CRYSTALLOGRAPHY AND INTERFACE BOUNDARY STRUCTURE OF PEARLITE WITH M$_7$C$_3$ CARBIDE LAMELLAE

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Abstract—The crystallography of pearlite with M$_7$C$_3$ carbide lamellae and the atomic structure at the ferrite/carbide interface have been examined by means of transmission electron microscopy in an Fe–8.2Cr–0.96C alloy. Two orientation relationships with the corresponding habit planes were determined: OR-I: (251)$_b$/*(1120)$_a$–habit plane, (113)$_b$//*(1100)$_a$; (311)$_b$ ///*(0001)$_a$; OR-II: (251)$_b$//*(1100)$_a$–habit plane, (113)$_b$//*(1120)$_a$; (311)$_b$ ///*(0001)$_a$. Variants of these orientation relationships have been frequently observed. Microscopic steps at the ferrite/carbide interfaces accommodate the curvature of the habit planes. Each of these orientation relationships provides a small misorientation between the {110}$b$ close packed planes of ferrite and the coincident planes of M$_7$C$_3$ carbide, and the atomic planes are perfectly matched through the interface. The orientation relationship between the parent austenite and M$_7$C$_3$ carbide was also deduced assuming the ferrite/austenite orientation relationship so far obtained.

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ations, namely the Pitsch–Petch OR and the Bagaryatskii OR probably do not exist.

Whereas the crystallography between pearlitic ferrite and cementite has been studied rather intensively, very little attention has been given to the crystallography between ferrite and alloy carbides. It is well defined that in pearlite of alloyed steel the cementite can be replaced by other alloy carbides, such as M₂₃C₆ [25] or M₇C₃ [26]. The crystallography of the austenite–pearlite transformation in Fe–Cr–C alloys has been studied by Howell et al. [25]. In all cases the M₂₃C₆ carbide lamellae were related to the adjacent ferrite by the Kurdjumov–Sachs orientation relationship [27].

To the knowledge of the present authors, the crystallography of the pearlite with M₇C₃ carbide lamellae has not been studied in detail. Thus the present study was undertaken in order to reveal the habit plane and orientation relationship between pearlitic ferrite and M₇C₃ carbide in an Fe–8.2Cr–0.96C alloy.

2. EXPERIMENTAL PROCEDURE

2.1. Sample preparation

The chemical composition of the alloys used in the present investigation is shown in Table 1. Specimens of 20 mm in diameter and 10 mm long were austenitized at 1250°C for 15 min in a dynamic argon atmosphere. They were transformed isothermally in a lead bath covered with a layer of active charcoal to prevent decarburization at 725°C for 720 s, then quenched into iced brine. These heat treatments resulted in the lamellar pearlite containing M₇C₃ carbide lamellae with an average interlamellar spacing of about 0.1 μm.

The specimens for the microstructural investigations were sliced from the centre of each bulk specimen. Thin foils for TEM studies were prepared from 3 mm discs, ground to a thickness of about 0.05 mm and electropolished by a conventional twin-jet polishing method using electrolyte containing 10% perchloric acid, 20% glycerol and 70% ethanol. The foils were examined in a JEM-3010 transmission electron microscope operating at 300 kV.

2.2. Method for determining the habit plane

The accurate determination of the habit plane was fulfilled through large-angle tilting in the transmission electron microscope. This method is described in detail in Ref. [9]. Tilting toward a point where the boundary image is the sharpest results in the case when the electron beam is parallel to the habit plane. Once found the habit plane was further checked by tilting around an axis which was found to be normal to the habit plane. The zone axis was changed but the interface remains perpendicular to the tilting axis. Since there are many reflections on the line normal to the direction of a habit plane trace in the stereographic projection, one of them close to the reflecting zone can be accepted as a true habit plane normal. Thus such a procedure will exclude the erroneous interface normal.

