

In situ synchrotron X-ray study of bainite transformation kinetics in a low carbon Si-containing steel

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Abstract

The bainite transformation in a low carbon Si-containing steel has been studied in situ by synchrotron X-rays. While the austenite is homogeneous prior to transformation, the carbon distribution becomes nonuniform as bainite plates form. This is because of the different degrees of physical isolation of films and blocks of residual austenite. The method for converting dilatational strain into bainite volume fraction, using lattice strain as a reference, during isothermal transformation was found to overestimate it. The bainitic and martensitic ferrite did not exhibit a tetragonal unit cell due to the low carbon content of the steel and the high transformation temperature.

Keywords: Synchrotron X-ray diffraction, Bainite transformation kinetics, Tetragonality, Carbon distribution

1. Introduction

Low-alloy high-strength steels which take advantage of retained austenite to improve ductility often use carbon as the austenite stabiliser, in conjunction with a cementite inhibitor,

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such as Si and Al. Apart from the classical TRIP-assisted steels, carbide-free bainitic steels of this kind have been studied widely, including in the context of rail steels [1–5] and nanostructured steels used in armour [6–8]. These steels utilise the bainite transformation to enrich the untransformed austenite with carbon, as carbon partitions to austenite from bainitic ferrite. The bainite transformation is frequently studied by following transformation strain using dilatometry, but accurate volume fractions of bainitic ferrite, martensite and retained austenite are difficult to obtain from such data. X-ray diffraction is a better method in this context, but the penetration depth of a normal laboratory X-ray is less than $9\ \mu\text{m}$ for iron samples over the typical scanning angle of $30 \leq 2\theta \leq 160$, using $\text{Cu K}\alpha$ radiation (calculated using PANalytical X’Pert HighScore Plus version 3.0). Moreover, normal X-ray diffraction is performed after heat treatment, it is almost impossible to distinguish bainite from martensite. Synchrotron X-rays can penetrate much deeper, and hence are not sensitive to surface conditions, and can provide information over a much larger volume of the specimen. For isothermal holding and subsequent cooling to ambient temperature, in situ X-ray diffraction during heat treatment can easily differentiate isothermal bainite from martensite formed during cooling as they form at different stages.

Recent studies show bainitic ferrite can be tetragonal [9–12], but all the steels studied were high in carbon; it could be interesting to check the symmetry of the bainitic ferrite lattice in low carbon steel as well.

The purpose of this work was to investigate in situ, the bainite transformation in a relatively low-carbon silicon-containing steel.

2. Experimental procedure

The experiments were carried out in Deutsches Elektronen-Synchrotron (DESY), Hamburg, Germany. The schematic equipment configuration is shown in Fig. 1. The chemical composition of the alloy used is shown in Table 1. Samples of diameter 3 mm and length 10 mm were machined from the mid-radius position of a hot rolled $\phi 32$ mm steel bar. They were then heated up to 1200 °C at a rate of 5 °C s⁻¹, held at the temperature for 5 min, then cooled at a rate of 10 °C s⁻¹ to the pre-set isothermal transformation temperatures, held for 5 min, followed by cooling to room temperature at a rate of 10 °C s⁻¹, Fig. 2. The samples were heat treated using a Bähr DIL 805 A/D dilatometer, temperature and length change were monitored during the whole process, with diffraction patterns recorded simultaneously at a time interval of 0.27 s by a Perkin Elmer XRD 1621 image plate, dimension of 410 mm × 410 mm, with a resolution of 200 μ m × 200 μ m and an array size of 2048 × 2048. The synchrotron X-ray energy used was 100 keV, spot size was 1 × 1 mm and sample to detector distance was 1280 mm, so the angular resolution is less than 0.01°.

Two dimensional X-ray diffraction patterns were converted to one dimensional spectra using FIT2D software version 17.054 [13], and the resulting one dimensional spectra were analysed using the Reitveld refinement method [14] with Materials Analysis Using Diffraction (MAUD) software version 2.7 [15]. Instrument broadening (i.e., Caglioti PV peak shape parameters) was calibrated using a Si standard, and these parameters were kept unchanged for subsequent analysis. The weighted profile R -factor (R_{wp}) was used as the indicator of the quality of fitting, and all fittings were checked graphically as well [16].

