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EFFECT OF NITROGEN ON THE DYNAMIC RECRYSTALLIZATION BEHAVIORS OF VANADIUM AND TITANIUM MICROALLOYED STEELS

The hot deformation behaviors of vanadium and titanium microalloyed steels containing different nitrogen contents were studied by performing hot compression tests at various temperatures ranging from 900 to 1050°C and strain rates ranging from 0.1 to 10 s^{-1}. The flow stress curves of the experimental steels were analyzed and the effect of nitrogen on the hot deformation behaviors of the vanadium titanium and nitrogen microalloyed steels was discussed. The results reveal that the flow stress increases with increasing nitrogen addition and the critical strain for the onset of dynamic recrystallization(DRX) also increases by adding nitrogen. Therefore, larger strain should be applied to start DRX in the experimental steel containing higher nitrogen content. The material constants and activation energies for hot deformation were determined by regression method and the effect of nitrogen on the activation energy was also discussed referring to the activation energies from the previous researches. It is found that higher nitrogen content contributes to higher activation energy for hot deformation. Furthermore, the DRX kinetics models for the experimental steels were constructed by regression method and the effect of nitrogen on the DRX rate under various deformation conditions was analyzed. And the inhibition of DRX by increasing nitrogen content is confirmed.

Keywords: vanadium titanium and nitrogen microalloyed steels; dynamic recrystallization; effect of nitrogen; critical strain; hot deformation behavior

1. Introduction

Microalloying technology contributes to achieving significant cost and energy saving. By material design and improving material forming process, steel products with high performances can be produced [1-6]. The concentrations of microalloying elements have marked effects on austenite recrystallization and growth because they form alloy carbides and nitrides during hot rolling that pin grain boundaries [7,8].Vanadium is often known as one of effective strengthening elements. It can be resolved in steel material at a lower temperature, which is favorable for a lower reheating temperature and results in smaller austenite grains. And vanadium plays an inhibition role in austenite grain growth or recrystallization during rolling process, which also contributes to smaller grains and refines the transformed ferrite [9,10]. Furthermore, carbonitrides of vanadium can precipitate during cooling and enhance the strength of the products by precipitation strengthening. Thus, vanadium microalloyed steels have attracted much attention [11,12]. Over the past decades, nitrogen was regarded as a harmful element due to its connection with low resistance to brittle fracture, so nitrogen was often decreased in steel. However, it has been found that the interaction between nitrogen and alloy elements can play a positive role in metal forming process, which leads to the production of various steels containing high nitrogen contents. Especially, the interaction between nitrogen and vanadium can make full use of vanadium in grain refinement and precipitation strengthening. In this case, nitrogen is regarded as a cost effective microalloying element [13,14]. So there is a significant amount of researches on high strength steel production by vanadium nitrogen micoalloying technology. Since dynamic recrystallization (DRX) process contributes to grain refinement and lower stress, which plays an important role in the formability, microstructure and mechanical properties of materials, it is very important to know the process. At present, there are some researches on the DRX behavior of vanadium microalloyed steels or vanadium and titanium microalloyed steels [15-17]. However, there is no systematical research on vanadium titanium and nitrogen microalloyed steels. Moreover, chemical composition has a great impact on the flow behaviors or phase transformation behaviors of materials. Therefore, it is of great practical importance to study the effect of nitrogen on the DRX behaviors of vanadium titanium and nitrogen microalloyed steels.

In the present work, three vanadium and titanium microalloyed steels containing different nitrogen contents were tested by performing uniaxial hot compression tests on a Gleeble-3800 thermo-mechanical simulator. Based on the flow curves, the

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characteristic parameters and mathematical models for the experimental steels were obtained and the effect of nitrogen contents on DRX behaviors of the steels was analyzed.

2. Material and experimental

Chemical compositions of the experimental steels are shown in Table 1.

TABLE 1

Chemical composition of the tested steels (wt %)

Steel No.	C	Mn	Si	V	Ti	Ni	N	S≤	P≤
LN	0.13	1.54	0.34	0.047	0.01	0.1	0.004	0.02	0.06
MN	0.13	1.6	0.3	0.05	0.02	0.1	0.015	0.03	0.05
HN	0.14	1.57	0.3	0.049	0.02	0.11	0.02	0.03	0.05

The experimental steels were melted and casted into an ingot of 200 kg in vacuum induction furnace. Cylindrical specimens were machined with a diameter of 10mm and a height of 12 mm. In order to reduce the occurrence of inhomogeneous compression, special anvils were employed. Both ends of the specimen were covered with tantalum foils to prevent adhesion between specimen and anvils. The specimens were austenitized at 1150°C for 5 min and cooled at the rate of 5°C/s to deformation temperatures and held there for 1 min before compression. Specimens were compressed to a true strain of 0.8 at the temperature ranging from 900 to 1050°C with an interval of 50°C and strain rates of 0.1, 1, 3 and 10 s^{-1}.

