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Research Article

Research on the Static Recrystallization and Precipitation Behaviors of a V-N Microalloyed Steel

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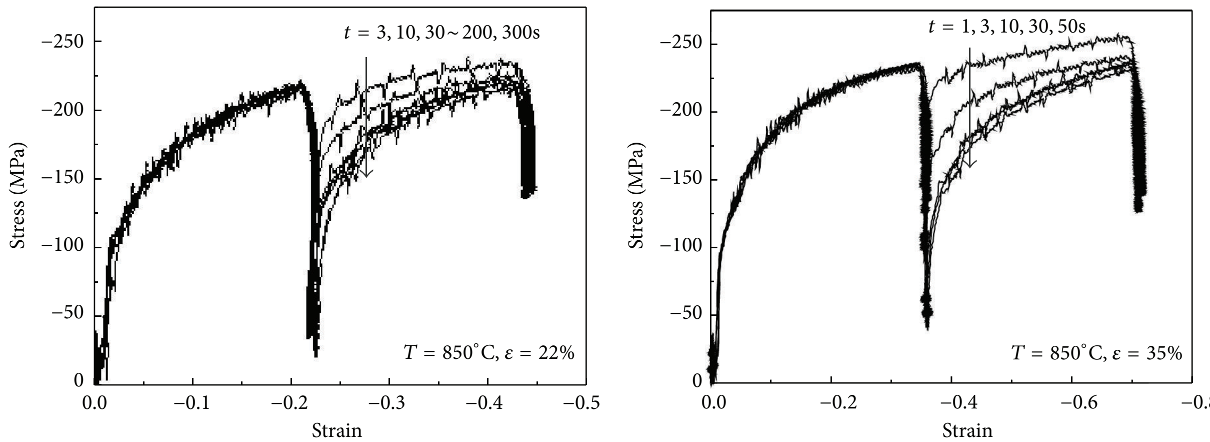
Double compression tests were performed on a Gleeble-3800 thermomechanical simulator to study the softening behaviors of deformed austenite in a V-N microalloyed steel. The static recrystallization volume fractions were calculated by stress offset method, and the kinetic model of static recrystallization was constructed. The effects of temperature, strain, and time interval on the softening behaviors were analyzed, and the interactions between precipitation and recrystallization were discussed. The results show that the softening behaviors of the deformed austenite at lower temperature or higher temperature are markedly different. At the temperature of 850°C or 800°C, pinning effects of the precipitates play the main role, and the recrystallization process is inhibited, which leads to the formation of plateaus in the softening curves. An increase in strain promotes the precipitation and recrystallization processes while reduces the inhibition effect of precipitation on recrystallization as well.

1. Introduction

Thermomechanical controlled processing is a well-known way to provide a good combination of strength, fracture toughness, and weldability in microalloyed steels [1]. Vanadium is known to be the microalloying element that can delay static recrystallization kinetics significantly by the formation of carbides, nitrides, and/or carbonitrides [2–4], which can be seen in the values of $t_{0.5}$, activation energy, and static recrystallization critical temperature for higher vanadium additions [5]. Furthermore, the precipitation of vanadium plays a major role in controlling the final microstructure [6, 7] and hence in the product properties. In consideration of the enormous practical importance of the precipitation process and static recrystallization, it is essential to understand the role of precipitation and static recrystallization including the interactions between precipitation and static recrystallization.

