Effect of Nb on the transformation kinetics of low carbon (manganese) steel during deformation of undercooled austenite

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Abstract: The hot compression tests using Gleeble 1500 were performed by varying the true strain up to 1.6 (80% reduction) in Nbfree and Nb-microalloyed steels. The effect of Nb addition on the transformation kinetics during deformation of undercooled austenite was investigated. It was found that as compared with Nb-free steel, the transformation incubation period of Nb-bearing steel was prolonged and the transformation kinetics curves parallelly moved to higher strain because of the solute Nb drag effect. Studies on kinetics also showed that the deformation-enhanced ferrite transformation (DEFT) of the two steels were composed of three stages, which can be expressed by the J-M-A equations individually. However, the parameter *n* related to the mode of nucleation and growth is somewhat different in the first and second stages of the two steels, and the same in the third stage for both the steels corresponding to the nucleation of retained austenite.

Key words: Nb-microalloyed steel; deformation-enhanced ferrite transformation; transformation kinetics; J-M-A equations

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1. Introduction

Microalloyed steels have received considerable interest and have been used widely in industrial applications over many years. Nb is one of the most frequently added microalloying elements because of its strong effect on microstructures and its mechanical properties. The element Nb remains in solution at high temperatures and upon cooling it combines with C and N to form carbonitride precipitation.

Recent studies have suggested a promising method for controlling the microstructure using deformationenhanced ferrite transformation (DEFT), leading to effective grain refinement and thus improved properties [1-3]. Unlike traditional thermo-mechanically controlled processing (TMCP), the driving force supplied by both undercooling and deformation simultaneously acts on the transformation process without crystallographic defect relaxation.

Although it has been found that the addition of Nb influences the transformation kinetics [4-5], systematic investigation and understanding of the effect in Nb-microalloyed steel is not yet well established. It is unclear whether the Johnson-Mehl equation is still appli-

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cable for the condition of transformation during deformation in the two steels, and the difference in the mechanisms of nucleation and growth of phase transformation during deformation with and without Nb addition is yet to be revealed. The purpose of the present study is, thus, to identify the effect of Nb on the transformation kinetics during deformation of undercooled austenite, based on both hot deformation simulation experiments and the analysis of transformation kinetics curves according to the J-M-A equations.

Materials

2. Experimental procedure

The chemical composition of the steel used in this investigation is given in Table 1. The specimens were prepared by vacuum induction melting to ingots, forged within the temperature range of 900-1100°C, and then normalized at 900°C for 20 min. The cylindrical specimens ($\phi 6 \text{ mm} \times 15 \text{ mm}$) were machined.

The hot compression tests were performed using a Gleeble 1500 hot simulation testing machine. Fig. 1 shows the schematic processing of deformationenhanced ferrite transformation. The primary austenite grain size of the two steels is about 100 μ m and the initial deformation temperature is above A_{r3} and below A_3 . The corresponding A_{r3} , A_3 and the prior austenite grain size of the tested steel are shown in Table 2. The sam-

ples were quenched in ice water immediately after deformation.

Table 1. Chemical composition of the steel							wt%
Steel	С	Mn	Si	S	Р	Nb	Al
Nb-bearing steel	0.093	1.18	0.12	0.0064	0.0100	0.024	< 0.01
Nb-free steel	0.110	1.24	0.17	0.0073	0.0092	—	< 0.01

Table 2.	A_{r3}, A_{3}	and prior	austenite	grain si	ize of	the tested	steel
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Steel	$T_{\rm A}$ / °C	Cooling rate / (°C·s ⁻¹)	$A_{r3} / °C$	$A_3 / ^{\circ}\mathrm{C}$	Prior austenite grain size / µm
Nb-bearing steel	1200	10	680	845	103.35 ± 14.90
Nb-free steel	1200	10	675	805	98.47 ± 11.45



Fig. 1. Processing scheme of deformation enhanced ferrite transformation.

Specimens for metallographic examination were etched in 2%-4% nital. The observation area was in the middle of the cross-section of the samples. The Leica imagine analysis and the linear intercept technique were used for ferrite grain size and volume fraction measurements. The microstructures were observed under scanning electron microscope.