3. Results and discussion

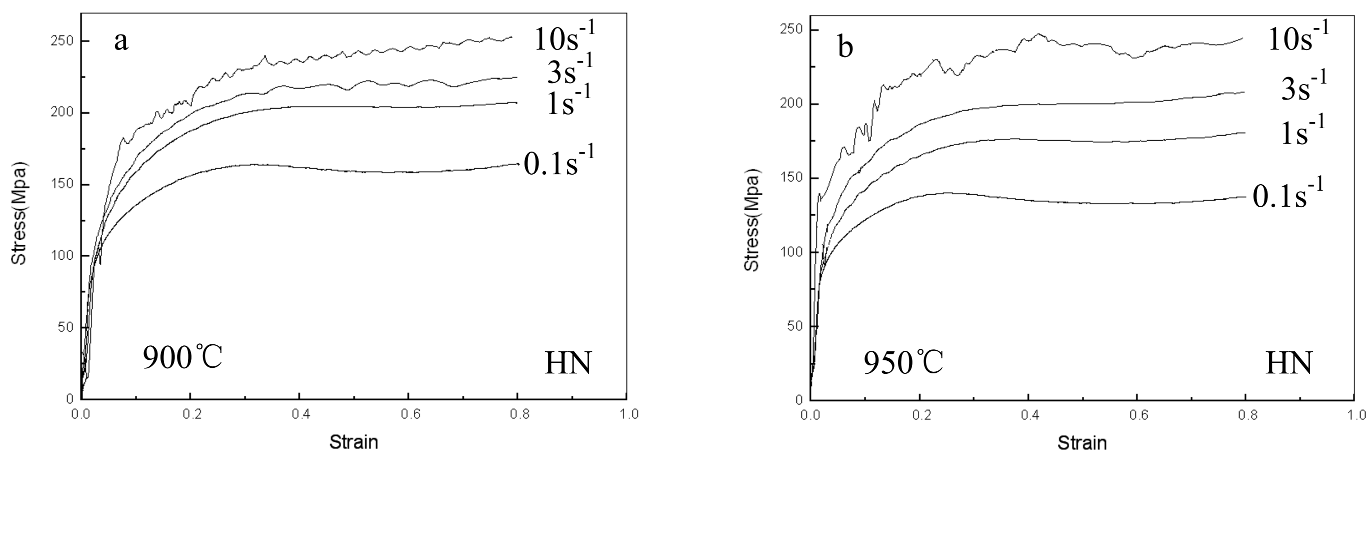
3.1. Flow curves

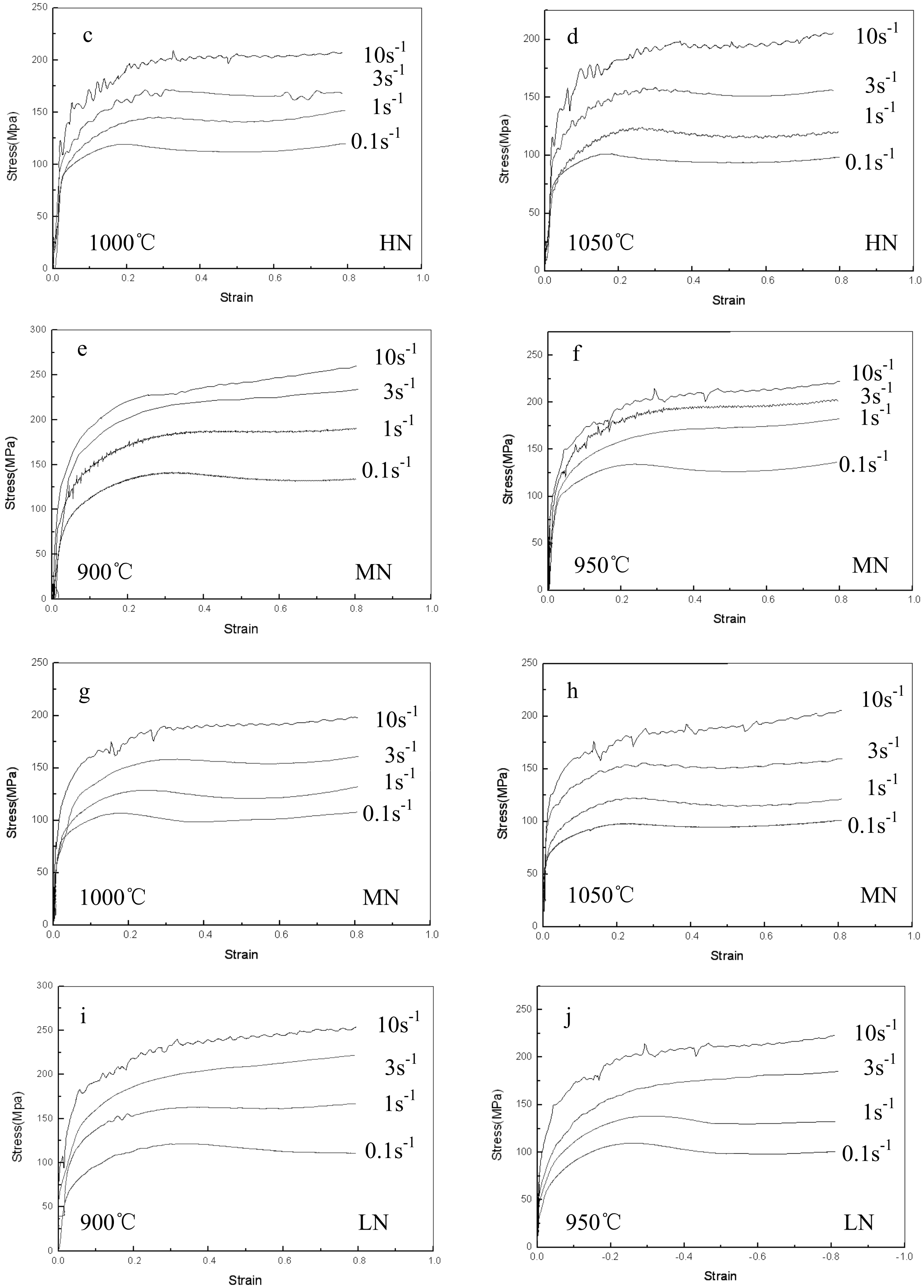
Fig. 1 presents the flow curves of the three experimental steels deformed under different deformation conditions. LN, MN and HN are designated as low nitrogen, medium nitrogen and high nitrogen respectively. To facilitate the comparison, the curves of the three steels deformed at different temperatures and strain rates were redrawn in Fig. 1(m) and (n).