Figure 1: Equipment configuration for synchrotron X-ray diffraction experiment.

Figure 2: Illustration of heat treatment scheme.

Table 1: Chemical composition of the steel in wt%

C	Si	Mn	Ni	Mo	Cr	Cu
0.22	0.91	1.37	0.06	0.14	1.54	0.1

The carbon content of retained austenite was calculated using the measured lattice parameter and the Dyson and Holmes equation [17]. Assume paraequilibrium condition, because the lattice parameters are measured in situ during the whole process, the carbon content of residual austenite during isothermal bainitic transformation can be estimated using the following equation:

$$w_C = \bar{w}_C + \frac{a_\gamma - a_{\gamma_0}}{0.033} \quad (1)$$

where w_C and \bar{w}_C are the carbon content of the residual austenite and the average of the alloy in wt%, a_γ and a_{γ_0} are the lattice parameter of the carbon-enriched residual austenite and that before transformation starts in Å.

Assuming a body-centred cubic structure of bainitic ferrite, the carbon content is calculated

using equation [17–19]:

$$\begin{aligned}
 a_{\text{BCC}} = & 2.8664 \\
 & + \frac{(a_{\text{Fe}} - 0.279 x_{\text{C}})^2 (a_{\text{Fe}} + 2.496 x_{\text{C}}) - a_{\text{Fe}}^3}{3a_{\text{Fe}}^2} \\
 & - 0.03 x_{\text{Si}} + 0.06 x_{\text{Mn}} + 0.07 x_{\text{Ni}} \\
 & + 0.31 x_{\text{Mo}} + 0.05 x_{\text{Cr}} + 0.096 x_{\text{V}}
 \end{aligned} \tag{2}$$

where $a_{\text{Fe}} = 2.8664 \text{ \AA}$ is the lattice parameter of pure BCC iron at room temperature and the alloy element concentrations are in mole fraction.

For a body-centred tetragonal structure of ferrite, the relation between axial ratio c/a and carbon content is

$$\frac{c}{a} = 1 + 0.045 w_{\text{C}} \quad \text{wt\%} \tag{3}$$

where w_{C} is the concentration of carbon in wt% [20, 21].

3. Result and discussion

3.1. Volume fraction of bainitic ferrite

For the carbide-free bainite, where the phases present are just bainitic ferrite and residual austenite at the transformation temperature, a method exists to calculate the volume fraction of bainitic ferrite (V_{α_b}) from dilatational strain [18, 22, 23]. It requires a knowledge of the accurate thermal expansion coefficients of ferrite and austenite. A specimen was austenitised in the dilatometer at $1100 \text{ }^\circ\text{C}$ for 20 min, followed by cooling to $900 \text{ }^\circ\text{C}$ at a rate of $0.5 \text{ }^\circ\text{C s}^{-1}$,

and the thermal expansion coefficient for austenite was measured from a graph of strain versus temperature. For ferrite, the sample was heated to 650 °C at a rate of 0.5 °C s⁻¹, annealed at 650 °C for 1 h, then cooled to room temperature at a rate of 0.5 °C s⁻¹, strain versus temperature plot during cooling was used to measure the thermal expansion coefficient. The annealing allows any austenite which may be retained in the specimen to decompose and carbide to form, leaving a ferrite plus carbide microstructure. As the carbide volume fraction is small, its effect on the thermal expansion coefficient can be neglected. The measured thermal expansion coefficients for austenite and ferrite are $2.27 \times 10^{-5} \pm 1 \times 10^{-7} \text{ }^\circ\text{C}^{-1}$ and $1.54 \times 10^{-5} \pm 5 \times 10^{-7} \text{ }^\circ\text{C}^{-1}$, respectively.

