THE LATTICE PARAMETERS OF AUSTENITE AND FERRITE IN Fe-C ALLOYS AS FUNCTIONS OF CARBON CONCENTRATION AND TEMPERATURE

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1. Introduction

Despite its relevance for various calculations involving phase transformations in Fe-C alloys, little information is available on the lattice parameter of austenite at elevated temperatures and its dependence on the carbon concentration. Furthermore, severe scatter exists in the literature for the lattice parameter of ferrite at high temperature. Most literature data were acquired using X-ray diffraction (1-6), although neutron diffraction seems to be a more suitable technique for determining the lattice parameter of austenite and ferrite in Fe-C alloys at high temperatures. Since neutrons penetrate and probe a large volume of material, the advantages of neutron diffraction over X-ray diffraction are its reduced sensitivity to surface decarburization and improved crystal statistics (both decarburization and grain growth can occur during diffraction experiments at high temperatures requiring long exposure times). In this work in-situ neutron diffraction experiments on Fe-C alloys were performed to determine the lattice parameter of the austenitic and ferritic phases in a temperature region from just below to just above the bi-phasic austenite/ferrite region.

2. Experimental

Five iron-carbon alloys were prepared by arc-melting iron under an argon flow while adding pure graphite to obtain the carbon concentration required. The chemical composition after hot rolling was determined using Inductively Coupled Plasma Optical Emission Spectroscopy combined with Atomic Absorption Spectroscopy. The chemical composition of each alloy is given in Table 1. The total impurity level in the alloys is less than 0.02 wt.%, with the exception of the Fe-0.8 C alloy which contains 0.06 wt.% of contaminants. In the case of the Fe-0.3 C, Fe-0.4 C and Fe-0.8 C alloys, cylindrical samples were made with a length of 50 mm and an outer diameter of 6 mm. The Fe-0.6 C sample diameter was 4 mm. The Fe sample consisted of an ensemble of two strips each of 50 mm length, 4 mm width and 2 mm thickness. The samples were not subjected to a normalising heat treatment before the measurements.

<table>
<thead>
<tr>
<th>Sample</th>
<th>C</th>
<th>Cr</th>
<th>Cu</th>
<th>Mn</th>
<th>Mo</th>
<th>Ni</th>
<th>P</th>
<th>S</th>
<th>Sn</th>
<th>Total</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fe</td>
<td>0.01</td>
<td>0.001</td>
<td>0.001</td>
<td>0.0007</td>
<td>0.002</td>
<td>0.003</td>
<td>&lt; 0.001</td>
<td>&lt; 0.005</td>
<td>0.002</td>
<td>&lt; 0.016</td>
</tr>
<tr>
<td>Fe - 0.3 C</td>
<td>0.28</td>
<td>0.002</td>
<td>0.001</td>
<td>0.0002</td>
<td>&lt; 0.002</td>
<td>&lt; 0.001</td>
<td>&lt; 0.004</td>
<td>&lt; 0.005</td>
<td>&lt; 0.004</td>
<td>&lt; 0.019</td>
</tr>
<tr>
<td>Fe - 0.4 C</td>
<td>0.38</td>
<td>&lt; 0.001</td>
<td>0.001</td>
<td>0.0001</td>
<td>&lt; 0.002</td>
<td>&lt; 0.001</td>
<td>&lt; 0.003</td>
<td>&lt; 0.005</td>
<td>&lt; 0.004</td>
<td>&lt; 0.016</td>
</tr>
<tr>
<td>Fe - 0.6 C</td>
<td>0.57</td>
<td>0.001</td>
<td>0.002</td>
<td>0.0009</td>
<td>0.002</td>
<td>0.001</td>
<td>&lt; 0.001</td>
<td>&lt; 0.005</td>
<td>0.001</td>
<td>&lt; 0.014</td>
</tr>
<tr>
<td>Fe - 0.8 C</td>
<td>0.80</td>
<td>0.0002</td>
<td>0.006</td>
<td>0.0005</td>
<td>&lt; 0.002</td>
<td>0.003</td>
<td>0.038</td>
<td>0.005</td>
<td>0.003</td>
<td>&lt; 0.056</td>
</tr>
</tbody>
</table>