In Fig. 1(m) and (n), it can be seen that the peak stress σ_p and its corresponding strain ε_p, the peak strain, decrease with an increase in deformation temperature. The main reason is that the driving force for atomic vacancies diffusion, dislocation slipping and rearranging increases with increasing temperature, which reduces strain hardening effect at the early stage of deformation. As a result, a low peak stress is recognizable on the flow curve. The effect of strain rate on the peak stress is shown in Fig. 1n. Higher strain rate leads to higher peak stress and larger peak strain. It can be explained by the concurrent processes of hardening and softening. The hardening process is due to dislocation generation or accumulation while the softening process is due to dislocation rearrangement or elimination. The time for dislocation rearrangement or elimination is short under high strain rate. Therefore, dislocation accumulation plays a dominant role, which results in the increase in dislocation density and higher stress. When larger strain is applied, dislocation rearrangement or elimination plays the main role, which contributes to the occurrence of DRX and the curves exhibit peaks. It can also be seen that the effect of nitrogen on the stress is marked and an increase in nitrogen contents in the experimental steels leads to peak stress or strain increasing, which will be discussed in details below.

3.2. Effect of nitrogen on peak stress or strain

Fig.2 presents the peak stress or strain of the experimental steels deformed at various temperatures and strain rates of 0.1 s^{-1} and 1 s^{-1}. Under the same deformation conditions, the peak stresses or strains increase with increasing nitrogen content. In Fig. 2, it can be seen that higher nitrogen content contributes to higher flow stress. As the chemical compositions of the experimental steels are considered, vanadium or titanium carbonitrides can precipitate under the deformation conditions [18] and these precipitated particles play a pinning effect on dislocations or grain boundaries. Moreover, an increase in nitrogen content can promote the precipitation process [19] and consequently, the pinning effect becomes stronger. So the inhibition of dynamic