The strains during isothermal holding for all transformation temperatures are shown in Fig. 3 a. The volume fraction of bainite, calculated from strain using MAP_STEEL_DILAT code [23], is shown in Fig. 3 b. The volume fractions of bainite for transformation at 370 °C and 400 °C reached almost 1, which is not reasonable as retained austenite was detected, and the code was unable to do the calculation for the later stages of transformation for these two cases. The problem with such calculation is that the volume fraction depends on the difference $(a_\gamma^3 - 2a_\alpha^3)$, a_γ and a_α are the lattice parameters of austenite and ferrite, so that small errors in the lattice parameters and thermal expansion coefficients are magnified. The method also assumes that carbon is uniformly distributed in the austenite whereas that is well known not to be the case [24, 25].

The volume fractions were also calculated using synchrotron X-ray diffraction, and MAUD program version 2.7 was used to do the Rietveld refinement [9, 12, 14, 15].

By comparing Fig. 3 b to Fig. 4, it may be concluded that X-ray diffraction gives a more

reasonable bainite volume fraction, while the inputs to MAP_STEEL_DILAT tend to over-estimate it.

Figure 3: (a) Strain as a function of time during isothermal soaking for different transformation temperatures. (b) Bainite volume fraction as a function of time during isothermal soaking for different transformation temperatures, calculated from strain using program MAP_STEEL_DILAT [23].

The final phase fractions of different phases after cooling to ambient temperature measured using synchrotron X-ray diffraction are shown in Table 2. The bainite fraction increases as the transformation temperature is reduced, while that of martensite decreases. The volume fraction of retained austenite after cooling to ambient temperature has a peak at 400 °C. All of these trends are as expected since a low fraction of bainite leads to less enrichment of austenite with carbon, and structural refinement of γ also plays a role in controlling its stability on cooling to ambient temperature [5, 26, 27].

Table 2: Phase fractions after cooling to ambient temperature, measured by synchrotron X-ray.

isothermal temperature	bainite	martensite	retained austenite
370 °C	0.88 ±0.01	0.02 ±0.01	0.10 ±0.01
400 °C	0.81 ±0.01	0.03 ±0.01	0.16 ±0.01
430 °C	0.75 ±0.01	0.11 ±0.01	0.14 ±0.01
460 °C	0.32 ±0.01	0.62 ±0.01	0.06 ±0.01
490 °C	0.09 ±0.01	0.85 ±0.01	0.06 ±0.01

Figure 4: Bainite volume fraction as a function of time during isothermal soaking for different transformation temperatures, calculated from X-ray diffraction patterns, measured at the transformation temperature.

Bainitic ferrite fraction from dilatational strain using in situ X-ray data

The number of iron atoms before transformation and during transformation must be the same, so that

$$\frac{4V_0}{a_{\gamma_0}^3} = \frac{2VV_{\alpha_b}}{a_{\alpha_b}^3} + \frac{4VV_{\gamma}}{a_{\gamma}^3} \quad (4)$$

where V_0 is the total volume before transformation starts, a_{γ_0} is the austenite lattice parameter before transformation starts, V is the total volume during transformation, V_{α_b} and V_γ are the volume fractions of bainitic ferrite and austenite during transformation, respectively, a_{α_b} and a_γ are the respective lattice parameters of bainitic ferrite and austenite during transformation. Hence,

$$\frac{V}{V_0} = \frac{\frac{1}{a_{\gamma_0}^3}}{\frac{V_{\alpha_b}}{2a_{\alpha_b}^3} + \frac{V_\gamma}{a_\gamma^3}} \quad (5)$$

The relative volume change is given by

$$\frac{\Delta V}{V_0} = \frac{V - V_0}{V_0} = \frac{V}{V_0} - 1 \quad (6)$$

The relative volume change can be related to relative length change (strain) by

$$\frac{\Delta V}{V_0} = 3 \frac{\Delta L}{L_0} \quad (7)$$

Combining the above equations the volume fraction of bainitic ferrite can be solved iteratively.

Fig. 5 shows V_{α_b} obtained using different methods, MAP_STEEL_DILAT and the modified method using the measured lattice parameter. They both give larger volume fractions than X-ray diffraction does, which means the measured strain is always larger than theoretical strain and the strain calculated from measured lattice parameters. This may be due to the thermal expansion of the dilatometer push rods (alumina), which add to the measured strain.