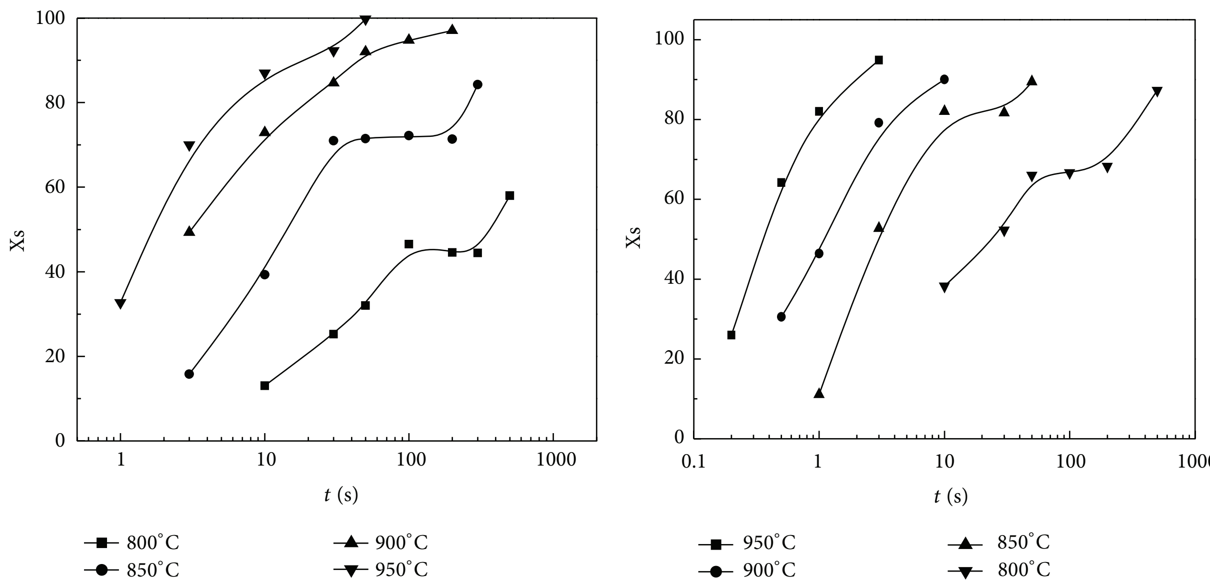
In the previous work, it has been proved that vanadium can exhibit its potential by the applications of microalloying technology, and the steel products with high strength can be produced by lower vanadium and higher nitrogen additions [8], which leads to a lower cost for steel industry. Some researches have been done to study the static recrystallization of vanadium microalloying steels. However, most of the works focus on the steel whose vanadium contents are often higher than 0.1 mass% or nitrogen contents are at a lower level. Therefore, few works on the steels with lower vanadium and higher nitrogen additions are reported. It has been proved in Yang and Zhang's [8, 9] and Bangming et al.'s [10] works that nitrogen is a very cost-effective microalloying element, which provides a new approach to produce high strength steels with low cost and high additional value.

In the present work, the softening behaviors of a microalloyed steel with low vanadium (0.05 mass%) and high nitrogen (0.018 mass%) additions were studied by performing double compression tests on a Gleeble-3800 thermomechanical simulator. The effects of the process parameters on recrystallized volume fraction, the precipitation and the interaction between precipitation, and recrystallization were analyzed to provide a reference for the prediction of the microstructural evolution and properties in the hot rolling process.



(a) (b)

FIGURE 1: The stress-strain curves in the double compression tests. (a) True strain $\varepsilon = 22\%$; (b) true strain $\varepsilon = 35\%$.



(a) (b)

FIGURE 2: The softening curves of the tested steel. (a) True strain $\varepsilon = 0.22$; (b) true strain $\varepsilon = 0.35$.

2. Experimental

The V-N microalloyed steel used in this study was melted in a vacuum induction furnace. The chemical composition is C 0.15, Mn 1.6, Si 0.3, S 0.037, P 0.051, Cu 0.05, Ni 0.1, V 0.05, Ti 0.02, N 0.0183, and balance Fe; all numbers are given in wt.%.

The double-compression isothermal simulation tests were performed by using Gleeble-3800 thermo-mechanical simulator. The samples were 8 mm in diameter and 12 mm in length. All the samples were reheated in vacuum to prevent oxidation and wrapped with tantalum foils on both sides to minimize conglutination.

The simulation test schedule involved austenitization at 1150°C for 3 min, followed by cooling at a rate of 5°C/s to the deformation temperature that was in the range of 800°C to 950°C and a hold for 60 s to homogenize the temperature within the samples. At the deformation temperature, the double compression tests were conducted with a strain rate of 5 s^{-1} and a strain of $\varepsilon_1 = 0.22$ or $\varepsilon_2 = 0.35$, respectively. Several holding times between 1 s and 500 s were used at the interval. In some samples, the second compression after the time interval was replaced by a water quench in order to study the precipitate and microstructure evolution.

3. Results and Discussion

3.1. Analysis of the Softening Behaviors. In general, the recrystallized fraction can be calculated by three methods that are the back extrapolation method, the mean flow stress method,