c
10s-1
3s-1
1s-1
0.1s-1
1000℃
HN
Stress(Mpa)
Strain
d
10s-1
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1s-1
0.1s-1
1050℃
HN
Stress(Mpa)
Strain
e
10s-1
3s-1
1s-1
0.1s-1
900℃
MN
Stress(MPa)
Strain
f
10s-1
3s-1
1s-1
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950℃
MN
Stress(MPa)
Strain
g
10s-1
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0.1s-1
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MN
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h
10s-1
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1050℃
MN
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Strain
i
10s-1
3s-1
1s-1
0.1s-1
900℃
LN
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j
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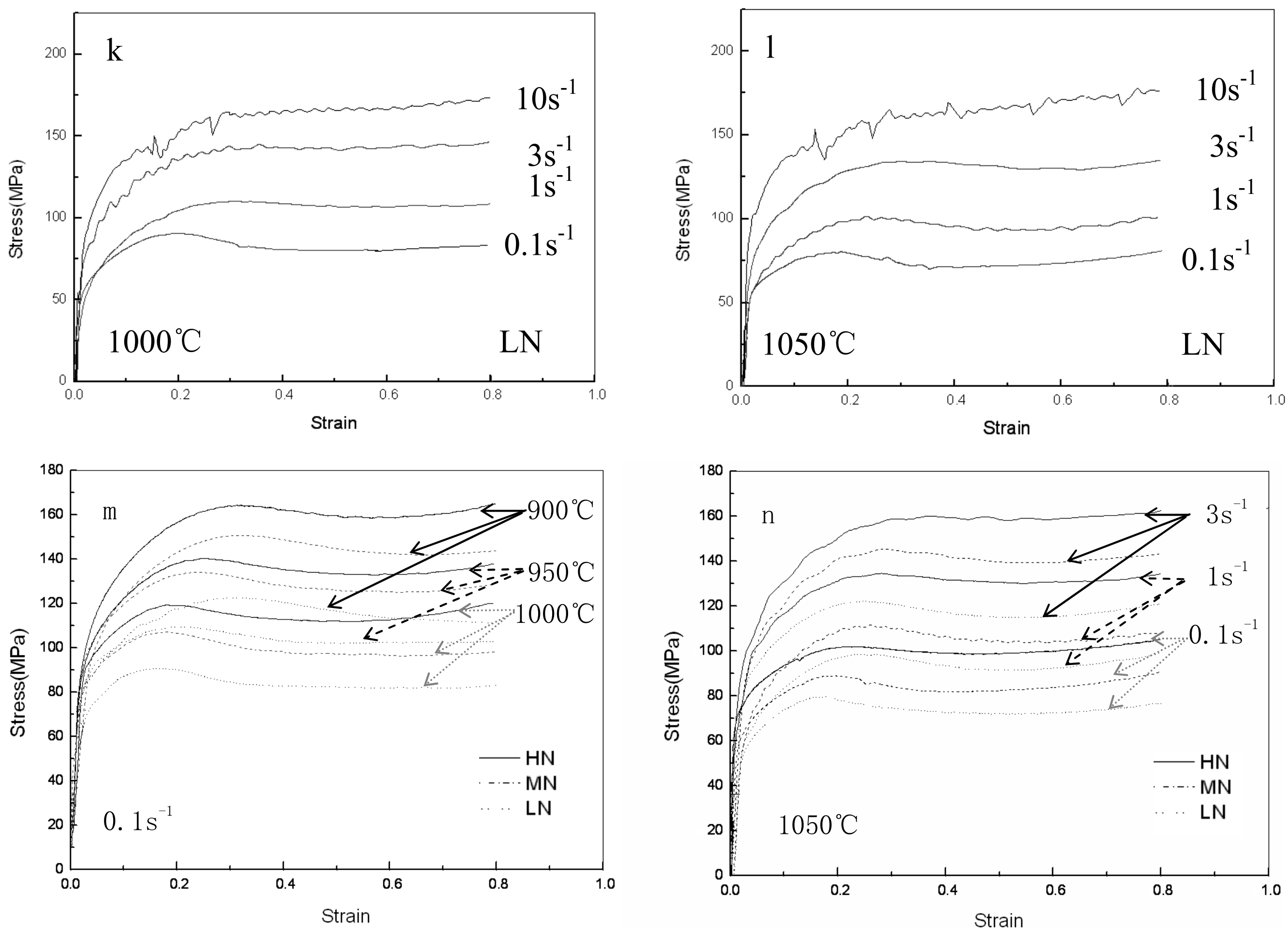


Fig. 1. True stress-strain curves of the experimental steels deformed under various deformation conditions
(a), (b), (c) and (d) Curves for high nitrogen steel (HN);
(e), (f), (g) and (h) Curves for medium nitrogen steel (MN);
(i), (j), (k) and (l) Curves for low nitrogen steel (LN);
(m) and (n) Comparison of the curves for the experimental steels

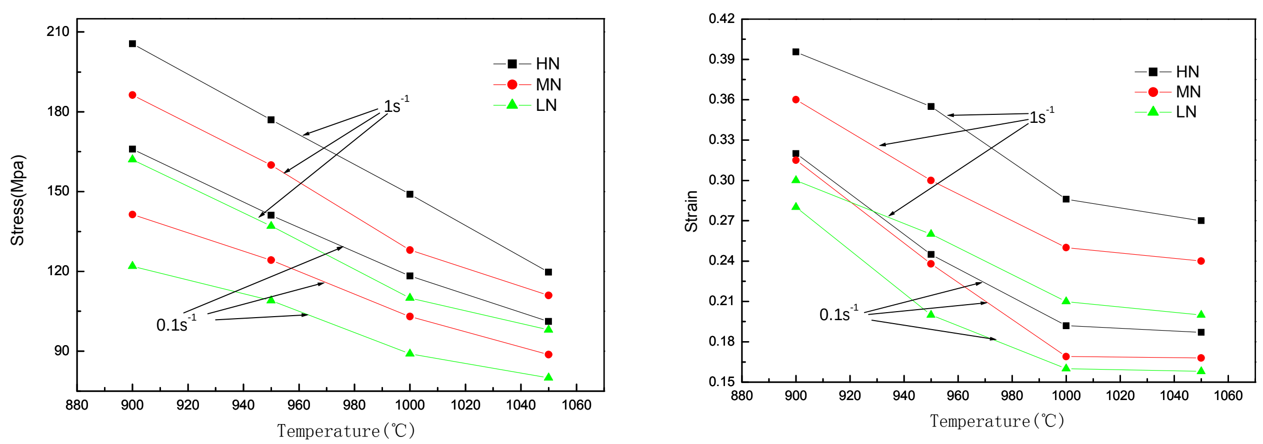


Fig. 2. Relation between temperature and peak stress (a) or peak strain (b)