Figure 5: Comparison of bainite fractions measured using different methods for transformation at 400 °C.

3.2. Carbon redistribution in untransformed austenite

As the alloy contains a significant concentration of Si, the precipitation of cementite is retarded, so carbon partitioning enriches any untransformed austenite. Consequently, films of untransformed austenite are left between bainitic ferrite subunits. Fig.6 shows the typical microstructure of the steel transformed at 370 °C for 5 min, films (γ_f) and blocks of retained austenites or $\gamma + \alpha'$ are present.

The carbon content of the bulk untransformed austenite will be lower than that of austenite film as the diffusion distance is much larger; also carbon diffuse from bainitic ferrite to austenite through their interface, larger interface area per unit volume of austenite should result in larger carbon enrichment, and the film austenite has a larger specific interface area than the blocky austenite does. It is expected therefore that carbon will not be homogeneously distributed in the austenite [5, 8, 24, 25, 28–30].

Figure 6: Typical microstructure of the steel transformed at 370 °C for 5 min.

The austenite $\{200\}_\gamma$ peak was chosen for the analysis as the $\{111\}_\gamma$ overlaps with the ferrite $\{110\}_\alpha$ peak. An example of the evolution of $\{200\}_\gamma$ peak for bainite transformation at 400 °C is shown in Fig.7 a. As transformation progresses, the austenite peak intensity decreases, and the peak shifts to lower angles, which means the lattice parameter of austenite increases. Asymmetrical broadening was also found to accompany the transformation process, attributed to the non-uniform distribution of carbon in austenite. The martensitic

transformation following the isothermal holding at 490 °C, Fig 7 b, shows negligible asymmetrical peak broadening, consistent with the work of Gong et al. [31], and the shifting of the peak position to higher angles is due to the thermal contraction upon cooling to ambient temperature.

In the early stages of bainitic transformation, the peak can be fitted assuming a homogeneous austenite as shown in Fig. 8 a, but as the transformation progresses, the austenite $\{200\}_\gamma$ peak can be deconvolved into two austenite peaks with different lattice parameters as shown in Fig. 8 b - d, which has been attributed to film and blocky austenite which have different carbon contents [24, 31, 32]. The deconvolution was carried out by adding two austenite phases with different lattice parameters which were allowed to change, after Rivteld refining, if these two lattice parameters became identical, then the peak could not be deconvoluted.

Figure 9 shows the evolution of measured volumetric ratio ($\frac{V_{\gamma_{\text{HC}}}}{V_{\gamma_{\text{LC}}}}$) of high carbon austenite over lower carbon austenite along with the sum of volume fractions of bainite and martensite ($V_{\alpha_b} + V_{\alpha'}$) as a function of time during transformation at 400 °C and 430 °C and subsequent cooling to ambient temperature. The curves of $\frac{V_{\gamma_{\text{HC}}}}{V_{\gamma_{\text{LC}}}}$ follow that of $V_{\alpha_b} + V_{\alpha'}$ very well. In the very early stage of transformation, when only one austenite is needed, the ratio is defined as 0. It was found that when the bainite volume fraction is smaller than about 0.4, the $\{200\}_\gamma$ peak cannot be deconvolved, Fig. 8 a.

As the bainite volume fraction increases, so does the amount of film austenite between bainitic ferrite platelets. When V_{α_b} reaches about 0.4, the homogenisation of carbon by diffusion in γ then becomes difficult, so two populations of carbon-enriched austenite are

detected, Fig. 10. This ratio stabilised at the later stages as the transformation approaches the maximum V_{α_b} given by the T_0 line. It should be noted that the ratio increased rapidly upon cooling to ambient temperature after 300 s of isothermal holding, which is because of martensitic transformation during cooling, as the low carbon blocky austenite transforms to martensite, while the high carbon film austenite is more stable. The observed development of high carbon austenite after a certain amount of ferrite formation is in agreement with Stone et al. [24].