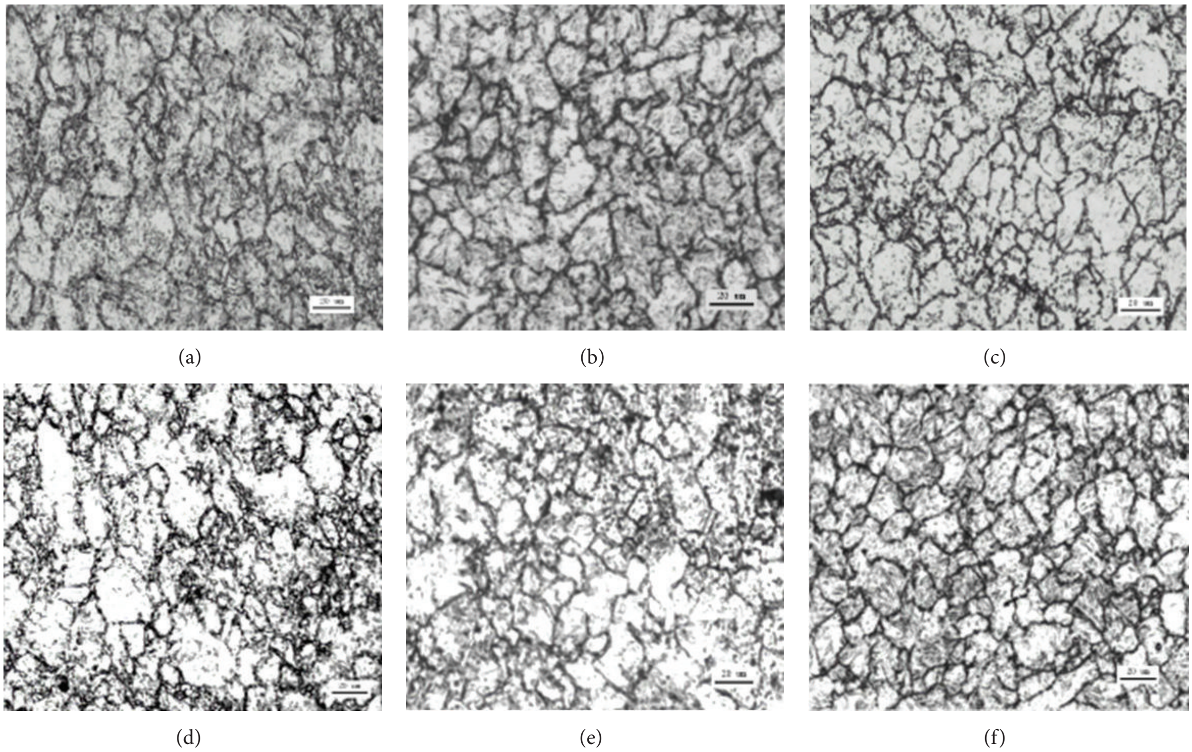


Figure 3: Microstructures obtained under different deformation conditions. (a) $T = 800^{\circ}$C, $\varepsilon = 0.22$, and $t = 10$ s; (b) $T = 800^{\circ}$C, $\varepsilon = 0.22$, and $t = 100$ s; (c) $T = 800^{\circ}$C, $\varepsilon = 0.22$, and $t = 200$ s; (d) $T = 850^{\circ}$C, $\varepsilon = 0.22$, and $t = 10$ s; (e) $T = 800^{\circ}$C, $\varepsilon = 0.35$, and $t = 10$ s; (f) $T = 850^{\circ}$C, $\varepsilon = 0.35$, and $t = 30$ s.

and the stress offset method. The latter one was used and the softening fraction X_s was calculated by using the following expression:

$$X_s = \frac{\sigma_m - \sigma_s'}{\sigma_m - \sigma_s}, \quad (1)$$

where σ_m is the flow stress corresponding to the end of the first pass, σ_s, and σ_s' are the stresses corresponding to the yield stresses in the first pass and the second pass, respectively.

By 0.002 offset method, the effect of the recovery is underestimated, which leads to larger softening fractions than the recrystallized fraction. In addition, the precisions in calculation are often disturbed by the stress-strain curves collected during the tests. 0.02 offset method is comparatively insensitive to recovery, and the softening fractions calculated by this method fit well to the recrystallized fractions [11]. Therefore, the 0.02 offset method was used to calculate the recrystallized fractions.

Figure 1 presents the stress-strain curves collected during the double compression tests. It can be seen that the softening behaviors change with the time intervals, and longer time intervals result in larger softening fractions. The softening fraction X_s versus time interval relations is shown in Figure 2, and the microstructures obtained under different deformation conditions are shown in Figure 3. In Figure 3(a), 3(b), and 3(c), it can be seen that the number of recrystallized grains increases with the increment of time interval. In consideration of the number of recrystallized grains in Figures 3(b) and 3(e), it can be said that large strain promotes recrystallization process. Furthermore, an increase in strain leads to a shorter time interval for the deformed austenite to complete recrystallization, which is recognizable in Figure 3(f).

