Kinetics and Morphology of Isothermal Transformations at Intermediate Temperature in 15CrMnMoV Steel

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Upper bainite (BU), lower bainite (BL) and granular structure (GS) are three distinguishable microstructures formed through isothermal treatment at temperature range from 350 to 520 °C in 15CrMnMoV steel. Although long time isothermal holding leads to the mixed microstructures, single microstructure can be obtained in the initial stage of isothermal transformation when the total transformation fraction is less than 20%. The Arrhenius equations were used to determine the overall activation energies of the transformations in the steel with accuracy of ±8 kJ/mole. To ensure the determined overall activation energy is the true value for single microstructure rather than for a mixture of two or more microstructures, the present determination of the kinetic data of isothermal transformations was carried out in terms of the isothermally transformed fractions of 1 and 5% at various temperatures. Experimental results show that the overall activation energy for single microstructure is significantly different from that for mixed microstructure, which was measured in terms of 50% transformation. Upon the determined kinetic data and the morphology of the microstructures, together with consideration of the reported atom diffusion activation energies, following conclusions are achieved. In steels, the BL transformation is controlled by carbon diffusion in the austenite; while, the formation of BU is only partially governed by carbon diffusion in austenite. As a different microstructure from other two, the GS transformation is controlled by carbon diffusion and has the common features of both equiaxed ferrite and massive ferrite transformations. These results, can be used to further understand the formation mechanism of BU, BL and GS in the 15CrMnMoV steel.

Keywords: bainite, 15CrMnMoV steel, arrhenius equations, activation energy

1. Introduction

Since Bain ¹ first reported bainite microstructure in the 1930s, its properties, morphology and transformation mechanism have been considerably studied in the past 70 years. One of the milestones in the history of bainite study was the establishment of the shear model of bainitic transformation in the 1950’s by Ko and co-workers.² As typical kinetic data of phase transformations in solid, the overall activation energy (OAE) has been widely used to understand the characteristics of bainitic transformations. However, in most cases, the reported experimental results are inconsistent with the calculated overall activation energy. The major reason for this discrepancy is that the previously published OAE data was experimentally determined for a mixture of two or more different microstructures due to the difficulty to separate them, rather than for single microstructure. Considerable experimental work³,⁴ has shown that isothermal treatment of low carbon low alloy steels at intermediate temperature ranging from 350 to 550 °C produces upper bainite (BU), lower bainite (BL), granular structure (GS) or their mixture. These three microstructures are not only morphologically different but also can be characterized by their distinguishable C-curves on the TTT diagram³,⁴ even though the GS, in most publications, is regarded as granular bainite or BU. Normally, in initial stage of the transformation at specified temperatures, single microstructure can be obtained when the totally transformed fraction is less than 20%, whereas long time isothermal holding leads to the formation of mixed microstructure. Hence, the aims of the present work are: (1) to experimentally determine the OAE for single BU, BL and GS in 15CrMnMoV steel in terms of the total isothermally transformed fractions of 1 and 5% (only single microstructure is obtained) using the Arrhenius equations; and (2) to compare these OAE values determined with those measured for 50% transformation, at which mixed microstructure is produced. The OAE data of phase transformations, together with the morphologies of microstructures and the atomic diffusion activation energies in steels, can be used to further understand the formation mechanism of BU, BL and GS in low carbon low alloy steels.

1.1 Arrhenius equations

The Arrhenius equation has two versions. One describes the transformation OAE as a function of time and temperature; another relates the OAE to transformation rate and temperature. The two versions of Arrhenius equation are as follows:

Version 1–OAE is a function of time and temperature:

\[ Q_{X1} = R \left[ \frac{\partial \ln t}{\partial \left( \frac{1}{T} \right)} \right]_{X_i} \]  \hspace{1cm} (1)

Version 2–relationship between OAE and transformation rate and temperature:

\[ Q_{X1} = R \left[ \frac{\partial \ln \left( \frac{\partial X}{\partial t} \right)}{\partial \left( \frac{1}{T} \right)} \right]_{X_i} \]  \hspace{1cm} (2)

Where \( Q_{X1} \) is the OAE, \( R \) the gas constant, \( t \) the isothermal transformation time, \( T \) the transformation temperature and \( X \) the transformed fraction. For a specified fraction of transformation, the slope of the straight line that graphically

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describes the relationships between \( \ln t \) and \( 1/T \) or between \( \ln(\partial X/\partial t) \) and \( 1/T \) is proportional to \( Q_{X1} \). The OAE at the transformed fraction \( X \) can be calculated by multiplying the gas constant. Although Hillert suggested that \( Q_{X1} \) from eq. (2) is more accurate, eq. (1) is usually used due to its simplicity.