recovery and DRX process becomes marked due to the increasing nitrogen content. Thus, the flow stress increases including the peak stress. In Fig. 2, it can also be seen that the effect of nitrogen on peak stress decreases with increasing temperature. The effect of temperature on precipitation process must be considered. It has been proved that vanadium carbonitrides can precipitate in a specific temperature range and the quantity of the precipitated particles has a close relation with the temperature [20]. With decreasing temperature, $V(C,N)$ precipitation process occurs. And the favorable temperature for the precipitation process is around 850°C [20]. Since the experimental temperatures are all above the value, it can be concluded that the quantity of precipitated particles decreases with increasing temperature. Therefore, the pinning effect of the precipitated particles on dislocations or grain boundaries is reduced and the effect of nitrogen on peak stress becomes weak.

3.3. Effect of nitrogen on critical conditions

The critical conditions are significant parameters for the onset of DRX. It is very important to find out the critical conditions for online production control. Many works on the determination of the critical conditions have been reported. One of the approaches [21] proposed by Poliak and Jonas has been widely used [22-24]. And they have proposed the critical condition kinetics based on the thermal irreversible principles and the critical conditions can be determined by analyzing the relation between strain hardening rate and flow stress. The strain hardening rate is the first derivative of the stress with respect to the strain. This approach was used in the present work to determine the critical conditions for the experimental steels. Fig. 3 presents the relation between strain hardening rate and flow stress.

In Fig. 3, the inflection points on the curves can be identified as the critical points and the stresses corresponding to the points are the critical stresses. Moreover, the stresses corresponding to zero points indicate the peak stresses. The peak stress σ_p (peak strain ε_p) versus critical stress σ_c (critical strain ε_c) relationships are shown in Fig. 4. The data for each experimental steels exhibit a high linear relationship. By linear regression, the correlation coefficients R for each line are shown in table 2.

TABLE 2

Correlation coefficients for each line

Steels	R (ε_p versus ε_c)	R (σ_p versus σ_c)
LN	0.9937	0.9991
MN	0.9989	0.9983
HN	0.9905	0.9993

The following equations can be obtained:
LN steel:

$$\sigma_c = 0.8768\sigma_p \tag{1}$$

$$\varepsilon_c = 0.6250\varepsilon_p \tag{2}$$

MN steel:

$$\sigma_c = 0.9027\sigma_p \tag{3}$$

$$\varepsilon_c = 0.5618\varepsilon_p \tag{4}$$

HN steel:

$$\sigma_c = 0.9337\sigma_p \tag{5}$$

$$\varepsilon_c = 0.5155\varepsilon_p \tag{6}$$

In the previous works [25,26], the linear relationship between σ_c (ε_c) and σ_p (ε_p) is commonly accepted, but there is still no detailed explanation for the slope of the line. According to the analysis above, the ε_c to ε_p ratio is not limited to the values between 0.6 and 0.8. In addition, low values for some steels have also been reported. So it may be related to the hot deformation behaviors of the material. The experimental steels containing different nitrogen contents exhibit different flow behaviors,

