Microalloying Effect on the Activation Energy of Hot Deformation

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Although extensive research has been made for evaluating the activation energy Q of hot deformation in microalloyed steels, almost no attempt has been made for directly comparing the influence of each element. In this study, through a series of hot compression tests of four different microalloyed steels, we have systematically explored the influences of Nb, V, Ti, and Mo on the Q and have directly compared the effect of each element in a quantitative manner.

Hot working process has been widely performed to obtain excellent mechanical performance of steels by grain refinement and microstructure homogenization.^[1,2] During the process, due to the low stacking fault energy of austenite phase, dynamic recrystallization (DRX) rather than dynamic recovery (DRC) plays an important role in controlling microstructure and mechanical properties.^[3] Thus, information about representative flow behavior during DRX-included hot deformation is essential for better understanding and developing of hot working process in the steel industry.^[3,4]

One of the most popular ways to describe the effects of temperature and strain rate on hot deformation is to link the exponent-type Zener–Hollomon parameter *Z* to the flow stress σ as^[5–7]

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

where $\dot{\epsilon}$ is the strain rate (in s⁻¹), *T* is the absolute temperature (in K), *R* is the gas constant (8.314 J mol⁻¹ K⁻¹), and *Q* is the activation energy of hot deformation (in J/mol). It is well known that in microalloyed steels, the alloying elements in austenite (such as Nb, V, Ti, and Mo) can induce the retardation of DRX and thus can seriously change the *Q*. For example, Nb and Ti atoms and their precipitates are known to retard the DRX by suppressing the grain boundary migration.^[8,9] In addition, V and Mo can tackle the DRX by inducing coarse prior austenite grains and densely-distributed fine MC-type precipitates, respectively.^[10,11] Despite extensive research to evaluate *Q* in specific microalloyed steels, somewhat interestingly,

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almost no attempt has been made to directly compare the influence of each element in a quantitative manner, which became a motive of this study. In this letter, we report our experimental comparative study on the relative effects of Nb, V, Ti, and Mo on the Q of hot deformation in microalloyed steels.

Chemical compositions of the examined low-carbon micro-alloyed steels are listed in **Table 1**. Cylindrical samples with 12 mm in length and 10 mm in diameter are prepared for hot compression tests. Before compression, austenizing treatment was performed at $1250 \,^{\circ}$ C for 300 s to dissolve the precipitates in the samples. Then, the samples were cooled down to the testing temperature at a cooling rate of $5 \,^{\circ}$ C s⁻¹ and maintained for 30 s before testing for the temperature stabilization.

Computer-controlled hot compression tests were carried out using a thermo-mechanical simulator Gleeble 1500 (Dynamic Systems, Inc., Poestenkill, NY) in the temperature range of 950–1100 °C at an interval of 50 °C. The maximum strain and strain rate were fixed as 0.8 and 0.1 s^{-1} , respectively. All the tests were made under Ar gas environment to avoid the oxidation, and tantalum plates were attached to the samples to minimize the friction effect.

Figure 1 shows flow curves obtained at four different temperatures. While true stress decreases with increasing temperature in all examined steels, typical DRX behavior (exhibiting a peak stress, $\sigma_{\rm p}$, followed by gradual decrease to a steady-state stress, $\sigma_{\rm ss}$) is observed only at 1050 and 1100 °C. Thus, following DRX analysis is made only for the curves at 1050 and 1100 °C.

The function of flow stress, $f(\sigma)$, in Equation (1) can be described as hyperbolic sine law for a wide range of hot deformation stress:^[3,6,7,12–14]

$$Z = f(\sigma) = A \cdot {\sinh(\alpha \cdot \sigma)}^{n}$$
⁽²⁾

where *n* and α are material constants. Note that there are different descriptions of *f*(σ) depending on the stress level;

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Class	С	Mn	Si	Р	S	Al	Nb	V	Ti	Мо	Ν
Nb	0.06	1.5	0.2	0.015	0.003	0.03	0.05	_	_	_	0.006
Nb–V							0.05	0.05	_	—	
Nb–V–Ti							0.05	0.05	0.02	—	
Nb-V-Ti-Mo							0.05	0.05	0.02	0.2	

Table 1. Chemical compositions (wt%) of the investigated steels.