### 1.2 Overall activation energy of bainitic transformation in steels

Previous work has shown that the OAE of bainitic transformation is associated with the carbon content of steels. In the late of 1950s, using eq. (1) Vasudevan et al. determined the OAE, \( Q_{Bu} \), and \( Q_{Bl} \) (as listed in Table 1) for \( B_u \) and \( B_l \), respectively, based on the transformed fraction of 50% in a plain carbon steel containing 0.97 mass%\( \mathrm{C} \). The result indicates that \( Q_{Bu} \) is greater than \( Q_{Bl} \). Because \( Q_{Bu} \) is very close to the value of diffusion activation energy of carbon in \( \gamma \)-Fe (130\( \text{kJ/mol} \)), Vasudevan considered that the transformation of upper bainite is controlled by carbon diffusion in \( \gamma \)-Fe. Similarly, as the determined \( Q_{Bu} \) value is close to the diffusion activation energy of carbon in \( \alpha \)-Fe (70\( \text{kJ/mol} \)), it was considered that the lower bainite transformation relies on carbon diffusion in \( \alpha \)-Fe. However, this hypothesis could not explain the subsequent experimental results that show much greater OAE values than the carbon diffusion activation energy in both \( \gamma \)-Fe and \( \alpha \)-Fe. Radcliffe and co-workers determined the OAE for both upper and lower bainite in steels with different carbon contents using the same method. It was found that the OAE of transformations increases with the decrease of carbon content in the steels, and that \( Q_{Bu} \) is always bigger than \( Q_{Bl} \). The same conclusion was reported by Yu et al. who measured the OAE based on the transformed fraction of 25–30%. All the reported OAE data for various steels is listed in Table 1, in which 613–623 K is used as the transition temperature from upper bainite to lower bainite.

In 1966, Barford determined the OAE for \( B_u \) and \( B_l \) in terms of transformed fraction of 10–75% (or 60%) in high carbon Mn-containing steels using both eqs. (1) and (2). Although the results are consistent with those previously reported for the steels with carbon content less than 1.1 mass%, conflict results were obtained in the steel containing 1.42 mass\%\( \mathrm{C} \). The OAE obtained from both equations shows \( Q_{Bu} > Q_{Bl} \) at the transformed fraction of 10%, but the opposite \( Q_{Bl} < Q_{Bu} \) at transformed fraction of 50%, as bolded in Table 1. The latter is abnormal. Although Barford did not explain the abnormal results, the reported data demonstrates that the experimentally determined OAE not only related to the carbon content of steels, but also depends on the transformed fraction, on which the determination is based. Hillert et al. reviewed the TTT diagrams of more than 20 steels, including plain carbon steels, alloy steels and Mn-containing steels. It was found that the \( Q_{Bl} \) value is constantly 80\( \text{kJ/mol} \) for the lower bainite transformation in all steels when the determination was in terms of transformed fraction of 1%. This is consistent with Barford’s results determined in terms of the transformed fraction of 10% in the 1.42 mass\%\( \mathrm{C} \) steel (see Table 1). In addition, the OAE of bainite transformation in continuous cooling conditions was also investigated. The published data is listed in Table 1. It can be seen that the \( Q_{Bl} \) is always bigger than the \( Q_{Bu} \) for all steels.

However, all previous researchers did not pay enough attention to the effect of the fraction of transformation on the

<table>
<thead>
<tr>
<th>Steels</th>
<th>Marked change temperature (K) from ( B_u ) to ( B_l )</th>
<th>( Q_{Bu} )</th>
<th>( Q_{Bl} )</th>
<th>Equation, Fraction used</th>
<th>Comments</th>
<th>Ref.</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.77 mass%( \mathrm{C} ) steel</td>
<td>573–603</td>
<td>77</td>
<td>127</td>
<td>eq. (1), ( X = 8% )</td>
<td></td>
<td>6</td>
</tr>
<tr>
<td>1.13 mass%( \mathrm{C} ) steel</td>
<td>573–603</td>
<td>35</td>
<td>52</td>
<td>eq. (1), ( X = 10–75% )</td>
<td></td>
<td>Steel No. 1, 2, 3</td>
</tr>
<tr>
<td>Steel No</td>
<td>1</td>
<td>2</td>
<td>3</td>
<td>4</td>
<td>50–63</td>
<td>80–121</td>
</tr>
<tr>
<td>mass%( \mathrm{C} )</td>
<td>0.81</td>
<td>1.09</td>
<td>1.10</td>
<td>1.42</td>
<td>113</td>
<td>87</td>
</tr>
<tr>
<td>mass%( \mathrm{Mn} )</td>
<td>1.00</td>
<td>1.00</td>
<td>1.49</td>
<td>2.37</td>
<td>60</td>
<td>105</td>
</tr>
<tr>
<td>26 steels</td>
<td>—</td>
<td>80</td>
<td>50</td>
<td>( X = 1% )</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Low carbon Cr-Mo-V steel</td>
<td>140</td>
<td>40</td>
<td>Continuous cooling</td>
<td>10</td>
<td></td>
<td></td>
</tr>
<tr>
<td>HSLA-100 steel</td>
<td>175 ± 25</td>
<td>Continuous cooling</td>
<td>12</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>0.4 mass%( \mathrm{C} ), 1.73 mass%( \mathrm{Cr} ), 1.03 mass%( \mathrm{Mn} ), 1.21 mass%( \mathrm{Si} ), 0.50 mass%( \mathrm{Mo} ), 0.1 mass%( \mathrm{V} )</td>
<td>126</td>
<td>74</td>
<td>eq. (1), ( X = 5% )</td>
<td>Single structure</td>
<td>3</td>
<td></td>
</tr>
</tbody>
</table>

**Table 1** The overall activation energy of bainite transformation in steels.
morphology of final microstructures as well as on the experimentally determined OAE data. In the early 1990s Kang et al. noticed this inherent flaw and suggested that more accurate QBU and QB can only be determined from eq. (2) in terms of the transformed fraction less than 5%, because low transformed fraction can guarantee the formation of single microstructure (BU or BL). The OAE data previously reported by Kang and co-workers is also listed in Table 1.

2. Experimental Procedure

The chemical composition of 15CrMnMoV steel used is listed in Table 2. The as received bars (20 mm in diameter) were homogenization annealed at 1470 K for 72 hours, then 3 mm diameter and 30 mm long thermo-magnetic specimens were machined. After austenization in NaCl salt bath at 1243 K for 10 hours, these specimens were then isothermally treated at different temperatures for various times in either liquid lead or liquid lead-tin baths, followed by water quenching. A thermo-magnetic apparatus fitted with an X-Y recorder, which has a sensitivity of 0.2%, was used to determine the transformed fraction according to the following equation. The transformed fraction, X:

\[
X = \frac{\alpha - \alpha_0}{\alpha_1 - \alpha_0}
\]

where \(\alpha\), \(\alpha_1\) and \(\alpha_0\) are the magnetic property related parameters to be read directly from the X-Y recorder. \(\alpha_1\) is taken at the annealed state, \(\alpha_0\) is the reading at austenization temperature and \(\alpha\) is the reading at a specified isothermal holding temperature and time before water quenching. Therefore, X only associates with the isothermal transformation fractions without effect of the martensite formed in the subsequent water quenching.

TEM specimens were prepared in the process as follows: 0.8–1.0 mm thick discs were cut off from the thermo-magnetic specimens using a spark cutter. After mechanically grinding to 0.08–0.1 mm thick, the discs were then jet polished in a GIKOCE-10 machine with voltage of 35 V at 15°C. The electrolyte used is 7–10% perchloric acid and 90–93% glacial acetic acid. All specimens were examined in H-800 TEM at an operating voltage of 200 keV.

3. Results and Discussions

3.1 TTT diagram and microstructure

The TTT diagram of 15CrMnMoV steel constructed based on the data from the thermo-magnetic apparatus is shown in Fig. 1. There are three independent 1% transformation C curves, corresponding to the lower bainite (BL), the upper bainite (BU) and the granular structure (GS), respectively. As shown by the dashed lines, longer isothermal holding leads to the overlap of these C curves. This implies that at beginning of the transformation at a specified temperature single microstructure can be obtained and as the increase in holding time mixtures of two or three types of microstructure form.

The morphology of these three microstructures is distinguishable. GS consists of massive ferrite matrix and randomly distributed martensite-austenite (M-A) islands in the matrix, as schematically illustrated in Fig. 2(a). The M-A islands are normally in irregular shape and are made of either martensite-austenite mixture or sole martensite or single retained austenite (Ar). B includes meta-upper bainite.
(BMU) and granular bainite (Bg). It consists of lath bainitic ferrite and either AR thin films (BMU) or M-A islands (Bg) in between the ferrite laths, as schematically illustrated in Figs. 2(b) and 2(c). BL in 15CrMnMoV steel is comprised of plate ferrite and systematically distributed AR thin films within the ferrite plates. The optical morphology of BL is needle-like and in TEM AR thin films can be observed as schematically shown in Fig. 2(d). The AR and M-A islands in both BU and BL will decompose into carbides and ferrite in the consequent long time isothermal holding or tempering, thus typical BU and BL form.

Isothermal holding of the 15CrMnMoV steel after austenitization at 643 K for 3 seconds produces needle-like BL as shown in Fig. 3(a) is the optical micrograph. Figure 3(b) is the TEM micrograph illustrates the AR thin films systematically distributed within the bainitic ferrite plate. Longer time holding (10 seconds) leads to the formation of BU beside the existing BL plates as shown in Fig. 3(c) (optical micrograph) and (d) (TEM micrograph). Similarly, single BU can only be obtained at the initial stage of the transformation within the BU transformation zone at higher temperatures. Isothermal holding at 703 K for 10 seconds produces Bg as shown in Fig. 4(a) and BMU exhibiting in Fig. 4(b). Both Bg and BMU can be regarded as a meta-stable or special type of BU and both share the same C-curve in TTT diagram. Similarly, if the isothermal holding time is long enough, BL forms at the same temperature as indicated in Fig. 1. Single GS forms as a
consequence of isothermal holding at 773 K for 200 seconds as shown in Fig. 5(a). It can be distinguished from Bg based on the shape of the ferrite matrix and the distribution of M-A islands on it. In GS, M-A islands are randomly distributed in the massive ferrite matrix. In contrast, the M-A islands in Bg distributes in between the ferrite laths.\textsuperscript{3,13} Generally, in low carbon steels the GS transformation obeys an independent C-curve on the TTT diagram. Like the transformation at other temperatures, if the isothermal holding time is long enough, Bg forms after GS as shown in Fig. 5(b), where arrow A indicates Bg, and arrow B points to the GS.

As the stated above, it can be concluded that, (1) single microstructure such as GS, Bg, B_U and B_L, can only be obtained at the very early stage of transformation; (2) long time isothermal holding at a specified temperature leads to the formation of mixed microstructures; (3) in terms of the starting C-curve on the TTT diagram, the transition from B_U to B_L occurs at a critical temperature, 673 K for 15CrMnMoV steel. However, B_L can also form after B_U at temperatures above 673 K, B_U can form as a consequence of B_L below 673 K if the isothermal holding time is long enough in 15CrMnMoV steel.

In addition, a similar phenomenon is also observed in 30CrMnSiN2 steel.\textsuperscript{3,4,14} There is no GS and Bg in medium carbon steels. Carbide-free (meta) bainite consisting of bainitic ferrite (BF) and retained austenite (AR) is the common microstructure in Si-containing steels.\textsuperscript{3,4,14}

3.2 The overall activation energy of bainitic transformation

The effect of temperature on transformation rate is well described by the linear relationship between $\ln(\partial X/\partial t)$ and $1/T$ for a given transformation fraction ($X$). The overall activation energy can be calculated based on the slope of the straight line using eq. (2). Figure 6 shows such a relationship in terms of $X = 1\%$ and $5\%$ for GS, B_U and B_L respectively for the 15CrMnMoV steel. For comparison the $\ln(\partial X/\partial t)$ vs. $1/T$ relationship at $X = 50\%$ is also included in Fig. 6 because mixed microstructure of B_U and B_L formed at this transformed fraction. Although the lines associated with $X = 1\%$ and $X = 5\%$ for a specified microstructure are parallel, the slopes of lines for different microstructures, including the mixed one vary. This indicates that the overall activation energies of GS, B_U and B_L are different. The parallelism of the $1\%$ and $5\%$ lines implies, at least when the total transformed fraction is below $5\%$, single microstructure is obtained. From the slope of the lines and using eq. (2) the OAEs of GS, B_U and B_L are calculated to be $Q_{GS} = 170 \text{ kJ/mol}$, $Q_{B_u} = 132 \text{ kJ/mol}$ and $Q_{B_l} = 52 \text{ kJ/mol}$, with accuracy of $\pm 8\%$. Because these OAE values were measured based on the onset of the transformation and on single microstructure, they are independent from the transformed fraction. In addition, although the nucleation sites also affect the overall activation energy, such as austenite grains and dislocations, the present determinations were based on the same austenization temperature and used the same cooling agent (NaCl salt.

![Fig. 5](image1.png)

**Fig. 5** Optical microstructure in 15CrMnMoV steel austempered at 773 K after austenitization: (a) Granular structure (GS) after isothermal holding for 150 s, (b) GS+Bg mixture after isothermal holding for 3000 s.

![Fig. 6](image2.png)

**Fig. 6** Relationship between $\ln(\partial x/\partial t)$ and $1/T$ at transformation fraction $X = 1, 5$ and $50\%$ within medial temperature transformation region in 15CrMnMoV steel.
bath). Therefore, it is reasonable to ignore these influences. Because the determined value of \( Q_{\text{Bu}} \) is very close to the reported diffusion activation energy of carbon in \( \gamma \)-Fe (\( Q_{\gamma} = 131 \text{kJ/mol} \)), \(^{8,15}\) it is reasonable to consider that the transformation of upper bainite is controlled by the diffusion of carbon in \( \gamma \)-Fe. The present \( Q_{\text{Bu}} \) value agrees well with Hillert’s data.\(^{10}\) Although it is close to the value of the diffusion activation energy of carbon in \( \alpha \)-Fe (\( Q_{\alpha} = 70 \text{kJ/mol} \)),\(^{30}\) in the initial stages of transformation, lower bainite cannot form through decomposition of martensite, which might form from austenite, within the lower bainite forming temperature range.\(^{25}\) This is because carbon diffusion in \( \alpha \)-Fe is more difficult at such low temperatures. In addition, since the determined \( Q_{\text{Bu}} \) is much lower than the \( Q_{\gamma} \), hence, the rate of the lower bainite transformation may be partially controlled by carbon diffusion in \( \gamma \)-Fe. The determined OAE of GS, \( Q_{\text{GS}} \), is in between the OAE value of equiaxed \( \alpha \) transformation, \( Q_{\text{Bu}} = 193–198 \text{kJ/mol} \),\(^{17}\) and the OAE value of the massive \( \alpha \) transformation, \( Q_{\alpha} = 144 \text{kJ/mol} \).\(^{18,19}\) Hence, GS transformation has the common characteristics of both equiaxed and massive transformations. It can be regarded as diffusion-less (for Fe atoms) ferrite/austenite interface immigration process controlled by carbon diffusion. The GS is a product of independent process that differs from both upper and lower bainite transformations.

The determined overall activation energy at \( X = 50\% \) is different from all the three OAE values for \( Q_{\text{Bu}}, \ Q_{\gamma}, \) and GS determined at \( X = 1\% \) and \( 5\% \). In Fig. 6, there are two nonparallel straight lines for \( X = 50\% \). One was determined within lower bainite temperature range and another within upper bainite temperature range (\( T < 673 \text{K} \)). From the slope of the line obtained at lower temperatures, the OAE for the mixed microstructure was calculated to be \( Q_{\text{Bu}} = 127 \text{kJ/mol} \). Because the transformation occurred within the lower bainite zone, the \( Q_{\text{Bu}} \) value should be regarded as the OAE of lower bainite. However, \( Q_{\text{Bu}} \) is much too higher than the \( Q_{\gamma} \). Similarly, the calculated overall activation energy, \( Q_{\text{Bu}} = 118 \text{kJ/mol} \), in terms of the slope of the \( X = 50\% \) line in Fig. 6, which was determined within the upper bainite region (\( T > 673 \text{K} \)). Although it is determined within upper bainite zone, the value is lower than the \( Q_{\gamma} \) determined for single \( \text{Bu} \). More importantly, one can notice that \( Q_{\text{Bu}} \) is greater than \( Q_{\text{Bu}} \), which is abnormal, even though a similar result was reported by Barford.\(^{9}\) This inconsistence is attributed to the mixing of \( \text{Bu} \) and \( \text{Bl} \) at \( X = 50\% \). Although single upper (or lower) bainite can be obtained at initial stage of the isothermal transformation within upper (or lower) bainite temperature range, as consequence, lower (or upper) bainite forms at 50% of the total transformed fraction. Thus, both \( Q_{\text{Bu}} \) and \( Q_{\text{Bl}} \) are actually the OAEs of the formation of mixture of \( \text{Bu} \) and \( \text{Bl} \). Neither of them is true OAE for \( \text{Bu} \) or \( \text{Bl} \). These two values are considered as false OAE and meaningless. They cannot be used to study of kinetics of the transformations. Therefore, accurate determination of OAE should be in terms of single microstructure, which can be obtained at the early stage of the transformation.

The presently determined values \( Q_{\text{Bu}} = 52 \text{kJ/mol} \) and \( Q_{\text{Bl}} = 132 \text{kJ/mol} \) with single microstructure in 15CrMnMoV steel are not only far below the self-diffusion activation energy of Fe in \( \alpha \)-Fe (\( Q_{\alpha} = 250 \text{kJ/mol} \)),\(^{4}\) and in \( \gamma \)-Fe (\( Q_{\gamma} = 285 \text{kJ/mol} \)),\(^{15}\) but also smaller than the OAE of equiaxed ferrite formation (\( Q_{\text{Bu}} = 193–198 \text{kJ/mol} \))\(^{17}\) and massive ferrite formation (\( Q_{\text{M}} = 144 \text{kJ/mol} \)).\(^{18,19}\) These experimental data suggests that bainitic ferrite, including upper and lower bainite, cannot form from austenite through either conventional diffusion controlled or massive diffusion-less transformation. In order to retain the lath morphology, both the \( \text{Bu} \) and \( \text{Bl} \) transformations can only occur by diffusion-less martensite-like shear that is partially controlled by carbon diffusion in austenite. However, as the \( Q_{\text{GS}} \) is in between the \( Q_{\gamma} \) and \( Q_{\text{M}} \), the granular structure is regarded as a product of massive type diffusion-less transformation.

### 4. Conclusions

1. Upper bainite (\( \text{Bu} \)), lower bainite (\( \text{Bl} \)) and granular structure (GS) in low carbon alloy steels have distinguishable independent C-curves in TTT diagrams, although the C-Curves may overlap. Single microstructure can only be obtained at initial stage of isothermal transformation with low total transformed fraction. Long time isothermal holding leads to the increase of total transformed fraction accompanied with the formation of mixed microstructures.

2. The overall activation energy for a phase transformation can only be accurately determined in terms of single microstructure, which normally appears when the total transformed fraction is less than 20%.

3. In steels, from the similarity of \( Q_{\text{Bu}} \) to \( Q_{\gamma} \), the \( \text{Bu} \) transformation is controlled by carbon diffusion in the austenite; however, the formation of \( \text{Bl} \) is only partially governed by carbon diffusion in austenite as \( Q_{\text{Bl}} \) is much lower than \( Q_{\gamma} \). As an independent microstructure, the GS transformation is controlled by carbon diffusion and has the common features of both equiaxed ferrite and massive ferrite transformations, because the \( Q_{\text{GS}} \) is in between \( Q_{\gamma} \) and \( Q_{\text{M}} \).

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### REFERENCES

8. D. G. Yu and D. K. Shi: Proc. the First Conf. on Heat Treatment,