3. Results and discussion

3.1. Change of ferrite grain number during isothermal treatment

It is already known that during isothermal treatment ferrite nucleates preferentially at the prior γ grain boundaries. Once the boundaries are occupied by allotriomorphic ferrites, the progress of transformation is mainly controlled by the growth of the ferrite. As shown in Fig. 2, during the early stage of transformation, when the holding time increased from 30 to 60 s, the value of N_A (ferrite grain numbers per area) increased dramatically, thereby indicating that nucleation dominated during this process. However, on increasing the holding time above 60 s, the value of N_A decreased and the ferrite grain size increased significantly, that is to say, the ferrite grains grew obviously.



Fig. 2. Variation of the ferrite grain number (a) and the ferrite grain size (b) with time during the isothermal transformation process at 760°C in the two steels (N_A : ferrite grain numbers per area).

The incubation period of Nb(CN) precipitation during isothermal transformation at 760°C is about 300 s according to TEM study [6]. Hence, during the holding time from 30 to 300 s, the N_A value of Nb-free steel is higher than that of Nb-bearing steel because the dissolved Nb retards the transformation by the solute drag effect. Commencing from 300 s, the N_A value of Nbfree steel is lower than that of Nb-bearing steel because the dispersed Nb(CN) particles produce a large number of additional preferential nucleation sites for ferrite transformation. However, the presence of 30 nm static Nb(CN) particles could not inhibit the ferrite grain growth effectively; therefore, there was no considerable difference between the ferrite grain size of Nbbearing steel (45.26 ± 10.22 µm) and that of Nb-free steel (47.62 ± 9.08 µm).

3.2. Change of the ferrite grain number during deformation of undercooled austenite

Fig. 3 illustrates the change of the ferrite grain number during deformation of undercooled austenite. It can be obviously seen that the N_A value of the two steels during deformation-enhanced ferrite transformation is 3-4 times more than that during isothermal transformation (shown in Fig. 2). The present experiment confirmed that the most important feature of deformationenhanced ferrite transformation is a nucleation dominated process. The nucleation is continuously repeated and the grains grow only slightly because of the lack of time and space until the transformation is completed.



Fig. 3. Variation of the ferrite grain number with time (a) and ferrite volume fraction (b) during deformation at 760°C in the two steels.

When compared with Nb-free steel, only when the strain increases to 0.69, the N_A value is higher in Nbbearing steel; at the same ferrite volume fraction, the N_A value of Nb-bearing steel is much higher than that of Nb-free steel. It can be obviously concluded that the ferrite grain size is much finer in Nb-bearing steel than that in Nb-free steel, as shown in Fig. 4.



Fig. 4. SEM images of the two steels during deformation at 760°C to strain of 1.61: (a) Nb-free steel; (b) Nb-bearing steel.

3.3. Transformation kinetics features of DEFT in the two steels

The relationship between the ferrite volume fraction and the transformation time is shown in Fig. 5(a). It can be obviously seen that the transformation incubation period of Nb-bearing steel was prolonged, and then the higher strain was needed to accomplish the transformation compared with that of the Nb-free steel. Therefore, the transformation kinetics curves parallelly moved to high strain. It is probable that dissolved Nb retarded the DEFT by the solute drag effect and the accumulation of strain energy for the precipitation of Nb(CN) during deformation. Also, the activity of carbon is reduced by the solute Nb because of strong interaction between carbon and Nb [4]. Thus, the driving force for DEFT is reduced resulting in a retardation of the transformation. After an incubation period, the ferrite volume fraction increases significantly because with precipitation of dynamic Nb(CN) particles, the solute Nb drag effect decreases dramatically, accumulated strain energy accelerates ferrite transformation, and also these small particles inhibit the movement of dislocations and the growth of ferrite grains severely.

As seen in Fig. 5(b), the kinetics curve of DEFT is composed of three straight lines. Every straight line can be expressed by the J-M-A equation:

$$f=1-\exp(-Kt^n) \tag{1}$$

The equation can be modified to another form:

$$\lg \left\lfloor \ln \left(\frac{1}{1-f} \right) \right\rfloor = \lg K + n \lg t \tag{2}$$

where *f* is the ferrite volume fraction, *t* is the transformation time and is given by $t = \varepsilon / \dot{\varepsilon}$, *n* is a parameter related to the mode of nucleation and growth, and *K* is a

parameter related to the rate of nucleation and growth.