When fitting the asymmetrical austenite peak, two austenite phases with different lattice parameters are needed, and these two lattice parameters should be allowed to change in order to fit the peak, because the carbon content of γ changes as the transformation progresses.

Figure 7: Evolution of austenite {200} peak. (a) Bainitic transformation at 400 °C. (b) Martensitic transformation during cooling after isothermal holding at 490 °C.

Figure 8: {200} $_{\gamma}$ peak with fitted curve for transformed at different stage. (a)-(d) 400 °C. (e)-(h) 490 °C.

Figure 9: Volume fraction of ferritic phase and the volumetric ratio of high carbon austenite over low carbon austenite evolution over time. (a) Transformation at 400 °C and subsequent cooling to room temperature. (b) Transformation at 430 °C and subsequent cooling to room temperature. ($V_{\gamma_{\text{HC}}}$ and $V_{\gamma_{\text{LC}}}$ are the volume fractions of austenite with high carbon content and low carbon content with respect to the whole untransformed austenite).

Figure 10: Schematic illustration of carbon redistribution in austenite during bainite transformation.

Fig. 11 shows the austenite lattice parameter as a function of time for transformation at 400 °C, Fig. 11 a is the case when one austenite phase is used in the Rietveld refinement

analysis of the whole spectrum, a_γ increases continuously, Fig. 11 b shows the lattice parameters of high carbon austenite ($a_{\gamma_{\text{HC}}}$) and low carbon austenite ($a_{\gamma_{\text{LC}}}$) obtained from fitting of $\{200\}_\gamma$ peak. Initially, the austenite peak can be fitted to a homogeneous austenite, so there is no $a_{\gamma_{\text{HC}}}$ data, then it decreases, and stabilises after some time, while $a_{\gamma_{\text{LC}}}$ increases slightly with time. The corresponding carbon contents of austenite according to the measured lattice parameters are shown in Fig. 12. When assuming carbon is homogeneously distributed, its content increase from 0.22 wt% to 0.66 wt%; if two austenites were used, the carbon content of γ_{HC} decreases from 1.30 wt% to 1.14 wt%, and for γ_{LC} it increases from 0.22 wt% to 0.34 wt%.

Figure 11: Lattice parameter of austenite as a function of isothermal transformation time at 400 °C. (a) Homogeneous austenite. (b) Two populations of austenite with different carbon contents.

Figure 12: Carbon content of austenite as a function of isothermal transformation time at 400 °C. (a) Homogeneous austenite. (b) Two populations of austenite with different carbon contents.

Figure 13: Evolution of carbon content in austenite during isothermal bainite transformation at different temperatures.

Fig. 13 shows the measured carbon contents as a function of time for all transformation temperatures. Carbon enrichment during bainite transformation is clearly observed for all temperatures. The anomaly of the carbon content dipped below the alloy's average content for transformation at 370 °C is due to fitting errors in the lattice parameter, which cannot be explained. The time for the curves to reach plateaus increases as the transformation temperature decreases, which is because of decreased carbon diffusivity and more bainite forms at lower temperature which reduces the effective diffusion path. The carbon parti-

tioning is slower than bainite transformation, especially at low transformation temperatures, Fig. 14. After the samples were cooled to ambient temperature, the carbon contents of retained austenite are shown in Fig. 15 along with the calculated T_0 and T'_0 lines. After the formation of martensite, the retained austenite has much higher carbon content, due to the preferred transformation of low carbon austenite, the carbon content in the retained austenite is close to the T'_0 lines.

Figure 14: Carbon contents of residual austenite and V_{α_b} as a function of isothermal transformation time at different temperatures, (a) 370 °C, (b) 400 °C, (c) 430 °C, (d) 460 °C, (e) 490 °C.

Figure 15: Carbon content in retained austenite after cooling to ambient temperature with T_0 and T'_0 lines superimposed; T_0 and T'_0 are calculated using MatCalc [33].