In Figure 2, the curves display the sigmoid-shaped behavior. At the deformation temperature of 900°C or 950°C, the recrystallized fraction increases markedly with the increase of the time interval. For the tested steel deformed at a strain of 0.22, the recrystallized fraction increases markedly within 10 s, and the recrystallization process is almost completed when the time interval reaches 50 s. With an increase in strain to 0.35, the recrystallization process is promoted, and complete recrystallization is recognizable when the deformation temperature is 950°C and the time interval is around 10 s, which can be seen in Figure 2(b) and it is consistent with microstructural evolution in Figure 3. It can be explained that an increase in strain leads to an increase in dislocation density and defects in the material, which contributes to the store energy accumulation that plays a role as the recrystallizing driving force. Especially, the effect of strain on recrystallization process is great at the early stage. At the deformation temperature of 950°C and 1 s time interval, true strains of 0.22 and 0.35 were applied. And the corresponding recrystallized fractions are 32.7% and 82%, respectively.

The effect of strain on the softening behavior in microalloyed steel can be expressed in two ways. On one hand, an

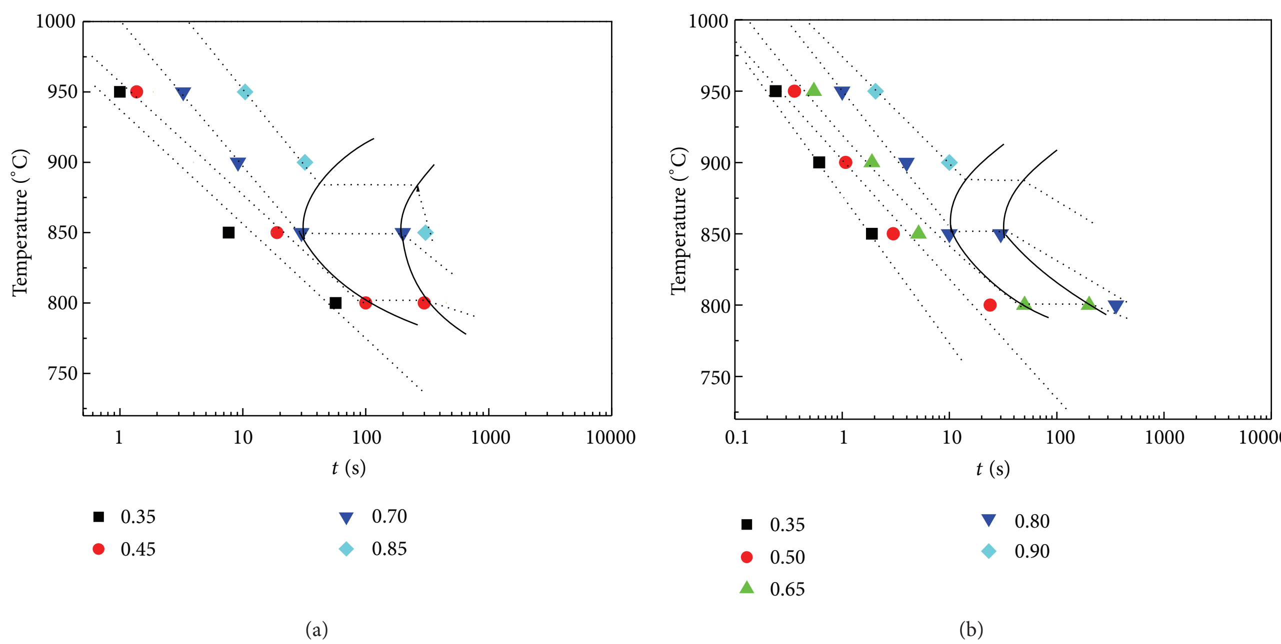


Figure 4: RPTT diagrams for the tested steel. (a) True strain $\varepsilon = 0.22$; (b) true strain $\varepsilon = 0.35$.

increase in strain promotes the grain boundary migration and as a consequence, the recrystallization process is accelerated; on the other, it is favorable for some microalloying elements to precipitate at certain temperature, and the precipitates play a pinning effect, which inhibits the recrystallization process. It has been proved that microalloying element vanadium in dissolution or precipitation plays a different role in steel [12]. It is favorable for the carbonitride of vanadium to precipitate under proper hot working conditions [13], which inhibits the recrystallization, and a resultant plateau is displayed in the recrystallized faction versus time diagram. This phenomenon also can be seen in the present work in Figure 2, as the plateaus are consistent with the formation of the precipitates that inhibit the recrystallization. And the precipitation behaviors of carbonitride of vanadium are to be discussed in details. In addition, the samples deformed at a strain of 0.22 or 0.35 exhibit a similar performance at the deformation temperature of 850°C or 800°C. However, the effect of strain on the incubation time for the precipitates is different, and an increase in strain promotes the precipitation to occur earlier.