Fig. 5. Kinetics of deformation-enhanced ferrite transformation under deformation of the two steels: (a) relationship between the ferrite volume fraction and time; (b) synthetic analysis of ferrite transformation kinetics according to the J-M-A equations. f is the ferrite volume fraction, and t is the transformation time.

The above equation can be plotted as $lg\{ln[1/(1-f)]\}$ vs lgt curves, as shown in Fig. 5(b). Cahn has derived

the kinetic law for site saturation [7]. For grain boundary nucleation, edge nucleation and corner nucleation corresponding to which n equals 1, 2, and 3, respectively, the law of site saturation can be expressed as:

$$f=1-\exp(-K_s t), K_s=6.7GD^{-1} (n=1)$$
 (3)

$$f=1-\exp(-K_{\rm e}t^2), K_{\rm e}=8.5\pi G^2 D^{-2} (n=2)$$
 (4)

$$f=1-\exp(-K_{c}t^{3}), K_{c}=16\pi G^{3}D^{-3}$$
 (n=3) (5)

where G is the radial growth velocity, and D is the austenite grain diameter.

When more than one type of site is active, the rate law can have the following form corresponding to n=4, where \dot{N} is the total nucleation rate per unit volume:

$$f=1-\exp\left(-\frac{\pi}{3}\dot{N}G^{3}t^{4}\right) (n=4)$$
(6)

In combination with the microstructure observation, the mechanism in each stage was clarified. The transformation kinetics curve of Nb-free steel is somewhat different from that of Nb-bearing steel. As shown in Table 3, in Nb-free steel, the first stage that corresponds to the nucleation at the grain boundaries, and the slope *n* of the first stage investigated in the present study is about 4, which coincides with Cahn's "site saturation" mechanism; the second stage indicates the nucleation at areas in front of the newly formed ferrite grains, and in which $n \approx 1.6$, and the nucleation rate is fairly high; and the third stage indicates the transformation of the isolated residual austenite phase, where *n* is very low.

Stage	Parameter	Nb-free steel	Nb-bearing steel	
	lg <i>K</i>	1.69	-0.54	
	n	4.05	2.65	
	lg <i>K</i>	0.07 ± 0.03	-0.11 ± 0.04	
	n	1.63 ± 0.08	4.07 ± 0.08	
	lgK	0.04 ± 0.01	-0.11 ± 0.04	
	n	0.71 ± 0.07	0.97 ± 0.06	

Table 3. Values of lgK and n corresponding to the three stages of the kinetics curves in Fig. 5(b)

However, as shown in Table 3, in Nb-bearing steel, the slope n of the first stage is about 2.35, and the required strain is increased to satisfy the diffusion conditions for transformation. The slope n of the second stage is about 4.0, because the accumulation of the accumulated strain energy is applied to the austenite grain effectively, and the density of the defects is increased significantly, and with strain-induced dynamic precipitation of Nb(CN), ferrite transformation is accelerated dramatically. Thus, the nucleation of the ferrite grains is an explosive process. In such a short duration of time, the kinetics obeys the model of site saturation. Also, the slope *n* of the third stage is almost the same as that in Nb-free steel.

4. Conclusions

Compared with Nb-free steel, the transformation incubation period of Nb-bearing steel is prolonged and the transformation kinetics curves parallelly move to higher strains; after an incubation period, the ferrite volume fraction increases significantly to accomplish the transformation during a relatively small time range.

Compared with Nb-free steel, the N_A value of Nbbearing steel increases significantly with increasing strain, and therefore, under the same ferrite volume fraction, the ferrite grain size of Nb-bearing steel is more refined than that of Nb-free steel.

According to the J-M-A equations, there is a remarkable difference between the transformation curve of Nb-bearing steel and that of Nb-free steel. In Nbbearing steel, the second stage is an explosive nucleation process; ferrite grains nucleate on austenite grains dispersively and the dynamic precipitation of Nb(CN) particles restrict ferrite grain growth effectively, so ferrite transformation is accelerated significantly.

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