Figure 1. True stress versus true strain curves obtained from hot compression tests at a strain rate of 0.1 s⁻¹; a) Nb steel, b) Nb–V steel, c) Nb-V-Ti steel, and d) Nb-V-Ti-Mo steel.

i.e., a power-law function for relatively low stresses $(\alpha\sigma < 0.8)$ and exponential-law function for relatively high stresses $(\alpha \sigma > 1.2)$.^[12] The stress multiplier α and the exponent n of Equation (2) are typically determined with the stress data obtained under different strain rates.^[3] Since the strain rate was fixed here, $\alpha \approx 0.015$ and $n \approx 5$ is adopted following previous studies.^[3,13,15]

Flow stress σ of Equation (2) can be either peak stress $(\sigma_{\rm p})$ or steady-state stress $(\sigma_{\rm ss})$, both of which are often used for analyzing DRX together with other characteristic stresses such as critical stress (σ_c) and saturation stress (σ_{sat}) .^[16] For determining σ_{p} and σ_{ss} , first the flow curves in Figure 1 were smoothened by polynominal fitting after removing elastic portion. Then, each smoothened curve was re-plotted as work hardening rate θ (=d σ /d ε) versus σ . Figure 2(a) shows representative example of the θ - σ curve

(for Nb steel at 1050 and 1100 °C). In the curve, $\sigma_{\rm p}$ and $\sigma_{\rm ss}$ can be determined as the stress at the first and second point of $\theta = 0$, respectively.^[16] Obtained values of $\sigma_{\rm p}$ and $\sigma_{\rm ss}$ are summarized in Figure 2(b). Note that the critical stress for the initiation of DRX (σ_c) is the stress at the inflection point of θ - σ curve (i.e., the minimum point of the $d^2\theta/d\sigma^2$ vs. σ plot), and σ_c/σ_p values of the examined steels are close to each other in the range of 0.865-0.921, which corresponds to $\varepsilon_c/\varepsilon_p$ (that can be determined by flow curves in Figure 1) of 0.382–0.518. Both σ_c/σ_p and $\varepsilon_c/\varepsilon_p$ values are in agreement with the literature data.^[3,17]

In the present study, $\sigma_{\rm p}$ rather than $\sigma_{\rm ss}$ was adopted as σ of Equation (2) for two reasons; first, the strain corresponding to $\sigma_{\rm p}$ is lower than that to $\sigma_{\rm ss}$, and second, $\sigma_{\rm p}$ is known to be more important for industrial process.[17] As shown in Figure 2(b), σ_p obtained here is in the range of



Figure 2. Evaluation of characteristic stresses: a) representative example showing how to determine the peak stress and steady-state stress from θ to σ plot (for Nb steel); b) summary of the obtained peak stresses and steady-state stresses.

70–97 MPa and thus $\alpha\sigma$ is 1.05–1.46, implying that application of hyperbolic sine function (Equation (2)) is more appropriate than that of power-law or exponential-law function. Combining Equation (1) and (2) and taking natural logarithm lead to

$$\ln \dot{\varepsilon} + \frac{Q}{R} \left(\frac{1}{T} \right) = \ln A + n \ln \{ \sinh(\alpha \sigma_{\rm p}) \}$$
(3)

from which, at a given $\dot{\varepsilon}$, *Q* could be calculated from the slope of the relationship between (1/T) and $\ln{\sinh(\alpha\sigma_p)}$.

Obtained Q values are summarized in Figure 3(a) where literature Q value of C-Mn non-microalloyed steel $(280 \text{ kJ} \text{ mol}^{-1})^{[14]}$ is also provided. It is obvious that microalloying significantly affects Q. Since the added amount of each alloying element is different from each other, it is need to be normalized for directly comparing the effect of each element. Thus, a simple regression was performed, resulting in the following equation of the Q.

$$Q [kJ mol-1] = 280 + 2.29 \times 10^{3} [Nb] + 1.62 \times 10^{3} [V] + 2.73 \times 10^{3} [Ti] + 3.10 \times 10^{2} [Mo]$$
(4)





Figure 3. Influence of microalloying on a) experimentally obtained Q and b) amount-corrected Q.

where [element] indicates the dissolved content in steels (in at%). This amount-corrected Q values are shown in Figure 3(b). It is seen that the Ti shows the largest influence, the effects of Nb and V are the second and third, respectively, and the influence of Mo is the smallest among the four elements; i.e., the microalloying effect on Q is in the order of "Mo < V < Nb < Ti." This also suggests that, since Q is proportional to the decreasing amount of σ_p during increasing T,^[13] σ_p can be reduced more largely by addition of Ti, Nb, and V rather than that of Mo. From all above results, the hot deformation behavior of the examined steels can be summarized as follows;

Nb steel:
$$i \exp\left(\frac{3.49 \times 10^6}{RT}\right)$$

= 4.62 × 10¹¹{sinh(0.015 · σ_p)}⁵
Nb-V steel: $i \exp\left(\frac{4.38 \times 10^6}{RT}\right)$
= 1.48 × 10¹⁵{sinh(0.015 · σ_p)}⁵
Nb-V-Ti steel: $i \exp\left(\frac{5.01 \times 10^6}{RT}\right)$
= 1.94 × 10¹⁷{sinh(0.015 · σ_p)}⁵
Nb-V-Ti-Mo steel: $i \exp\left(\frac{5.37 \times 10^6}{RT}\right)$
= 4.66 × 10¹⁸{sinh(0.015 · σ_p)}⁵.

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