3.3. Tetragonality

The Rietveld refinement method [14] was used to check for any tetragonality in the unit cells of bainite and martensite during transformation. The polynomial background function, total incident X-ray intensity, ferrite phase fraction (the residue being austenite), microstrain, crystallite size, texture parameter (Harmonic model [14]) and applicable lattice parameters were permitted to refine. The weighted profile factor R_{wp} is the most relevant indication of the quality of fitting, a smaller value means better fitting. Two sets of analysis were carried out with body-centred cubic lattice ($\text{Im}\bar{3}\text{m}$, BCC) and body-centred tetragonal lattice ($\text{I}\frac{4}{\text{m}}$ mm, BCT) for ferrite.

By comparing the R_{wp} of fittings with BCC and BCT structure, tetragonality can be decided as adopted by Hulme-Smith and Bhadeshia [9, 12]. Fig. 16a shows the R_{wp} as a function of V_{α_b} during isothermal bainite transformation at 400 °C. It is clear that the BCC

structure gives better fit, hence the bainite at this temperature for this composition is body centred cubic. The corresponding plot for transformation during cooling after isothermal holding at 490 °C for 5 min is shown in Fig. 16 b. Note that transformation at this temperature gives $V_{\alpha_b}=0.09$, and martensite forms during cooling. The result still suggests the BCC structure gives better fit. The reason for cubic martensite might be the high transformation temperature ($M_s = 360\text{ °C}$), and the low overall carbon content of 0.22 wt%, which can be trapped in dislocations or other defects [5].

Figure 16: R_{wp} evolution as a function of ferritic phase fraction. (a) 400 °C. (a) 490 °C.

BCT structure originates from the ordering of carbon along the c lattice axis. Zener has proposed a simple equation to calculate the temperature below which ordering will occur [34].

$$T_Z = 1330 w_C \quad \text{wt\%} \quad (8)$$

where T_Z is the Zener ordering temperature in K, w_C is the carbon content in wt%.

Fig. 17 shows the measured carbon content of the mixture of bainite and martensite as a function of the volume fraction of them ($V_{\alpha_b} + V_{\alpha'}$), with $V_{\alpha_b} = 0.09$ after isothermal transformation. Fig. 17 a is calculated assuming BCC structure, while Fig. 17 b is calculated from the c/a ratio assuming BCT structure. The carbon content of the mixture increases with the increase of its volume fraction. BCC structure gives higher carbon content, the highest carbon content is 0.20 wt%, while BCT gives a maximum value of 0.16 wt%.

The calculated maximum carbon content in bainitic ferrite is 0.023 wt% when in equilibrium with γ at 400 °C, and 0.012 wt% for 370 °C, and no solution was found for other

transformation temperatures. It is 0.20 wt% for the martensite transformation during cooling after isothermal holding at 490 °C. Using these values, the highest Zener ordering temperature is less than 10 °C, which is much lower than the M_S temperature, hence any ordering and tetragonality caused by the Bain strain could not survive [34], so it is not surprising that the bainite and martensite in this steel are body-centred cubic.

Figure 17: Carbon content of the mixture of bainite and martensite ($V_{\alpha_b} = 0.09$) for transformation at 490 °C and subsequent cooling to ambient temperature. (a) Calculated from lattice parameter assuming BCC structure. (b) Calculated from c/a ratio assuming BCT crystal structure.

4. Conclusions

The bainite transformation in a low carbon Si-containing steel was studied by synchrotron high energy X-rays. As observed in previous work, the distribution of carbon in the residual austenite becomes heterogeneous as the transformation progresses. [Trapped regions \(films\) of \$\gamma\$ acquire greater enrichment where as larger regions \(blocks\) are less enriched.](#) The latter therefore transform preferentially into martensite upon cooling to ambient temperature. As a consequence, the ratio of high carbon austenite to low carbon austenite increases dramatically following cooling to room temperature after isothermal bainite transformation.

The method for converting dilatational strain into bainite volume fraction [18, 22], using lattice strain as a reference, during isothermal transformation was found to overestimate it.

Carbon partitioning was found to lag behind the bainite transformation as expected; more time is needed as the transformation temperature is reduced.

Tetragonality was not observed in either the bainitic ferrite or α' , because the carbon content of the alloy is relatively low, and the Zener ordering temperature of the alloy is

below the bainite and martensite transformation temperature.

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