Effects of alloying elements on isothermal transformation behaviour of Ti/Mo microalloyed steel

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Abstract: Isothermal transformation is the key stage to carbide precipitation in microalloyed steel. Detailed phase transformation data are needed in order to control and study the precipitation process of carbides accurately. However, alloying elements will change the transformation process in this microalloyed low carbon steel. Therefore, the effects of titanium and molybdenum elements on isothermal transformation behaviour were studied by a Gleeble 3800 thermal-mechanical simulator. Microscopic test and dilatometry were applied to analyse the isothermal transformation of steels. The results show that the ferrite in steel changes from polygonal ferrite to quasi-polygonal ferrite to acicular ferrite with the decrease of temperature from 700°C to 550°C, and the addition of titanium and molybdenum elements retards the isothermal transformation and result in prolonged transformation time. Besides, alloying elements also tend to inhibit the phase transformation in specific temperatures and lead to more untransformed austenite.

1. Introduction
Microalloying technology is widely used in the development of high strength steel. In recent years, JFE[1] and Zhujiang Steel[2] have done a lot of work on titanium microalloying technology, the steels with high yield strength up to 700 MPa and high formability have been produced. Relevant researches revealed that the addition of microalloying elements, combined with controlled rolling and cooling can significantly refine the ferrite grains and result in fine grain strengthening. Besides, the precipitation of nano-sized carbides during cooling and coiling will result in precipitation hardening. It is a potential mechanism to further improve the strength of microalloyed steel[3]. However, the precipitation of carbides and the growth of ferrite are two synchronous processes during isothermal transformation. The interaction between them has been studied[4], but there are many problems to be solved. Isothermal process is the key stage of carbide precipitation. Detailed phase transformation data are needed in order to control and study the precipitation process of carbides accurately. In this paper, the
effects of titanium and molybdenum microalloying elements and temperature on isothermal transformation of steels are studied, in order to reveal the phase transformation behaviour of microalloyed steel and provide the basis for further research.

2. Experimental Procedure
The steels used in this study were performed using a vacuum induction furnace, titanium and molybdenum elements were added on the basis of low carbon steel to obtain microalloyed steels. After smelting, the steels were cast into ingots and forged into rods for experimental use. The chemical composition of steels is shown in Table 1.

Table 1. Chemical composition of experimental steels, mass-%.

| Chemical composition | Steel | C   | Si | Mn | Ti   | Mo |
|----------------------|-------|-----|----|----|------|----|
| C                   | 0.057 | 0.26 | 1.60 |
| Ti                  | 0.061 | 0.28 | 1.61 | 0.11 |
| Ti-Mo               | 0.064 | 0.30 | 1.65 | 0.12 | 0.22 |

The controlled rolling and controlled cooling process was simulated with Gleeble-3800 thermal-mechanical simulator. The specimens (size, Φ10 × 15mm) were heated to 1200 °C at a rate of 10 °C/s and kept for 5 min. Then, cooled down to 860 °C at a rate of 10 °C /s and compressed with a strain of 0.2 and strain rate of 5/s after being held for 5s. After the hot deformation, specimens were cooled down to isothermal temperature(750°C~550°C) at a rate of 20 °C /s, holding until the γ-α transformation finished. A dilatometer was used to record the changes in diameter.

After the isothermal treatment, the specimens were water quenched to freeze the transformed microstructure, and etched by 4% nitric acid alcohol solution before being analyzed by a LEICA DMLM optical microscope.

3. Results and Discussion

3.1 Transformation time analysis
The transformation time of the steels at 700°C, 650°C, 600°C and 550°C was measured using dilatometer, as shown in Table 2. It can be clearly seen that at all temperatures, the phase transformation time required by three steels is: TiMo steel > Ti steel > C steel.
In general, the isothermal transformation time of steels reflects the growth process of new phase. The strong carbide forming elements titanium and molybdenum in steels will form a “solute drag” effect\[5\], which causes the diffusion rate of carbon atoms to decrease and hinders the nucleation and growth of ferrite. Therefore, the addition of titanium and molybdenum elements leads to the prolongation of phase transformation time.

