strain rates of 0.01, 0.1 and 1 s^{-1}, respectively. The grain size distributions are also presented in these figures by the relevant histograms. The micrographs are almost covered by equiaxed grains. The bimodal distributions of grain sizes in Fig. 2 (1100 °C and 1200 °C) can be attributed to the abnormal grain growth of some grains at low strain rate and high temperature. Effects of the strain rate and temperature on the average grain sizes of the present results are shown in Fig. 5(a). As can be found from these results, the DRX grain size is increased by the deformation temperature and decreased by the strain rate. In other words, the DRX grain size is decreased by the Z parameter. Similar dependency has been formulated in previous studies by a power law equation [25–27]. Here, the DRX grain size (D_{DRX}) in the power law equation is normalized by the Burger's vector (b):

$$\frac{D_{DRX}}{b} = C\ Z^{-m} \tag{3}$$

in which C and m are material constants. The Burger's vector is calculated at various temperatures form the lattice parameter of austenite [28]. $\ln(D_{DRX}/b)$ of the present results is depicted versus $\ln(Z)$ in Fig. 5(b). As shown in this figure, a line can fit in accordance with Eq. (3). However, a large deviation from the fitted line is evident (R-squared of 0.83). From Fig. 5(c), it is obvious that this deviation is much less when two different lines are fitted to data sets of $T < 1100$ °C and $T \geq 1100$ °C. The relevant values of C and m are summarized in Table 2. The index m is almost the same for both fitted lines of Fig. 5(c), but different to that of Fig. 5(b).

The natural logarithm of (D_{DRX}/b) is also drawn against $\ln(\varepsilon_p)$ in Fig. 6(a). A large deviation from the fitted line is apparent (R-squared of 0.64). As can be seen in Fig. 6(b), much better fittings with R-squared > 0.9 can be made when data points of $T < 1100$ °C and $T \geq 1100$ °C are treated separately.

In the earlier investigations, D_{DRX} is treated as a function of the flow stress in the form of [29,30]: