Metastable Austenite Transformation Kinetics of Medium-Carbon Silicon-Rich Steel during Partitioning in a Q & P Process

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Abstract: In the present study, quenching and partitioning (Q & P) treatment of a medium-carbon silicon-rich steel was processed by a dilatometer. The volume fraction of the retained austenite at different partitioning times was determined by X-ray diffraction (XRD). The metastable austenite transformation process after different partitioning times was studied by a combination of dilatometry, XRD, and transmission electron microscopy (TEM). Analysis of the transformation kinetics of metastable austenite during partitioning by means of dilatometry and the Johnson–Mehl–Avrami–Kolmogorov (JMAK) equation was carried out. The results show that the volume fraction of retained austenite increases first and then decreases with the increase of the partitioning time. The transformation active energy $Q = 141 \text{ kJ mol}^{-1}$ and the Avrami exponent $n = 1$. The transformation product of metastable austenite is needle-like bainite, which grows perpendicularly towards the boundary of the austenite. Finally, the blocky austenite was divided into lath-shaped forms.

Keywords: medium-carbon silicon-rich steel; quenching and partitioning process; metastable austenite; transformation kinetics

1. Introduction

The quenching and partitioning (Q & P) process can significantly improve the toughness and ductility of steels and has received extensive attention since it was proposed by Speer in 2003 [1]. It is considered to be the main means of developing the third generation of ultra-high-strength steel. The core of the Q & P process is the preservation of a multiphase microstructure consisting of martensite and an amount of retained austenite in the final microstructure of steel. Martensite presents high strength, and the retained austenite provides excellent plasticity and toughness, resulting in excellent comprehensive mechanical properties in Q & P steel.

C is one of the most important strengthening elements in steel. It can be dissolved in austenite, expand the γ-phase region, stabilize austenite, delay the phase transformation of ferrite and bainite, and decrease the $M_s$ temperature of steel. Low C content leads to insufficient strength in steel and insufficient stability of residual austenite; however, excessive C reduces the plasticity and toughness of steel [2]. Si is a non-carbide-forming element and has a low solubility in carbides. In the process of Q & P treatment, it can suppress the precipitation of carbides and promote the diffusion of carbon between martensite and austenite, thereby improving the stability of austenite and saving more austenite to room temperature [3,4]. In Q & P steel, the fraction of restrained austenite, the partitioning process of carbon, and the morphology of the final phase have a significant effect on its mechanical properties. It is
considered that the redistribution of carbon from martensite to austenite occurs only in the partitioning stage, and the amount of retained austenite and martensite does not change in the constrained carbon equilibrium (CCE) model proposed by Speer [1]. According to this model, volume fraction and carbon concentration in each phase were obtained. However, in subsequent research, some scholars found that metastable austenite undergoes a certain transformation during the partitioning process. Kim et al. [5] proposed that metastable austenite will decompose during the isothermal process and that the nascent phase has a bending boundary with retained austenite. Boheman [6,7], Silva [8], Sajjadi [9] and Samanta [10], and others believe that the lower bainite is formed during the isothermal process. Somani et al. [11] suggested that an isothermal martensitic transformation will occur at the beginning of the isothermal phase, followed by a bainite transformation. In a study by Samanta et al. [12], the experimentally obtained volume fraction of retained austenite was largely deviated from the theoretical calculation of the CCE model. Li et al. [13] studied the isothermal decomposition of austenite during the partitioning of 0.25C–3Mn–2Al (wt.%) steel. Chen et al. [14] studied the austenite stability and isothermal bainite transformation of low-carbon silicon-containing steels during Q & P. HajyAkbary et al. [15] studied the relationships between carbon distribution, carbide precipitation, and bainite transformation during Q & P treatment of 0.3C–1.6Si–3.5Mn (wt.%) steel and proposed that unstable austenite will decompose to bainite during the isothermal partitioning process.

Although there have been research articles on the metastable austenite transformation during the partitioning process, little research has been done on metastable austenite transformation during Q & P treatment of medium-carbon silicon-rich steel.

Therefore, in this study, a commercial medium-carbon silicon-rich high-strength 60Si2CrVA steel was selected as the research object to study the transition kinetics of metastable austenite systematically. This will provide a reference for improving the performance of similar medium-carbon silicon-containing commercial steels, so as to allow for precise control of the microstructure and final mechanical properties.

In the present paper, the dilatometry curves and XRD patterns of 60Si2CrVA steel under different partitioning times and isothermal temperature conditions were analyzed. Transformation kinetics of metastable austenite during partitioning were discussed, based on experimental observations and Johnson–Mehl–Avrami–Kolmogorov (JMAK) modeling.

2. Experimental Materials and Methods

The steel used in this work was a round bar that annealed after forging with a diameter of 32 mm. The chemical composition of the 60Si2CrVA is shown in Table 1.

| Table 1. Chemical composition of 60Si2CrVA steel (wt.%). |
|-------|--------|--------|------|------|--------|------|
| C     | Si     | Cr     | V    | Mn   | P      | S    |
| 0.535 | 1.725  | 1.035  | 0.127| 0.631| 0.017  | 0.0025|

Specimens of dimensions Φ4 × 10 mm were processed from the annealed steel and tested with a DIL805 A/D dilatometer. These heat treatment schedules are shown in Figure 1. The material was completely austenitized by a dilatometer at 860 °C for 15 min, then quenched to 200 °C at a rate of 10 °C/s for a soaking time of 15 s; subsequently, two different heat treatment processes were carried out. One group was partitioned at different times (30, 90, 150, 240, 270, 300, 900, and 3600 s) at 270 °C (Figure 1a); the other group was partitioned at different temperatures (270, 300, 330, and 360 °C) (Figure 1b). The cooling rate after partitioning was 10 °C/s. The phase transformation behavior of steels during partitioning was analyzed using a dilatometer curve. According to the relationship between the amount of transition and isothermal time, the experimental data were fitted with the JMAK equation. The samples were subjected to X’Pert Powder XRD experiments to measure the volume fraction of retained austenite at ambient temperature. The carbon concentration of retained austenite
was determined by XRD analysis using a Bruker D8 Advance diffractometer (Bruker, Fremont, CA, USA). Cu Kα radiation was used, with a 2θ range from 30° to 135°, containing the (111), (200) and (220) austenite reflections, and scanned using a step size of 0.05°. TEM experiments were also utilized on the Tecnai G2 F20 (FEI Corporation, Hillsboro, OR, USA) with an acceleration voltage of 200 kV, and the samples were prepared using a twin-jet technique in a 10% perchloric acid and 90% acetic acid solution.

![Figure 1](image.png)

Figure 1. Schematic graph of the quenching and partitioning treatment used in this study. (a) Different partitioning times. (b) Different partitioning temperature.

3. Results and Discussion

In order to research the effects of partitioning time on the volume fraction of retained austenite, we conducted experiments with a dilatometer and XRD. Figure 2a is the continuous cooling curve of 60Si2CrVA steel, and 2b is the length change during the whole Q & P process. The $M_t$ temperature was measured to be 260 °C. It was obvious that not all of the austenite transformed into martensite, and some austenite remained when firstly quenched to 200 °C ($T_q$). We call this remaining austenite “metastable austenite”. As shown in Figure 3, the dilatation of the sample was observed upon partitioning. There was no incubation period because the curve began to swell as soon as the partitioning began. At the beginning of the partitioning, the length changed rapidly, and, with the increase of isothermal time, the rate of dilatation of the sample dropped significantly. Length changes were very conspicuous at 0–3000 s and gentle at 3000–3600 s. The expansion of the sample indicated that some changes had occurred during the isothermal process. According to relevant literature, there may have been martensite or bainite formation in the metastable austenite during the partitioning stage, which may have led to volume expansion.

It is indicated that there was a large transition rate at the beginning of partitioning, and with the increase of the isothermal time, the rate gradually decreased, and the transition was nearly completed after 3000 s. In order to acutely investigate the decomposition of metastable austenite, the volume fractions of the retained austenite at different partitioning times of 30, 90, 150, 240, 270, 900, and 3600 s (Figure 1a) were measured by XRD, and the volume fractions of the retained austenite were 8.5%, 10.6%, 13.1%, 17.6%, 16.9%, 14.64%, and 11.4% respectively. As shown in Figure 4, the volume fraction of the retained austenite increased as the partitioning time increased, and it arrived at the maximum when the partitioning time was 270 s, and then it decreased. The maximum volume fraction of the retained austenite was 17.6% when the partitioning time was 270 s. To account for this phenomenon, the temperature change in the length curve during cooling to room temperature was analyzed.

As shown in Figure 5, a bent phenomenon was observed in the temperature change in the length curve during cooling, indicating that the phase transformation of the second martensite had occurred. The temperature corresponding to the bending is considered to be the temperature in which the phase transformation of the second martensite occurred ($M_t'$). As the partitioning time increased, the $M_t'$ decreased continuously. The relationship of temperature change in length became linear when the partitioning time exceeded 270 s. It is indicated that there was no secondary martensite formation. According to Figure 4, when the partitioning time was 270 s, the volume fraction of retained austenite reached the maximum.
As shown in Figure 5, a bent phenomenon was observed in the temperature change in the length curve during cooling, indicating that the phase transformation of the second martensite had occurred. The temperature corresponding to the bending is considered to be the temperature in which the phase transformation of the second martensite occurred ($M_S'$). As the partitioning time increased, the $M_S'$ decreased continuously. The relationship of temperature change in length became linear when the partitioning time exceeded 270 s. It is indicated that there was no secondary martensite formation.

When the partitioning time exceeded 270 s, the reduction of retained austenite content was considered to be caused only by the decomposition of metastable austenite. The change of the curve in Figure 3 was positively correlated with the amount of metastable austenite decomposition. The length expansion from 270 to 900 s partitioning was 4.78 µm (Figure 3: A,B), corresponding to the metastable austenite decomposition of 2.86% (Figure 4: D,E); from 900 to 3600 s, the length expansion was 4.22 µm (Figure 3: B,C), corresponding to the metastable austenite decomposition of 2.53% (Figure 4: E,F). Therefore, it can be concluded that every 1 µm expansion of the sample during the partitioning process corresponded to the decomposition of 0.6% in the metastable austenite. Therefore, based on this relationship, the following expression can be given:

$$ f_{MA} = 17.6 + 0.6 \times (I_{270} - I_t) \quad (1) $$
where \( f_{MA} \) is the volume fraction of metastable austenite during different partitioning times, \( l_{270} \) and \( l_t \) represent the length change at the partitioning time 270 s and \( t \), respectively. When the partitioning time was 30, 90, 150, and 240 s, the value of \((l_{270} - l_t)\) was 3.73, 2.27, 1.38, and 0.23 \( \mu \)m, respectively. Therefore, according to Equation (1), the volume fraction of metastable austenite \( f_{MA} \) was calculated to be 19.48%, 18.96%, 18.43%, and 17.74%, respectively. The amount of secondary martensite \( (M') \), which was produced during the cooling to ambient temperature when the partitioning time was 30, 90, 150, and 240 s, can be obtained through the metastable austenite volume fraction minus that of retained austenite measured by XRD, correspondingly. The contents were 10.91%, 8.76%, 6.93%, and 3.24%, respectively.

![Figure 5](https://example.com/figure5.png)

**Figure 5.** Change in length of different partitioning times during final cooling.

The volume fraction of prime martensite is given by the Koistinen–Marburger relationship [16,17]:

\[
\frac{\alpha}{M} = 1 - \exp(-\alpha \times (T_p - T_q))
\]

(2)

where \( \frac{\alpha}{M} \) is the volume fraction of prime martensite when the prime quenching was completed; \( \alpha \) is a constant related to the material, which is usually \( a = 1.1 \times 10^{-2} \) (K\(^{-1}\)) in low alloy steel; and \( T_q \) is the temperature of prime quenching.

According to Equation (2), when the quenching temperature was 200 °C, the volume fraction of prime martensite was 45.12%.

Finally, the variation of various microstructure contents with the partitioning time is shown in Figure 6.

![Figure 6](https://example.com/figure6.png)

**Figure 6.** The effect of partitioning time on the volume fraction of the individual microstructural constituents.
After the partitioning time exceeded 270 s, no secondary martensite transformation occurred during the final quenching to ambient temperature. This phenomenon is due to carbon diffusion from supersaturated martensite to metastable austenite during the partitioning process. Increased carbon content in the metastable austenite caused the \( M_S' \) temperature to decrease [18]. The stability of austenite was enhanced by the addition of carbon content. When the partitioning time was 270 s, the carbon content in the metastable austenite was just enough to make it stable when cooled to ambient temperature. It is indicated that it takes a period of time for the carbon to be partitioned from supersaturated martensite to metastable austenite during the partitioning process. Therefore, when the partitioning time exceeded 270 s, the final content of the retained austenite was determined by the amount of metastable austenite decomposition only. The volume fraction of the retained austenite decreased as the partitioning time increased.

According to the CCE model, which was initially proposed by Speer et al. [1,19], the carbon concentration of metastable austenite is 0.94 wt.% when the quenching temperature is at 200 °C. Therefore, according to the Equation (3), which was proposed by Andrew [20], the \( M_S' \) temperature should be 96.7 °C.

\[
M_S' = 539 - 423X_C - 30.4X_{Mn} - 12.1X_{Cr} - 7.5X_{Si}
\]  

(3)

where \( X_C, X_{Mn}, X_{Cr}, \) and \( X_{Si} \) represent the concentrations of carbon, manganese, chromium, and silicon, respectively, in wt.%. 

In fact, the \( M_S' \) was 160 °C after 30 s partitioning (shown in Figure 5). It is suggested that the carbon concentration in the metastable austenite, which was calculated based on the CCE model, was higher than that obtained by the experiment.

The carbon concentration of the retained austenite with partitioning for 30 s was measured by XRD. The carbon concentration \( X_C' \) of the retained austenite was obtained using [21–23]:

\[
\alpha_y = 0.3556 + 0.00453X_C' + 0.000095X_{Mn} + 0.0006X_{Cr}
\]  

(4)

where \( \alpha_y \) is the austenite lattice parameter, in nm.

The XRD pattern of the sample that was partitioned for 30 s is shown in Figure 7. The lattice parameter of the retained austenite (\( \alpha_y \)) was 3.5986. According to Equation (4), the carbon concentration in the retained austenite was 0.79 wt.%. This deviation between the actual measurements and calculations by the CCE model is due to the CCE model ignoring the precipitation of carbides during the partitioning process and assuming full carbon partitioning, which is not the case.
3.1. The Kinetics of Transformation

As can be seen from the above analysis, the thermal expansion curve can reflect the decomposition law of metastable austenite in the process of partitioning. According to Equation (2), the variation of metastable austenite decomposition can be obtained during the partitioning treatment. Then, it is fitted with the classical JMAK equation to obtain the Avrami exponent and phase transition activation energy. The JMAK equation was adjusted as follows [24, 25]:

\[
f / f_{\text{max}} = 1 - \exp(-bt^n) \tag{5}\]

where \(f\) is the amount of metastable austenite that has been decomposed during the partitioning process, \(f_{\text{max}}\) is the maximum amount of metastable austenite after transformation, \(b\) is a constant related to the activation energy of the phase transition, \(t\) is the partitioning time, and \(n\) is the Avrami exponent, which can reflect the nucleation and growth mechanism of the new phase; and where \(b\) can be written as:

\[
b = K_0 \exp\left(\frac{Q}{RT}\right) \tag{6}\]

where \(K_0\) is the pre-exponential factor, \(Q\) is the transformation active energy, and \(R\) is the gas constant.

Appropriate simplification of the JMAK equation can be obtained as follows:

\[
\ln(n\ln(\frac{1}{1 - f / f_{\text{max}}})) = \ln b + n \ln t \tag{7}\]

\[
\ln b = \ln K_0 - \frac{Q}{R} \cdot \frac{1}{T} \tag{8}\]

The data of Figure 3 were processed, with \(\ln t\) as the abscissa and \(\ln(n\ln(\frac{1}{1 - f / f_{\text{max}}}))\) as the ordinate as shown in Figure 8. The slope is the Avrami exponent (\(n\)) when the metastable austenite was decomposed.

![Figure 8](image)

*Figure 8. Fitting curve \(\ln t—\ln \ln(1/(1 - f / f_{\text{max}}))\) diagram.*

According to the Avrami model, during the process of isothermal transformation, if it is a site saturation nucleation model, the Avrami exponent \(n = d/m\), where \(d\) is the dimensionality of the growth, \(m\) depends on the growth mode: \(m = 1\) for interface-controlled growth, and \(m = 2\) for diffusion-controlled growth [26].

According to the expansion curve at the partitioning temperatures of 270, 300, 330, and 360 °C, \(\ln \ln(1/(1 - f / f_{\text{max}}))\) versus \(\ln t\) was plotted, as shown in Figure 8. It can be seen that the Avrami exponent was close to 1 during the partitioning process, indicating that the new phase may have
been a 2-dimensional growth model controlled by diffusion. According to the phase transition theory, the formation of a new phase may have been needle-like.

Combining Equation (8) and Figure 8, the activation energy for phase transformation $Q$ was obtained to be 141 kJ·mol$^{-1}$, which is almost the same as that of the lower bainite formed at a lower temperature studied by Okamoto et al. [27]. Therefore, it can be inferred that the new phase formed by metastable austenite decomposition during the partitioning process was likely to be lower bainite, or its formation mechanism was the same as that of lower bainite.

The transformation kinetics equation of metastable austenite during the partitioning process was:

$$f / f_{\text{max}} = 1 - \exp(-0.00128t)$$  (9)

It can be assumed that with the increase of partitioning time, the metastable austenite decomposed to bainite continuously. The carbon potential in the bainite was higher than that in the metastable austenite. Therefore, some carbon was transported to the remaining metastable austenite. For this reason, the carbon content of the remaining austenite increased and the stability also increased gradually. The metastable austenite decomposition was reported to stop when the austenite was enriched to a carbon concentration near $T_0$ [28,29] ($T_0$ is the locus of temperatures/compositions where the free-energy of ferrite and austenite are equal [19].) This is also an important reason to increase the stability of metastable austenite.

3.2. Microstructure Analysis

Following quenching to 200 °C after austenitizing at 860 °C, a mixed microstructure of martensite and metastable austenite was formed [30]. Figure 9a,b shows the microstructure with 30 s partitioning. It can be found that there were two morphologies of martensite, namely, lath martensite and lenticular martensite. It has been proven by the curve in Figure 5 that secondary martensite was produced in metastable austenite with a higher carbon concentration than bulk alloys (carbon diffused to metastable austenite during the isothermal process). The high carbon concentration in steel tends to produce lenticular martensite during quenching. Therefore, the lenticular martensite in Figure 9b is considered to be secondary martensite. In the subsequent isothermal process, a part of the metastable austenite was decomposed. Figure 9c,d is the light and dark field images with 3600 s partitioning, respectively. The black part in Figure 9c is the isothermal products out of metastable austenite, and the white part in Figure 9d is the retained austenite [31]. In contrast to the retained austenite, the products formed during the partitioning process curve at the boundary. During the partitioning process, the new phase formed by the metastable austenite decomposition was perpendicular to the phase interface of retained austenite/martensite and grew with a short needle-like shape to the interior of the retained austenite. The length of the new phase was about 1.43 µm, and the width was about 0.15 µm. After partitioning, the length and width of the retained austenite was about 0.59 µm and 0.07 µm, respectively. The morphology of the new phase provides confirmation that the Avrami exponent derived from the JMAK equation is 1. The decomposition process of metastable austenite in the process of partitioning was similar to the bainite growth model proposed by Bhadeshia [32], i.e., that the subunit of the new phase grows continuously along the phase boundary. Finally, the blocky metastable austenite was divided into lath shapes by the new phase, and the carbon in the supersaturated martensite and new phase diffused to metastable austenite, resulting in untransformed metastable austenite with excellent stability.
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Figure 9. TEM image after Q & P: (a) lath martensite (b) lenticular martensite (c) isothermal production, 
and (d) retained austenite (dark film).

4. Conclusions

(1) The partitioning time of Q & P treatment of 60Si2CrVA steel can significantly affect the volume 
fraction of retained austenite. In this study, the volume fraction of the retained austenite firstly increased, 
with the increase of the partitioning time, and then decreased. When the partitioning time was less 
than 270 s, the volume fraction of the retained austenite was determined by both the decomposition of 
metastable austenite during the isothermal process and second martensite transformation occurring 
during the final quenching. When the partitioning time exceeded 270 s, the final volume fraction of the 
retained austenite was merely determined by the amount of metastable austenite decomposition.

(2) Metastable austenite undergoes a transition from the γ phase to the α phase during the 
partitioning process. Through fitting the JMAK equation, the Avrami exponent of metastable austenite 
decomposition process is 1 and the transformation active energy is 141 kJ·mol⁻¹.

(3) The original block metastable austenite was divided into laths by the decomposition product 
that was formed during the partitioning process, and the decomposition product of the same metastable 
austenite grains presented the same crystallographic orientation relationship. According to the kinetic 
model, the decomposition product of metastable austenite during the partitioning process was bainite. 
Diffusion of carbon from both primary martensite and bainite to metastable austenite is a process that 
increases the stability of metastable austenite.

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