The neutron diffraction experiments were performed over the temperature range 863 K - 1253 K. The specimens were radiation heated in a vacuum (typically of the order of 3x10^{-8} bar) using a graphite resistance heater. The temperature was measured using two chromel-alumel thermocouples, one positioned at the top and one positioned at the bottom of the specimen. Temperature differences along the length of the specimen were measured to be within 2-3 K. Both thermocouples were shielded from the neutron beam by boron nitride caps to avoid diffraction from the
thermocouple material. The exposed volume for the Fe-0.3 C, Fe-0.4 C and Fe-0.8 C alloys was about 1100 mm³, for the Fe-0.6 C alloy about 500 mm³ and for the Fe sample about 640 mm³. A third thermocouple, not connected to the specimen, was used for temperature control. The time-temperature profile imposed on the specimen was as follows. The samples were heated to a temperature of 1250 K in approximately 40 minutes. To equilibrate the system the sample was kept at this temperature for 20 minutes. The specimens were subsequently cooled in steps of 20 K. At each temperature two diffraction patterns were collected, in order to check whether equilibrium had been reached. The time required for the collection of one diffraction pattern was 20 minutes. The temperature was always within ±2 degrees of the set temperature.

The experimental arrangement allowed free expansion of the sample in all directions, while accurate specimen alignment was maintained. The sample was not rotated during the experiment. The layout of the experimental arrangement is shown schematically in Figure 1. The DUALSPEC high-throughput neutron diffractometer includes an 800 wire, position-sensitive detector with a single BF₃ gas chamber. The detector arc spans 80° in steps of 0.1° 2θ, starting at a continuously variable angle. The starting angle in this study was 39.46° 2θ. The monochromator for the incident neutron beam is a squeezed silicon single crystal with a mosaic spread of about 0.2°. Diffraction from the (115) planes at a diffraction angle of 109.39° 2θ was used to obtain a monochromatic beam. The neutron wavelength and the detector starting angle were determined by collecting the diffraction pattern from a certified standard powder, Al₂O₃ (7). The neutron wavelength was found to be 0.170597 ± 0.000005 nm.

In the diffraction patterns the following reflections were observed (depending on the temperature): [111], [200], [220], [113] and [222] for austenite and [110], [200], [112] and [220] for ferrite. The intensities in the peak maximum varied between 400 and 9000 counts for the least and most intense peaks. The full width at half maximum of the observed reflections varied between 0.2 and 0.3° 2θ over the entire range of 2θ. A Gaussian function was fitted to each of the peaks observed after subtraction of a linear background. For each fit the intensities from 21 detector wires were used, 10 on each side of the wire with the highest intensity. The mean scattering angle of each diffraction peak was calculated to a precision of ± 0.005°. For each reflection the lattice parameter was calculated. No systematic deviation was observed for the lattice parameters. Therefore, the lattice parameter at each temperature was determined simply by averaging over the lattice parameters calculated from the individual peaks. The standard deviations of the lattice parameters of both austenite and ferrite were typically 0.00005 nm, with upper and lower limits of 0.002 nm and 0.00001 nm.

3 Results and Discussion

3.1 Lattice Parameters of Fe-C Austenite

In Figure 2 the lattice parameter of austenite is shown as a function of temperature. At high temperatures a linear dependence of the lattice parameter on temperature is observed. Below the A₃ temperature the austenite (γ) decomposes into ferrite (α) and carbon-enriched austenite. The expansion of the austenite due to carbon enrichment more than compensates for the thermal shrinkage, resulting in a net increase of the lattice parameter with decreasing temperature. The heavy dashed line in the figure indicates the temperature dependence of the lattice parameter of the equilibrium austenite in the bi-phase region, obtained using the carbon concentrations and temperatures at the γ(α+γ) phase boundary in the equilibrium Fe-C phase diagram (8). The discrepancy between the heavy dashed line and the experimental data below the A₃ temperature suggests that in the present experiments the equilibrium carbon enrichment had not been reached yet. The time dependence of the carbon enrichment of austenite in the bi-phase region will be described elsewhere. In this analysis only data for temperatures higher than the A₃ temperature, i.e. data for austenite in the single phase region, are used.

For each alloy a linear fit of the lattice parameter of the austenite, a₀, against temperature according to equation [1] was performed.
\[ a_y(T) = a_{1000} \cdot (1 + \alpha \cdot (T - 1000)) \]  \[ (1) \]

with the temperature in Kelvin. Recognising a possible temperature dependence of the linear thermal expansion coefficient, \( \alpha \), a temperature of 1000 K is used as the reference temperature instead of room temperature. The results are shown in table 2.

<table>
<thead>
<tr>
<th>Name</th>
<th>( \text{at}% \text{ C} )</th>
<th>( \text{atoms C / 100 atoms Fe} )</th>
<th>Temperature range ( (K) )</th>
<th>( a_y(T) ) ( (\text{nm}) )</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fe</td>
<td>0.05</td>
<td>0.05</td>
<td>[1180, 1250]</td>
<td>0.365320 ( \cdot (1 + 24.72 \times 10^{-6} \cdot (T - 1000)) )</td>
</tr>
<tr>
<td>Fe-0.3 C</td>
<td>1.30</td>
<td>1.32</td>
<td>[1080, 1250]</td>
<td>0.365399 ( \cdot (1 + 24.53 \times 10^{-6} \cdot (T - 1000)) )</td>
</tr>
<tr>
<td>Fe-0.4 C</td>
<td>1.75</td>
<td>1.78</td>
<td>[1060, 1250]</td>
<td>0.364365 ( \cdot (1 + 24.3 \times 10^{-6} \cdot (T - 1000)) )</td>
</tr>
<tr>
<td>Fe-0.6 C</td>
<td>2.60</td>
<td>2.72</td>
<td>[1030, 1250]</td>
<td>0.365111 ( \cdot (1 + 23.23 \times 10^{-6} \cdot (T - 1000)) )</td>
</tr>
<tr>
<td>Fe-0.8 C</td>
<td>3.65</td>
<td>3.79</td>
<td>[1000, 1250]</td>
<td>0.365956 ( \cdot (1 + 22.93 \times 10^{-6} \cdot (T - 1000)) )</td>
</tr>
</tbody>
</table>

**TABLE 2.** The Lattice Parameter of the Fe-C Austenite. The Subscript indicates the Uncertainty in the Last Digit.

**FIG. 2.** The lattice parameter of austenite as a function of temperature. The solid lines indicate the fits according to eq. [2a]. The heavy dashed line indicates the lattice parameter of Fe-C austenite in the bi-phase region obtained using equation [2a] and the carbon concentrations and temperatures at the \( \gamma (\alpha + \gamma) \) phase boundary in the Fe-C phase diagram.

**FIG. 3.** Comparison of the lattice parameter of austenite of the pure Fe sample with literature data.

Literature data on the lattice parameter of pure Fe austenite as a function of temperature are shown in figure 3. Thermal expansion coefficients derived from the data in (1-4) are listed in table 3. Both the present values of the lattice parameter and of the thermal expansion coefficient of pure Fe are slightly higher than the values derived from the literature. The total impurity level of the present samples is comparable to that of the samples in (1-3). No detailed chemical analysis was provided in (4), only a total impurity level of 0.33 wt.% was reported. The observed differences in lattice parameters cannot be attributed to differences in impurity level.
TABLE 3

Lattice Parameters and Thermal Expansion Coefficients for Pure Fe Austenites as derived from (1-4). The Subscript indicates the Uncertainty in the Last Digit.

<table>
<thead>
<tr>
<th>Ref.</th>
<th>Temperature range (K)</th>
<th>α(T) (nm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>this work</td>
<td>(1150, 1250)</td>
<td>0.36320± (1 + 24.7) x 10^-6 (T-1000)</td>
</tr>
<tr>
<td>1</td>
<td>(1189, 1661)</td>
<td>0.3630± (1 + 23.6) x 10^-6 (T-1000)</td>
</tr>
<tr>
<td>2</td>
<td>(1185, 1528)</td>
<td>0.3632± (1 + 21.5) x 10^-6 (T-1000)</td>
</tr>
<tr>
<td>3</td>
<td>(1221, 1634)</td>
<td>0.36375± (1 + 20.3) x 10^-6 (T-1000)</td>
</tr>
<tr>
<td>4</td>
<td>(1193, 1343)</td>
<td>0.36341± (1 + 19.5) x 10^-6 (T-1000)</td>
</tr>
</tbody>
</table>

The lattice parameter of austenite is plotted in figure 4 as a function of the carbon concentration for three temperatures. In this figure the carbon concentration is represented as the number of C atoms per 100 atoms Fe, N_C. In the case of substitutional alloys a linear dependence of the lattice parameter on the atomic fraction of solute, n_S, is expected (i.e. Vegard's rule; cf. (9)). However, for interstitial alloys, such as Fe-C alloys, the lattice parameter would depend linearly on the number of solute atoms per solvent atom, since solvent atoms are not replaced at their sublattice by solute atoms. The expected linear dependence is indeed observed. The quality of the fit is comparable to that of a fit against the atom fraction carbon. The dependence of the lattice parameter of austenite on carbon concentration is slightly different from the relations given in (3,6), which were derived through a combination of extrapolations both in composition and temperature.

![Graph](image)

FIG. 4. The lattice parameter of austenite as a function of the carbon concentration.

![Graph](image)

FIG. 5. The thermal expansion coefficient of austenite as a function of the carbon concentration.

The dependence of the linear thermal expansion coefficient on carbon concentration is shown in figure 5. The observed slight dependence has not been reported previously from diffraction experiments. A dilatometric analysis of the thermal expansion coefficient of Fe-C alloys in the same composition range (10) showed a similar dependence on carbon concentration, although absolute values of the thermal expansion coefficient were lower than in the present work.
Finally, the observed dependencies of the lattice parameter of austenite on temperature and carbon concentration can be combined and expressed as (the subscript indicates the uncertainty in the last digit):

\[ a_\gamma (X_C^*, T) = (0.363068_6 + 0.000752 \cdot X_C^*) \cdot [1 + (24.92 - 0.61 \cdot X_C^*) \cdot 10^{-6} \cdot (T - 1000)] \]  

\[ \text{nm} \]  

[2a]

Or as:

\[ a_\gamma (X_C^*, T) = (0.363067 + 0.000783 \cdot X_C^*) \cdot [1 + (24.92 - 0.51 \cdot X_C^*) \cdot 10^{-6} \cdot (T - 1000)] \]  

\[ \text{nm} \]  

[2b]

3.2. Lattice Parameters of Ferrite

The measured lattice parameters of ferrite are presented in Figure 6 as a function of temperature. A negligible amount of carbon dissolves in the ferrite (8). Hence, the lattice parameter of ferrite should not depend on the carbon concentration of the alloy. There is very good agreement for the ferrite lattice parameter for the various alloys with the exception of the Fe-0.8 C alloy. The Fe-0.8 C alloy contains a relatively high amount of phosphorus, but this does not explain the difference observed, since a higher phosphorus concentration would reduce the lattice parameter of ferrite (11). Ignoring the results for the Fe-0.8 C alloy, the average lattice parameter of ferrite, \( a_\alpha \), in the temperature regime 800 - 1200 K is given by:

\[ a_\alpha (T) = 0.288634 \cdot [1 + 17.55 \cdot 10^{-6} \cdot (T - 800)] \]  

\[ \text{nm} \]  

[3]

In this case 800 K was used as the reference temperature. Equation [3] holds both above and below the \( A_1 \) temperature.

**FIG. 6.** The lattice parameter of ferrite as a function of temperature. The solid line indicates the fit according to eq. [3]

**FIG. 7.** Comparison of the lattice parameter of ferrite in the pure Fe sample with literature data
Finally, our data for the pure Fe ferrite are compared to data from the literature. In Figure 7 it can be seen that the lattice parameters measured in (4) are somewhat high. This is attributed to a higher impurity level. The lattice parameters determined in (3) agree with the present data at temperatures below 920 K but at higher temperatures they deviate rather strongly. There is excellent agreement between the lattice parameters measured in this work and those of (1) and (2).

4. Acknowledgements

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5. References

10. H. Esser, H. Easterbrock, Arch. Eisenh. 7, (1941), 341