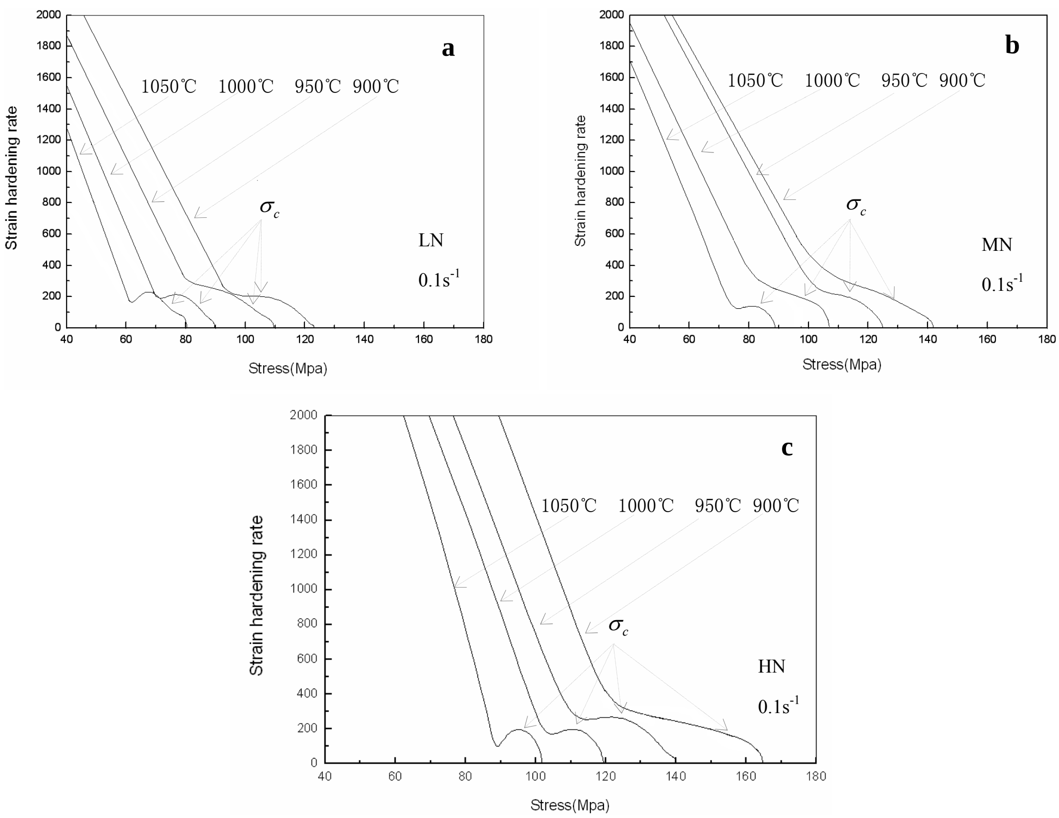
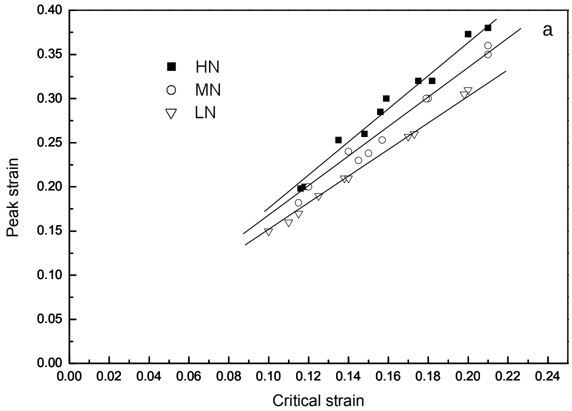


Fig. 3. Strain hardening versus stress curves of the tested steel (a) low nitrogen steel (b) medium nitrogen steel (c) high nitrogen steel



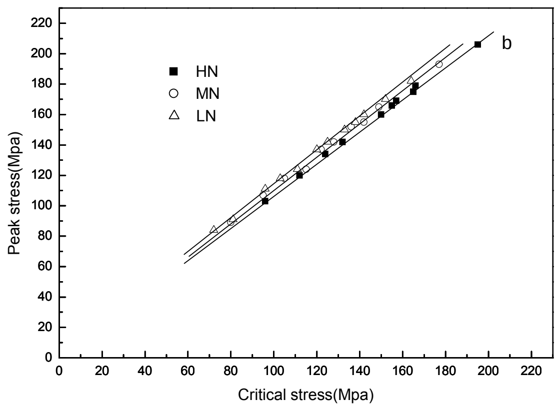


Fig. 4. Relationship between (a) critical stress and peak stress (b) criticalstrain and peak strain

which may be the reason for the ε_c to ε_p ratio to decrease with increasing nitrogen content. However, the peak strain increases with increasing nitrogen content (Fig. 2n). The resultant critical strains increase with increasing nitrogen content. Therefore, larger strain should be applied to start DRX for the steels containing higher nitrogen content.

3.4. Effect of nitrogen on activation energy for hot deformation

Activation energy for hot deformation serves as an indicator of deformation difficulty degree for plasticity deformation. The analysis of the effect of nitrogen on the hot deformation behaviors of the experimental steels reveals that adding nitrogen can change the activation energy of the vanadium and titanium microalloyed steels. Under isothermal compression conditions, the relation between temperature and strain rate on the deformation behaviors can be represented by the Zener-Hollomon parameter as:

$$Z = \dot{\varepsilon}\exp\left(\frac{Q}{RT}\right) \tag{7}$$

Where, $\dot{\varepsilon}$ is the strain rate, S^{-1}; T is the deformation temperature, K; Q is the activation energy, J mol^{-1}; R is the gas constant, 8.314 J mol^{-1}K^{-1}

According to the dependence of temperature, strain rate and stress, Sellars has proposed the hyperbolic sine law [27,28] that can give better relations between the Z parameter and flow stress. The equation is given as:

$$A\left[\sinh(\alpha\sigma_p)\right]^n = \dot{\varepsilon}\exp\left(\frac{Q}{RT}\right) \tag{8}$$

To obtain parameter α, the following equations are also used:

$$A'\sigma_p^{n'} = \dot{\varepsilon}\exp\left(\frac{Q}{RT}\right) \tag{9}$$

$$A''\exp(\beta\sigma_p) = \dot{\varepsilon}\exp\left(\frac{Q}{RT}\right) \tag{10}$$

Where, A, A', A'', n', β, α and n are the material constants respectively.

Taking natural logarithms on both sides of Eq. (8), (9)and (10) respectively, the results are as follows:

$$\ln A' + n'\ln\sigma_p = \ln\dot{\varepsilon} + \frac{Q}{RT} \tag{11}$$

$$\ln A'' + \beta\sigma_p = \ln\dot{\varepsilon} + \frac{Q}{RT} \tag{12}$$

$$\ln\dot{\varepsilon} + \frac{Q}{RT} = \ln A + n\ln[\sinh(\alpha\sigma_p)] \tag{13}$$

And the method to process the data can be found elsewhere [17,29]. By linear regression, the activation energy for hot deformation and material constants for the experimental steels can be obtained. The results are given in Table 3.

TABLE 3

Activation energies for hot deformation and constants of the tested steels

Steels	*Q*	*n'*	*β*	*α*	*n*	*A*
LN	270.493	7.870	0.0625	0.0079	5.952	7.849×10^{10}
MN	293.228	8.091	0.0571	0.0071	6.061	6.922×10^{11}
HN	314.739	8.695	0.0526	0.0060	6.173	4.794×10^{12}

The activation energies for the experimental steels were determined as 270.493, 293.228 and 314.739 kJ mol^{-1} respectively. It can be seen that the activation energy for hot deformation increases with increasing nitrogen content, which is consistent with the effect of nitrogen on the critical stress or strain. The activation energy for hot deformation for LN steel is very close to austenite lattice self-diffusion activation energy and close to that for C-Mn steel in Ref. [30]. While the activation energy for hot deformation for MN steel is determined as 293.228 kJ mol^{-1} and higher than that for vanadium nitrogen microalloyed steel by other researchers [16]. Since chemical composition has great impact on activation energy, it is suggested that the difference results from the vanadium and nitrogen content. It has been reported that activation energy for hot deformation can be increased by vanadium addition [31] while no effect of vanadium addition is also reported. It can be concluded that nitrogen plays the role in enhancing the activation energy for hot deformation. The activation energy for HN steel is determined as 314.739 kJ mol^{-1}, which confirms the conclusion.

Moreover, the hot working equations for the experimental steels can be expressed as:

LN:

$$\dot{\varepsilon} = 7.85\times 10^{10}[\sinh(0.0079\sigma_p)]^{5.95}\exp\left(-\frac{270493}{RT}\right) \quad (14)$$

MN:

$$\dot{\varepsilon} = 6.92\times 10^{11}[\sinh(0.0071\sigma_p)]^{6.06}\exp\left(-\frac{293228}{RT}\right) \quad (15)$$

HN:

$$\dot{\varepsilon} = 4.79\times 10^{12}[\sinh(0.0060\sigma_p)]^{6.17}\exp\left(-\frac{314739}{RT}\right) \quad (16)$$

3.5. Effect of nitrogen on DRX volume fraction

At present, there are two ways to calculate DRX volume fraction: one is metallograph observation and the other is modeling calculation. The latter one can be expressed as:

$$X = \frac{\sigma_p - \sigma}{\sigma_p - \sigma_s} \quad (17)$$

Where, σ_s is the steady state stress, σ_p is the peak stress and σ is the specific stress from flow curves

The equation is described in details elsewhere [32,33]. As metallograph observation involves much experimental work, Eq. (17) is often used to calculate DRX volume fraction. To modeling DRX volume fraction for the experimental steels, the following kinetics model is used [34,35]:

$$X = 1 - \exp\left(-k\left(\frac{\varepsilon - \varepsilon_c}{\varepsilon_p}\right)^n\right) \quad (18)$$

Where, k and n are the material constants.

At the early stage of softening process, ε_c can be replaced by ε_p. And taking natural logarithms on both sides of Eq. (18) two times and Eq. (19) can be obtained.

$$\ln\ln\left(\frac{1}{1-X}\right) = \ln k + n\ln\left(\frac{\varepsilon - \varepsilon_p}{\varepsilon_p}\right) \quad (19)$$

By linear regression, the parameters of n and k for the experimental steels can be obtained and shown in Table 4.

TABLE 4

Parameters n and k for the experimental steels

Steels	*n*	*k*
LN	1.632	1.526
MN	1.861	1.794
HN	2.123	1.091

The resultant kinetics models for the experimental steels are given as:

LN:

$$X = 1 - \exp\left(-1.526\left(\frac{\varepsilon - \varepsilon_c}{\varepsilon_p}\right)^{1.632}\right) \quad (20)$$

MN:

$$X = 1 - \exp\left(-1.794\left(\frac{\varepsilon - \varepsilon_c}{\varepsilon_p}\right)^{1..861}\right) \quad (21)$$

HN:

$$X = 1 - \exp\left(-1.091\left(\frac{\varepsilon - \varepsilon_c}{\varepsilon_p}\right)^{2.123}\right) \quad (22)$$

Fig. 5 presents the time dependence of DRX volume fraction for the experimental steels. It can be seen that DRX volume fraction increases with increasing strain and the curves exhibit ‘S’ shapes. With deformation temperature and strain unchanged, DRX volume fraction decreases with increasing strain rate. Although DRX nucleation rate is faster at higher strain rate, the time for the new formed nucleus growth becomes shorter due to faster deformation and it plays the main role, which leads to a smaller DRX volume fraction. However, under high strain rate condition, larger strain can be applied at a shorter time and large strain promotes DRX process. Consequently, DRX volume fraction increases markedly with increasing time, which is shown in Fig. 5b. From the comparison of the curves, it can be seen that the effect of nitrogen on DRX rate is marked. DRX rate decreases with increasing nitrogen contents. In other words, DRX can be restrained due to adding nitrogen, which is consistent with the effect of nitrogen on activation energy for hot deformation.

4. Conclusions

1. Uniaxial hot compression experiments were performed on vanadium and titanium microalloyed steels containing different nitrogen contents to study the effect of nitrogen on the hot deformation behaviors of the experimental steels. The results reveal that the effect of nitrogen on the hot deformation behaviors is marked.
2. Adding nitrogen into vanadium and titanium microalloyed steel can enhance the flow stress, including the peak stress. The critical stress(strain) versus peak stress(strain) relationships of the experimental steels are linear and the slopes of the lines decrease with increasing nitrogen content. Higher nitrogen content contributes to larger critical strain for the onset of DRX of the experimental steels.
3. The activation energies for hot deformation of the experimental steels were determined as 270.493 (LN), 293.228 (MN) and 314.739 kJ mol^{-1} (HN) respectively. And the values were compared with those determined in the previous researches. It can be concluded that adding nitrogen into vanadium and titanium microalloyed steel can enhance the activation energy for hot deformation.
4. The DRX kinetics models for the experimental steels were constructed by regression method and the strain or time de-

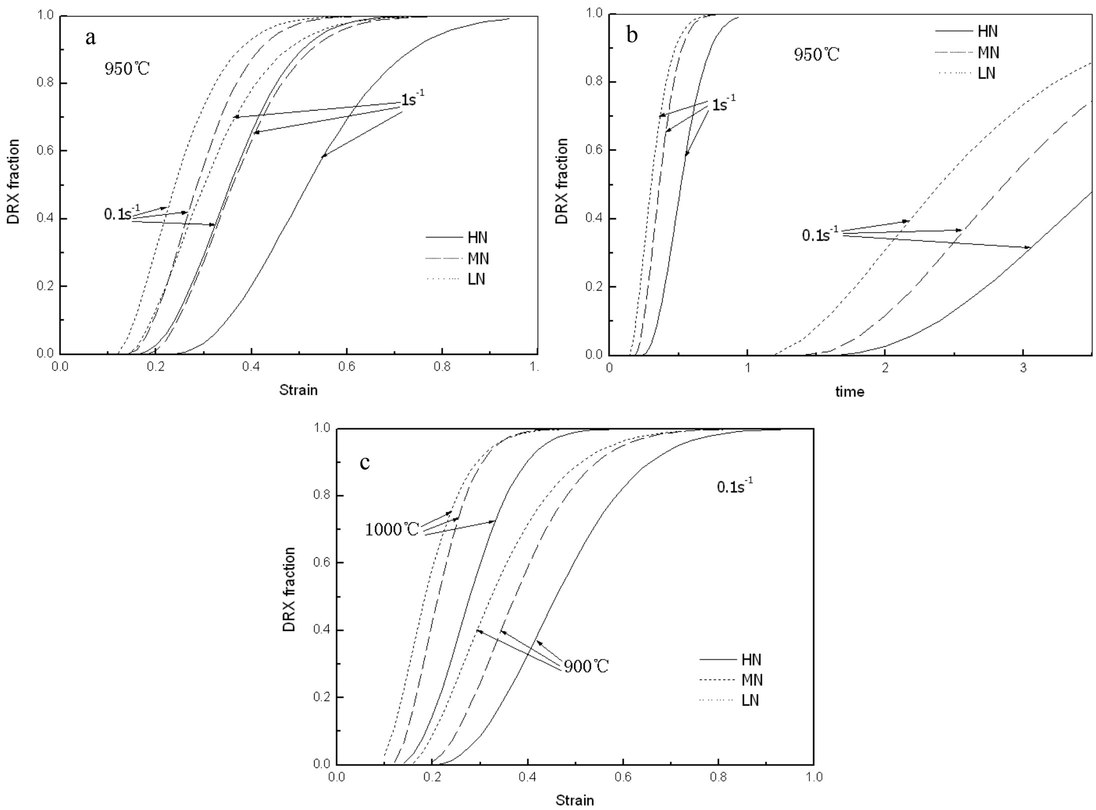


Fig. 5. Relation between DRX volume fractions and(a) strain at 950°C (b) deformation time at 950°C (c) strain at 0.1^{-1}

pendence of DRX volume fraction was obtained. DRX rate decreases with increasing nitrogen content, which confirms the inhibition of DRX by adding nitrogen into vanadium microalloyed steels.

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