3.2. Analysis of Precipitation. Figure 4 presents the recrystallization-precipitation-time-temperature (RPTT) diagrams resulting from the relationship between temperature and time corresponding to different recrystallized fractions. The points that define the start and the end of the plateau were taken from the data in Figure 2. In Figure 4, the recrystallized fraction changes at a small amplitude between the precipitate start and finish curves that show "C" shapes, the curves in solid, and the nose of the precipitates is located around 850°C. In addition, an increase in strain leads the minimum incubation time of the precipitates to reduce from 30 s to 10 s, and the precipitate finish time also shifts toward shorter times.

The TEM images of precipitates at the deformation temperature of 850°C are shown in Figures 5 and 6. The precipitates in the initial holding are quadrate and quite similar regardless of the strain of 0.22 or 0.35. The precipitates are mainly about titanium carbonitride by energy dispersive spectroscopy (EDS) analysis. Figure 5(a) presents the TEM image of the sample deformed at strain of 0.2. The corresponding EDS image is shown in Figure 6(a). After longer holding, the particles are spherical and about several to tens of nanometers in size, Figure 5(b). The corresponding EDS image is shown in Figure 6(b), and the particles are mainly about carbonitrides of titanium and vanadium. So these precipitates that restrain recrystallization process by pinning effect are the main reason for the formation of the plateaus in Figure 2. The precipitation process exhibits a similar performance with an increase in strain. However, the precipitated particles are even smaller in size but decrease in number. It is reported that vanadium precipitates nucleate preferentially at dislocation [14]. An increase in strain contributes to greater work hardening and higher density of dislocation. Therefore, more nucleation sites for vanadium precipitates are formed. While the precipitates are dissolving due to being above the VC dissolution temperature after strain induced precipitation, the number of precipitates decreases with 30 more seconds holding, which leads to an increase in recrystallized fraction after the plateau, and the pinning forces of the precipitates must be lower than the driving forces for recrystallization. In addition, an increase in strain also promotes the recovery and recrystallization process that is to reduce dislocation density sharply, and as a consequence, the nucleation sites for vanadium precipitates are decreased.

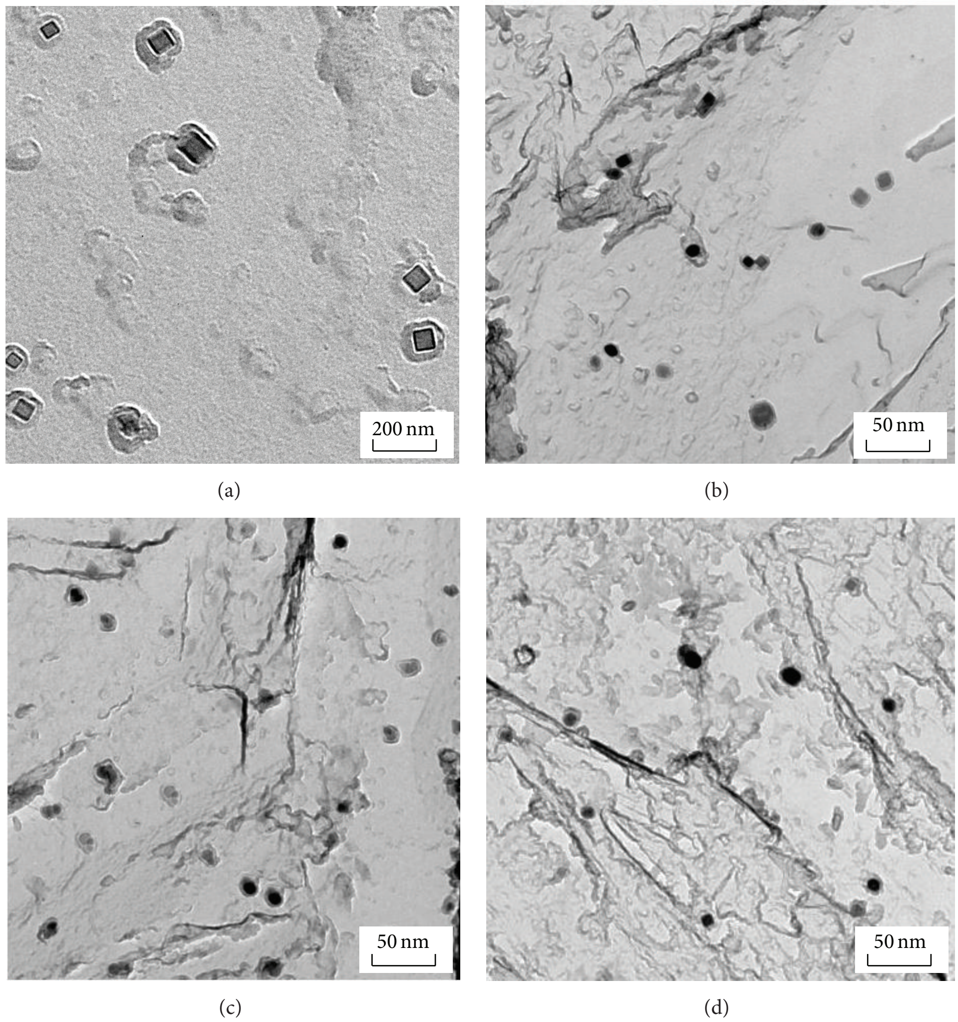


Figure 5: The precipitations under different deformation conditions. (a) $\varepsilon = 0.22$, $t = 10$ s; (b) $\varepsilon = 0.22$, $t = 50$ s; (c) $\varepsilon = 0.35$, $t = 20$ s; (d) $\varepsilon = 0.35$, $t = 50$ s.

In consideration of the effect of strain on precipitation and recrystallization, the effect of strain on recrystallization plays a dominant role, which leads to a decrease in nucleation sites for vanadium precipitates and a decrease in the number of the precipitates, Figure 5(d). It can be predicted that the pinning effect of the precipitates will not play a role in recrystallization after strain is increased to a critical strain. Later work should be done to prove whether the critical strain is equal to the critical strain for dynamic recrystallization. Once it is proved to be true, the present work is to be consistent with Chen's work [15].

3.3. Static Recrystallization Model. The static recrystallization kinetics of austenite are usually described by an Avrami equation in the following way [16]:

$$X_s = 1 - \exp\left[-B\left(\frac{t}{t_F}\right)^{n_A}\right], \quad (2)$$

where t_F is the time corresponding to F recrystallization fraction, $B = -\ln(1-F)$, and n_A is a constant independent of deformation conditions

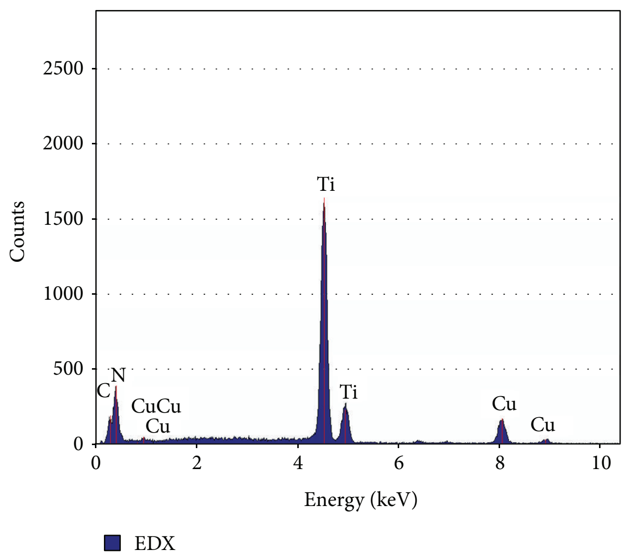
With "F" equal to 50%, the equation can be expressed in the following way:

$$X_s = 1 - \exp\left[-0.693\left(\frac{t}{t_{0.5}}\right)^{n_A}\right]. \quad (3)$$

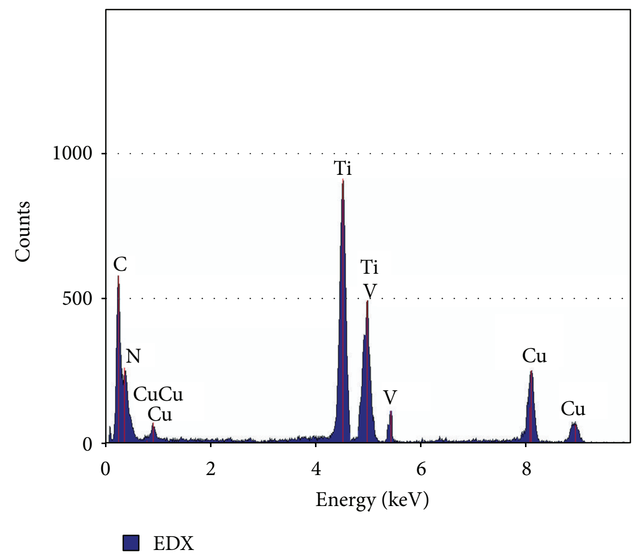
By taking the natural logarithm two times on both sides of (3), the equation is as follows:

$$\ln\ln\frac{1}{1-X_s} = n_A \ln\frac{t}{t_{0.5}} + \ln c. \quad (4)$$

In (4), $\ln\ln(1/(1-X_s))$ versus $\ln(t/t_{0.5})$ relation is linear on condition that n_A is a constant. According to some authors, the value of n_A depends on temperature [17]. And it depends on the pinning effects of the precipitates in consideration of the softening behaviors of the tested steel. At the temperature of 950°C or 900°C, n_A is calculated by 1east squares method, $n_A = 0.6112$, shown in Figure 7.



(a)



(b)

FIGURE 6: EDS image of the precipitates. (a) $\varepsilon = 0.22$, $t = 10$ s; (b) $\varepsilon = 0.22$, $t = 30$ s.

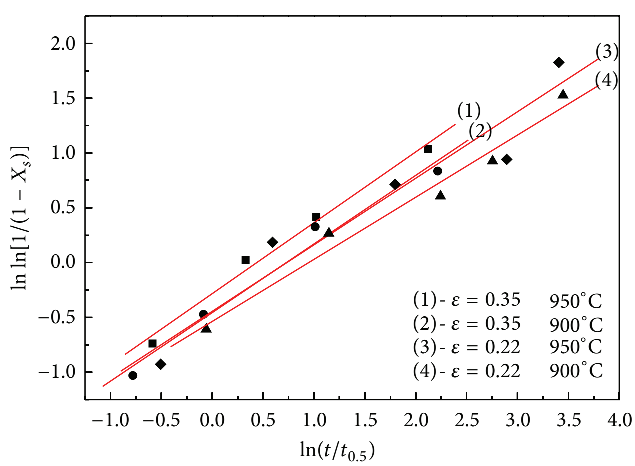


FIGURE 7: The relation between $\ln\ln[1/(1-X_s)]$ and $\ln(t/t_{0.5})$.

The static recrystallization kinetic equation is expressed as follows:

$$X_s = 1 - \exp\left[-0.693\left(\frac{t}{t_{0.5}}\right)^{0.6112}\right]. \tag{5}$$

Previous studies have shown that $t_{0.5}$ can be used to determine the activation energy, as expressed by the following equations:

$$t_{0.5} = A\varepsilon^{p}\dot{\varepsilon}^{q}\exp\left(\frac{Q_{rex}}{RT}\right)$$
$$\ln t_{0.5} = \ln A + p\ln\varepsilon + q\ln\dot{\varepsilon} + \frac{Q_{rex}}{RT}. \tag{6}$$

As proved elsewhere [18], activation energy Q_{rex} is mainly affected by the chemical composition of materials and independent of deformation conditions. Under certain deformation conditions, $\ln t_{0.5}$ versus $1/T$ relation is linear. Therefore, activation energy Q_{rex} can be obtained by linear regression. It is worth to note that there are vanadium precipitates in the tested steel at 850°C or 800°C, which leads to a nonlinear relation between $\ln t_{0.5}$ and $1/T$. In consideration of the presence of precipitates, activation energy Q_{rex} is expressed in the following way:

$$Q_{rex} = Q + \Delta Q, \tag{7}$$

where Q is the activation energy in the absence of precipitates and ΔQ is the increase in activation energy due to the precipitates.

By piecewise linear fitting method, the activation energy in the absence of precipitates was $Q = 242.43$ kJ/mol. As described above, the effect of strain on recrystallization or precipitation process was different. At the strain of 0.22, the increase in activation energy due to the pinning effect of the precipitates was $\Delta Q_1 = 256.41$ kJ/mol while at a larger strain of 0.35, the value of ΔQ_2 was only 81.78 kJ/mol. Therefore, larger strain is liable to reduce the pinning effect of the precipitates and leads to smaller activation energy, which is consistent with the analysis of precipitation.

4. Conclusions

(1) Double compression tests were performed on a Gleeble-3800 thermo-mechanical simulator. Based on the true stress-true strain curves of the samples deformed at the temperature of 950°C or 900°C, the exponent n_A in Avrami's law was calculated by least squares method and it was 0.6112. The corresponding static recrystallization model was obtained.

(2) The static recrystallization softening curves were of sigmoidal shape at the temperature of 950°C or 900°C while the curves presented plateaus due to the pinning effects of the vanadium and titanium precipitates at the temperature of 850°C or 800°C, regardless of the strain of 0.22 or 0.35. An increase in strain promotes the recrystallization and precipitation processes. Especially, at the temperature of 850°C, the increase in strain reduces the inhibition effect of precipitation on recrystallization.