2.3. Structure of the M₇C₃ carbide

Selected area electron diffraction (SAED) patterns from the M₇C₃ carbide are generally complex because of the pseudo-hexagonal structure containing 56 iron atoms and 24 carbon atoms in a unit cell with lattice parameters $a = 1.3982$ nm and $c = 0.4506$ nm [28]. It has frequently been observed that the corresponding SAED patterns from the M₇C₃ carbide contain long streaks through the reciprocal lattice space perpendicular to {1010} planes [29, 30]. These streaks result from a faulted structure with the fault vector being one half of the unit cell repeat distance [31]. The observed distribution of the diffuse intensity along the (1120) directions in the Fe-C₃ carbide has been attributed to a structure composed of ordered microdomains separated by non-periodic {1120} defects [32]. In the present study, since the streaks were frequently observed along (1120) and (1100) directions, this phenomenon is thought to arise from the presence of defects in planes perpendicular to the basal plane. The structure of the M₇C₃ phase can also be interpreted as the orthorhombic crystal structure with $a = 0.4532$ nm, $b = 0.7015$ nm and $c = 1.2153$ nm as reported by Fruchart and Rouault [33] and was investigated in detail by Morinroli et al. [32].

3. EXPERIMENTAL RESULTS

A typical pearlite area with M₇C₃ carbide lamellae is shown in Fig. 1(a). The SAED pattern taken from the ferrite/carbide area [Fig. 1(c)] allows their crystallography to be determined. The stereographic analysis for this diffraction pattern is shown in Fig. 1(e). The incident beam direction is $[113]_b/[[1100]_{hex}}$. (Hereafter, the subscripts b, f, hex and orth denote the b.c.c. structure of ferrite, the f.c.c. structure of austenite and the hexagonal or orthorhombic structure of the M₇C₃ phase, respectively.) Thus, the following orientation relationship between ferrite and M₇C₃ carbide was deduced:

$$[113]_b/[[1100]_{hex}}$$

$$[251]_b/[[1120]_{hex}}$$
The angle between \( \langle 251 \rangle_b \) and \( \langle 1120 \rangle_{\text{hex}} \) was determined to be about 1.5° whereas the \( \langle 311 \rangle_b \) is not strictly parallel to the \( \langle 0001 \rangle_{\text{hex}} \). Thus the above expression for this orientation relationship is just an ideal approximation. This is termed the OR-I. The habit plane determined is the \( \langle 251 \rangle_b \) parallel to \( \langle 1120 \rangle_{\text{hex}} \). However, with an orientation such as in Fig. 1(c), it is difficult to assert that the habit plane is not \( \langle 0110 \rangle_{\text{hex}} \) but \( \langle 1120 \rangle_{\text{hex}} \).
Figures 1(b) and (d) are, respectively, a bright field image of pearlite and the corresponding SAED pattern taken after tilting around the axis [251]_{hex}/[1120]_{hex} on the angle of 17.9°. In this case the incident beam direction is [324]_{b}. The corresponding displacement of the ferrite and M7C3 carbide reflections is shown in the stereographic projection by arrows. From such an orientation it is rather easy to see that the habit plane is indeed the (251)_{b}/(1120)_{hex}.

The HRTEM micrograph of Fig. 2 shows the atomic structure of the interphase boundary at the edge of the M7C3 carbide lamella. The corresponding SAED pattern is shown in Fig. 1(b). The incident beam is [113]_{b}/[1100]_{hex}. In this figure, the ferrite/carbide interface is atomically flat. It can be seen that the M7C3 carbide habit plane is accurately parallel to the atomic habit plane (251)_{b}/(1120)_{hex}. The (110)_{b} ferrite close packed planes are parallel to the (3361)_{hex} planes of the M7C3 phase and atomic planes are perfectly matched across this interface. Thus, this M7C3 carbide should maintain good coherency with respect to the matrix.

Figures 3(a) and (b) show the TEM micrographs of a pearlite area in different view directions. Their diffraction patterns and the summarized stereographic analysis are shown through Figs 3(c)–(e). The incident beam direction in Figs 3(a) and (c) is [113]_{b}/[1120]_{hex} whereas Figs 3(b) and (d) were obtained through tilting around the axis [251]_{b}/[1100]_{hex} through an angle of 25.5°. Such a tilting procedure once again allows the habit plane to be unambiguously recognized. The lamellar area in this case is parallel to the atomic habit plane (251)_{b}/(1100)_{hex}. The orientation relationship between ferrite and M7C3 carbide lamellae can be expressed as follows:

\[
[113]_{b}/[1120]_{hex}
\]

This is termed the OR-II. Similar to the OR-I, the angle between [251]_{b} and [1100]_{hex} was determined to be 1.5° but the [311]_{b} is deviated from the [0001]_{hex}. These deviations were seen in all of the areas studied at different view directions although this orientation relationship itself obeys well. Note that the OR-II is identical to the orientation relation no. 2 for M7C3 carbide in ferrite after tempering of alloy steels reported by Dyson and Andrews [33].

Figure 4 is the HRTEM micrograph showing an edge-on view of the atomic structure of the broad face of the M7C3 carbide lamella. The incident beam direction is [113]_{b}/[1120]_{hex}. The corresponding SAED pattern is shown in Fig. 3(b). Note that in the ferrite area, only the (110)_{b} lattice fringes can be seen. The angle between (110)_{b} and (251)_{b} planes in ferrite is 25.35°, thus the (251)_{b} planes are almost parallel to the (1100)_{hex} planes. It can be seen that the (110)_{b} planes are almost parallel to the (550)_{hex} and the atomic planes are perfectly matched across the interface suggesting good coherency, because extra half planes at the broad face of the M7C3 carbide lamella are not observed.

Figure 5 is a lattice image showing the (101)_{b} planes of ferrite and (1100)_{hex} planes of M7C3 carbide. This micrograph was taken at the diffraction conditions shown in Fig. 3(d). It can be seen that the ferrite habit plane deviates about 1.5° from the (251)_{b}/(1100)_{hex}, which is the broad face on the habit plane. Figure 5 also shows that there are steps on both sides of the ferrite/carbide interface aligned in the growth direction of the carbide [311]_{b}/[0001]_{hex}. The step heights are one to three unit lattice heights of the M7C3 phase in the [1100]_{hex} direction. These steps accommodate the divergence of the interface from the atomic habit plane, which is similar to that observed in the ferrite/cementite pearlite [2]. Note that the edge dislocation is visible within M7C3 carbide lamella. The core region of the extra half (1100)_{hex} plane is shown in this figure by a circle.

Figure 6 is an HRTEM image showing a good atomic matching between the (011)_{b} close packed planes of ferrite and the (4621)_{hex} planes of the M7C3 carbide. The angle between them was determined to be about 2.5°.

Two sets of the bright field images and the corresponding SAED patterns in Fig. 7 show two different variants of the OR-II. In Figs 7(a) and (c) the incident beam is close to [011]_{b}/[1121]_{hex}. The stereographic projection in Fig. 7(e) shows that the OR-II is fulfilled within a few degrees as follows:

\[
(512)_{b}/(1100)_{hex}
\]
The [113]_h zone axis shows even better correlation between ferrite and carbide reflections for the OR-II [as shown in Fig. 7(c) by arrows]. It can be easily seen that the habit plane of the carbide lamellae is (512)_h/(1100)_hex. Meanwhile, for the sake of clarity, the following discussion is more convenient to continue using the [011]_h zone axis. Figures 7(b) and (d) are the bright field micrograph and the cor-
responding SAEDP taken from another pearlite colony. The incident beam direction is \( [113]_b / [1120]_h \).

The habit plane is close to \( (251)_h / (1100)_h \).

Figure 4. HRTEM image of the edge of the M\(_7\)C\(_3\) carbide lamella. The incident beam direction is \( [113]_b / [1120]_h \).

The habit plane is close to \( (251)_h / (1100)_h \).

Finally, it should be mentioned that the two orientation relationships reported above were observed in 15 different pearlite colonies. The OR-II was observed somewhat more often than OR-I and no other orientation relationships were observed.

4. DISCUSSION

4.1. Ferrite/carbide crystallography

In the present study, the crystallography and atomic structure of the interfaces in the ferrite/M\(_7\)C\(_3\) pearlite was studied. The experimental observations described above can be summarized as follows.
1. The ferrite/M₇C₃ pearlite mainly grows along (311)ₜ//(0001)ₜ.

2. Two specific orientation relationships were found to exist between the M₇C₃ carbide lamellae and the ferritic matrix.

3. Each of these orientation relationships has a unique habit plane:

   **OR-I:**

   \[ (251)ₜ//(1120)ₜ \]  habit plane

   \[ (113)ₜ//(1100)ₜ \]

   \[ (311)ₜ \approx // (0001)ₜ \]

   **OR-II:**

   \[ (251)ₜ//(1100)ₜ \]  habit plane

   \[ (113)ₜ//(1120)ₜ \]

   \[ (311)ₜ \approx // (0001)ₜ \]

Since the angle between (311)ₜ and (521)ₜ is 7.75°, both orientation relationships are very close but the habit planes are quite different. Because of the hexagonal symmetry of the M₇C₃ phase, its three \[ (1010)ₜ \] planes and three \[ (2110)ₜ \] planes are equivalent. Thus the OR-I and OR-II can be obtained by a rotation of 30° about their common axis \[ (311)ₜ \approx // (0001)ₜ \] to one another.

4. In the case of the OR-II, the habit plane was observed to deviate about 1.5° from the \[ (251)ₜ//(1100)ₜ \].

5. The ferrite close packed planes were observed to be within a few degrees of the \[ (5051)ₜ \], \[ (3361)ₜ \] or \[ (2461)ₜ \] planes of the M₇C₃ carbide lamella. Across the interface, these planes are matched with perfect coherency, since the interface does not contain extra half planes.

6. The growth of the pearlite is accompanied by steps at the broad face of the M₇C₃ carbide lamella.

For further consideration of the orientation relationships between ferrite and M₇C₃ carbide, it is useful to express the structure of the M₇C₃ phase as orthorhombic. Using the convention \[ a = 0.4532 \text{ nm}, \quad b = 0.7015 \text{ nm} \] and \[ c = 1.2153 \text{ nm} \] reported by Fruchart and Rouault [34], the OR-I and OR-II can be expressed as follows:

   **OR-I:**

   \[ (251)ₜ//(010)ₘ \]  habit plane

   \[ (113)ₜ//(001)ₘ \]

   \[ (311)ₜ \approx // (100)ₘ \]

   **OR-II:**

   \[ (251)ₜ//(001)ₘ \]  habit plane

   \[ (113)ₜ//(010)ₘ \]

   \[ (311)ₜ \approx // (100)ₘ \]

It is important to emphasize that the OR-II is similar to the Pitsch–Petch orientation relationship, which was often observed between ferrite and cementite in pearlite. For the sake of completeness,
it should be noted that the structure of cementite can also be described as hexagonal [35, 36].

It has been shown by Ohmori et al. [22] and confirmed by Zhou and Shiflet [9] that the Pitsch–Petch orientation relationship yields low interface energy. Minimization of interface energy is considered as one of the most important factors of the fixed orientation relationship and a one-to-one correspondence between the habit plane and orientation relationship. Minimization of strain energy at the interface requires good atomic matching. Zhang and Kelly [11, 37, 38] suggested that the orientation
relationships should be expressed in terms of the close packed planes that show good atomic matching.

Figure 9 represents the stereographic projection on which the common axis for the OR-I and OR-II parallel to the growth direction of the pearlite lamellae is fixed. The OR-I or OR-II can simultaneously be expressed as follows:

**OR-I:**

\[
\begin{align*}
(51\bar{2})_h/\langle 1120 \rangle_{\text{hex}} \\
(\bar{1}3\bar{1})_h/\langle 1100 \rangle_{\text{hex}}
\end{align*}
\]

**OR-II**

\[
\begin{align*}
(2\overline{5}\bar{1})_h/\langle 1100 \rangle_{\text{hex}} \\
(3\bar{1}\bar{1})_h/\langle 11\bar{2}0 \rangle_{\text{hex}} \\
(11\bar{3})_h \approx \langle 0001 \rangle_{\text{hex}}
\end{align*}
\]

The stereographic projection also shows that each of the \{110\}_h close packed planes of ferrite is within a few degrees of one of the following planes in the M-C₃ phase: \{13\overline{2}2\}_\text{hex}, \{\overline{5}0\overline{5}1\}_\text{hex}, \{33\overline{6}\}_\text{hex}, \{6\overline{2}4\}_\text{hex}, \{7\overline{3}4\}_\text{hex} or \{1\overline{0}\overline{1}\}_\text{hex}. Each of these planes has the \(d\) spacing close to that of the \{110\}_h ferrite. The lattice misfits between these planes of the M-C₃ phase and the \{110\}_h planes of ferrite are summarized in Table 2. It can be seen that even the largest misfit does not exceed 5% whereas it is less than 2% in most cases.

### Table 2. Lattice misfit between ferrite and M-C₃ carbide planes

<table>
<thead>
<tr>
<th>(hh\phi_h)</th>
<th>Lattice spacing, (d_1) (nm)</th>
<th>(hh\phi_h)</th>
<th>Lattice spacing, (d_2) (nm)</th>
<th>((d_1-d_2)/d_1)</th>
</tr>
</thead>
<tbody>
<tr>
<td>{50\overline{5}1}</td>
<td>0.213</td>
<td>{\overline{6}3\overline{3}1}</td>
<td>0.207</td>
<td>{\overline{6}2\overline{4}1}</td>
</tr>
</tbody>
</table>
The 30° rotation around the common axis \( \{113\}_b \parallel \{100\}_\text{hex} \) yields 12 variants of either orientation relationship. Note that only in half of them is the \( f_{110} \) plane of the hexagonal \( M_7C_3 \) phase less than 2.5° to the close packed plane \( \{110\}_b \) of ferrite as shown in Fig. 9. It is obvious from the experimental observations [compare Fig. 1(e) with Fig. 3(e) and Fig. 7(e)] that the last condition is not strictly fulfilled.

4.2. Austenite/pearlite crystallography

The orientation relationships between the austenite and the two constituents of pearlite are also very important to observe the crystallography of the pearlite reaction as a whole. In the classical theory of pearlite growth developed by Smith [39] and Hillert [16], it is implicitly assumed that either of the pearlite constituents has no reproducible orientation relationship with the austenite grain it was growing into and the austenite/pearlite interface is disordered. This conclusion was supported by a number of other studies [40, 41]. Meanwhile, it has been shown by Shackleton and Kelly [42] that if the Pitsch austenite–cementite relationship and the Kurdjumov–Sachs austenite–ferrite relationship are assumed then the ferrite and cementite can be related by the Bagaryatskii orientation relationship within a few degrees.

Figure 10 represents the stereographic projection when the zone axis is \( \{311\}_b \parallel \{121\}_\text{hex} \). The OR-II found in the present study is shown by dashed lines. If we assume that the ferritic pearlite and austenite are related by the Kurdjumov–Sachs relationship (as shown by dotted lines), the following orientation relationship between the austenite and \( M_7C_3 \) carbide can be deduced (shown by solid lines):

- \( (111)_f \parallel \{01\overline{1}0\}_\text{hex} \)
- \( (\overline{1}01)_f \parallel \{2\overline{1}1\overline{0}\}_\text{hex} \)
- \( (\overline{1}2\overline{1})_f \parallel \{0001\}_\text{hex} \)

If the orthorhombic symmetry of the \( M_7C_3 \) phase is adopted this orientation relationship can be rewritten as follows:

- \( (111)_f \parallel \{001\}_\text{orth} \)
- \( (\overline{1}01)_f \parallel \{010\}_\text{orth} \)
- \( (\overline{1}2\overline{1})_f \parallel \{100\}_\text{orth} \)

This orientation relationship is quite similar to the Pitsch orientation relationship in the case of cementite precipitation in austenite. Although this orientation relationship should experimentally be confirmed, it should be noted that, if this is true, the austenite/pearlite interface becomes coherent. The recent results by Hackney and Shiflet [10] also indicated the partial coherency at the austenite/ferrite/cementite interfaces despite the fact that rational orientation relationships are seldom confirmed.

5. SUMMARY

The crystallography and ferrite/carbide interface structure of pearlite with \( M_7C_3 \) carbide lamellae have been investigated by means of TEM using an SAED pattern technique. The following results were obtained.

1. In pearlite, two orientation relationships between ferrite and the \( M_7C_3 \) phase have been determined. Each of them has a unique atomic habit plane:
   - OR-I:
     \( \{2\overline{1}1\}_f \parallel \{1\overline{1}20\}_\text{hex} \) habit plane
   - OR-II:
     \( (13)_f \parallel \{\overline{1}100\}_\text{hex} \)
     \( (\overline{3}1\overline{1})_f \parallel \{0\overline{0}01\}_\text{hex} \)
In the case of the OR-II, the habit plane of an M-C₃ carbide lamella deviates by 1.5° from the (251)h // (1100)hex habit plane.

\[(113)_{\text{h}} // (1120)_{\text{hex}}\]

\[(311)_{\text{h}} \approx // (0001)_{\text{hex}}\]

2. In the case of the OR-II, the habit plane of an M-C₃ carbide lamella deviates by 1.5° from the (251)h // (1100)hex.

3. The misorientation between the close packed planes of ferrite and the coincident planes of M-C₃ carbide is always within a few degrees and atomic planes are perfectly matched through the interface. This implies that both pearlite constituents form coherently to one another.

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