With the decrease of temperature from 700℃ to 600℃, the diffusion ability of alloy elements in Ti steel and TiMo steel decreases, and the time required for phase transformation increases, and reaching the maximum at 600℃. The growth of new phase in C steel is not much affected by temperature, and maintain a low transformation time even at 600℃ compared with Ti and TiMo steel. When the isothermal temperature is decreased to 550℃, the primary transformation is bainite transformation, which is a half-diffusion one mainly dominated by carbon diffusion and occurs at a very fast speed\[6\]. When the phase transformation proceeds to a certain extent, the solute drag effect will completely inhibit the growth of ferrite, leads to incomplete transformation and residual austenite.

### Table 2. Transformation time of three steels at different isothermal temperatures

| Temperature, ℃ | Steel | Start time, s | End time, s | Transformation time, s |
|----------------|-------|--------------|-------------|-----------------------|
| 550            | C     | 3.3          | 16.3        | 13.0                  |
|                | Ti    | 20.9         | 40.3        | 19.4                  |
|                | TiMo  | 12.2         | 32.0        | 19.8                  |
| 600            | C     | 7.1          | 39.4        | 32.3                  |
|                | Ti    | 22.8         | 2312.6      | 2289.8                |
|                | TiMo  | 705.0        | 8171.0      | 7466.0                |
| 650            | C     | 14.2         | 96.9        | 82.7                  |
|                | Ti    | 106.6        | 478.7       | 372.1                 |
|                | TiMo  | 619.5        | 2266.7      | 1647.2                |
| 700            | C     | 25.8         | 186.0       | 160.2                 |
|                | Ti    | 91.8         | 413.4       | 321.6                 |
|                | TiMo  | 208.0        | 1088.0      | 880.0                 |

### 3.2 Isothermal transformation microstructure analysis

As shown in Figure 2(a1), the 550℃ isothermal treated C steel is composed of fine microstructure. These larger ferrite grains with coarse boundaries are so-called quasi-polygonal ferrite\[7\]. A series of rings formed by these grains implicitly show the original austenite grain boundaries. Besides, an aggregation of inclusions is observed in quasi-polygonal ferrite grain boundaries, these defects promote the non-uniform nucleation of ferrite. It can be considered that quasi-polygonal ferrites grow mainly at the austenite grain boundaries during isothermal process\[8\]. High magnification microscopic observation reveals that within the prior austenite grains, the finer microstructure is consist of granular bainitic ferrite, with the features of elongated, cracked islands of retained austenite scattered over ferrite matrix, and the rest is martensite.

The 550℃ isothermal treated Ti steel is mainly consist of granular bainitic ferrite, Figure 2(b1). But as to 550℃ isothermal treated TiMo steel, Figure 2(c1), with finer and undistinguishable parallel laths structure, elongated acicular ferrite is different from the first two specimens, it is a mixture of lath ferrite and retained austenite.

When the isothermal treatment temperature increases to 600℃, the constitution of microstructure of C steel remains nearly unchanged, as shown in Figure 2(a2). With the reduction of undercooling degree, the nucleation ratio decreases, so the grains at 600℃ are slightly coarsened. Ti steel sample treated at 600℃ is largely composed of quasi-polygonal ferrite, the rest is polygonal ferrite, a small amount of residual austenite and martensite(Figure 2(b2)). In comparison, the microstructure of TiMo steel isothermal treated at 600℃ is similar to Ti steel’s, but the ferrite grains are larger than that of Ti steel.
Figure 2. Optical microstructures of specimens after isothermal treatment (water quenched):
   a - C steel; b - Ti steel; c - TiMo steel;
   1-550°C; 2-600°C; 3-650°C; 4-700°C
steel (Figure 2(c2)). In addition, more martensite is observed in the TiMo steel sample.