(3) By the calculation of activation energy, the increase in activation energy due to the pinning effect of the precipitates was 256.41 kJ/mol corresponding to a strain of 0.22 while the increase in activation energy was 81.78 kJ/mol corresponding to a larger strain of 0.35. Therefore, larger strain is liable to reduce the pinning effect of the precipitates and leads to smaller activation energy, which is consistent with the analysis of the interaction between precipitation and recrystallization.

Conflict of Interests

The authors do not have any possible conflict of interests with any trademark mentioned in the paper.

Acknowledgment

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References

[1] T. Gladman, *The Physical Metallurgy of Microalloyed Steels*, The Institute of Materials, The University Press, London, UK, 1997.

[2] L. Haibo, L. Hongyu, and C. Hao, "Microstructure refinement in a non-quenched and tempered vanadium nitrogen steel," *Transactions of Materials and Heat Treatment*, vol. 33, no. 4, pp. 85–90, 2012.

[3] M. Gomez, S. F. Medina, and J. I. Chaves, "Static recrystallization of austenite in a medium-carbon vanadium microalloyed steel and inhibition by strain-induced precipitates," *Materials Science Forum*, vol. 550, pp. 417–422, 2007.

[4] A. M. Elwazri, E. Essadiqi, and S. Yue, "Kinetics of metadynamic recrystallization in microalloyed hypereutectoid steels," *ISIJ International*, vol. 44, no. 4, pp. 744–752, 2004.

[5] S. F. Medina, A. Quispe, and M. Gomez:, "Strain induced precipitation effect on austenite static recrystallisation in microalloyed steels," *Materials Science and Technology*, vol. 19, pp. 99–108, 2003.

[6] J.-S. Byun, J.-H. Shim, J.-Y. Suh et al., "Inoculated acicular ferrite microstructure and mechanical properties," *Materials Science and Engineering A*, vol. 319-321, pp. 326–331, 2001.

[7] Z. Ma, D. Peisker, and D. Janke, "Grain refining of structural steels by dispersion of fine oxide particles," *Steel Research*, vol. 70, no. 4, pp. 178–182, 1999.

[8] C. Yang and Y. Zhang, "Applications of V-N microalloying technology in HSLA steel," *Iron and Steel*, vol. 37, no. 11, pp. 42–47, 2002.

[9] C. Yang and Y. Zhang, "Role of Nitrogen in non-quenched and tempered steel," *Iron Steel Vanadium Titanium*, vol. 21, no. 3, pp. 16–22, 2000.

[10] S. Bangming, J. Huaizhong, Y. Caifu, and Z. Yongquan, "Precipitation behavior of vanadium in V-N microalloyed steel," *Iron and Steel*, vol. 36, no. 2, pp. 44–47, 2001.

[11] A. I. Fernández, B. López, and J. M. Rodríguez-Ibabe, "Relationship between the austenite recrystallized fraction and the softening measured from the interrupted torsion test technique," *Scripta Materialia*, vol. 40, no. 5, pp. 543–549, 1999.

[12] A. M. Sege, *The Role of Vanadium in HSLA Steel. Application of Vanadium in Steel*, CSM, Beijing, China, 1992.

[13] M. Gómez, L. Rancel, and S. F. Medina, "Effects of aluminium and nitrogen on static recrystallisation in V-microalloyed steels," *Materials Science and Engineering A*, vol. 506, no. 1-2, pp. 165–173, 2009.

[14] F. Fang, Q. Yong, and C. Yang, "Precipitation behavior in vanadium micro-alioyed steel," *Iron and Steel*, vol. 45, no. 3, pp. 66–69, 2010.

[15] C. Liqing, Z. Yang, X. Xiangqiu, and L. Xianghua, "Dynamic recrystallization and precipitation behaviors of a kind of low carbon V-microallyed steel," *Acta Metallurgica Sinica*, vol. 46, no. 10, pp. 1215–1222, 2010.

[16] E. Anelli, "Application of mathematical modelling to hot rolling and controlled cooling of wire rods and bars," *ISIJ International*, vol. 32, no. 3, pp. 440–449, 1992.

[17] P. Choquet, A. le Bon, and C. Perdrix, in *Proceedings of the 7th International Conference on Strength of Metals and Alloys*, H. J. McQueen, J. P. Bailon, J. I. Dickson, J. J. Joans, and M. G. Akben, Eds., vol. 2, pp. 1025–1030, Pergamon Press, Oxford, UK, 1985.

[18] A. M. Elwazri, E. Essadiqi, and S. Yue, "The kinetics of static recrystallization in microalloyed hypereutectoid steels," *ISIJ International*, vol. 44, no. 1, pp. 162–170, 2004.