When the isothermal temperature rises to 650℃, the quasi-polygonal ferrite in the microstructure of Ti steel and TiMo steel is almost disappeared, while a considerable proportion of quasi-polygonal ferrite remains in the C steel sample. Quasi-polygonal ferrite is formed through short range diffusion, and the irregular growth of ferrite lead to rough grain boundaries[8]. So alloying elements titanium and molybdenum in steel changed the formation mechanism of ferrite and result in flat grain boundaries.

These steels treated at 700 ℃ is composed of polygonal ferrite and residual austenite, and a part of residual austenite transforms into martensite during water quenching after isothermal treatment. The amount of residual austenite in three steels treated at 700℃ increases significantly compared with that of steels treated at 650℃.

3.3 Dilatometric measurement analysis
During isothermal treatment process, the austenite transforms into ferrite and carbides, and according to the metallographic observation above, the austenite does not undergo complete transformation. Due to the difference in crystal structures of austenite and ferrite, austenite is more dense than ferrite, so the phase transformation will lead to the expansion of sample. The change of diameter $\Delta d$ from start to end of the phase transformation of the sample was recorded by a dilatometer. The diameter of the sample at the start of the phase transformation is $d_0$, and $\Delta d/d_0$ reflects the relative expansion ratio caused by the phase transformation, which is related to the proportion of transformed austenite, as shown in Figure 3.

The percentage of isothermal transformed ferrite phase $f_\alpha$ was calculated by measuring the area of untransformed austenite(including martensite and retained austenite) using metallographic photographs of steels treated at 700℃, as evidenced in Figure 4. At 700℃, the relationship between $\Delta d/d_0$ and $f_\alpha$ is completely proportional, the more ferrite phase, the larger expansion ratio. There is a large amount of retained austenite and martensite in the TiMo steel after isothermal treatment, and the amount of retained austenite is more than that of martensite, resulting in the percentage of ferrite phase of only 53.9 %. Alloying elements titanium and molybdenum in steel change the decomposition kinetics of austenite, and the austenite becomes more stable and not apt to decompose into ferrite during isothermal treatment, even does not transform into martensite during quenching, lead to incomplete transformation of austenite. Ti steel and C steel are similar in percentage of ferrite phase, which are 74.8 % and 73.8%, respectively, indicating that titanium element in Ti steel retards the
decomposition of austenite at 700℃, but not completely inhibit the decomposition.

For 600℃ isothermal treated samples, due to the solute drag effect of titanium and molybdenum elements, and the reduction of element diffusion ability, the growth of ferrite is inhibited. Austenite decomposes slowly for thousands of seconds at 600℃ until the phase equilibrium state. According to the microstructure in Figure 2(b2)/(c2), there is a considerable proportion of untransformed austenite in Ti steel and TiMo steel after phase transformation, make $\Delta d/d_0$ lower than that of C steel.

The relative expansion ratio of three steels isothermally treated at 550℃ are similar. At this temperature, the three steels all undergo rapid semi-diffusional bainite transformation. Therefore, the influence of titanium and molybdenum elements on the austenite decomposition kinetics is not evident at this temperature. Similarly, at 650℃, titanium and molybdenum elements delay the decomposition of austenite, but the inhibition effect on austenite transformation is not evident.

Figure 4. Percentage of isothermal transformed ferrite and relative expansion ratios of steels at 700℃

4. Conclusion

According to the above results, ferrite in isothermal transformed steels changes from polygonal ferrite to quasi-polygonal ferrite to acicular ferrite with the decrease of temperature from 700℃ to 550℃. Alloying elements titanium and molybdenum will lead to more untransformed austenite, especially at 700℃ and 600℃.

The transformation time of C steel decreases with the decrease of temperature from 700℃ to 550℃. But the transformation time of Ti steel and TiMo steel increases with the decrease of temperature from 700℃ to 600℃. At 550℃, the transformation time of three steels are similar. The prolongation of transformation time of microalloyed steels can attribute to the solute drag effect of alloying elements.

Basic analysis of microstructure morphology at different isothermal temperatures and accurate measurement of transformation time make precise control of transformation possible in these steels. Further investigation on precipitation process of carbides during isothermal transformation will be done based on these experimental results.

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