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## Tetragonality of bcc Phases in a Transformation-Induced Plasticity Steel

Zhiping Xiong

*University of Wollongong, zx868@uowmail.edu.au*

David R. G Mitchell

*University of Wollongong, dmitchel@uow.edu.au*

Ahmed A. Saleh

*University of Wollongong, asaleh@uow.edu.au*

Elena V. Pereloma

*University of Wollongong, elenap@uow.edu.au*

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## Tetragonality of bcc Phases in a Transformation-Induced Plasticity Steel

### Abstract

In a low-alloyed multi-phase transformation-induced plasticity steel, solute carbon content in polygonal ferrite, bainitic ferrite, and martensite was characterized using site-specific atom probe tomography. Selected area diffraction patterns were obtained using transmission electron microscopy, and the geometric distortion thereof was determined. The results showed that the lattice distortion increased in a sequence of polygonal ferrite, lath-like bainitic ferrite, and martensite. This increasing distortion corresponded to an increase in carbon content of the phase.

### Disciplines

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# 1 **Tetragonality of bcc phases in a transformation-induced** 2 **plasticity steel**

3  
4 ZHIPING XIONG<sup>1</sup>, DAVID R.G. MITCHELL<sup>2</sup>, AHMED A. SALEH<sup>1</sup>, and ELENA V.  
5 PERELOMA<sup>1, 2, \*</sup>  
6

7 <sup>1</sup>School of Mechanical, Materials, Mechatronic and Biomedical Engineering,  
8 University of Wollongong, Wollongong, NSW, 2522, Australia

9 <sup>2</sup>Electron Microscopy Centre, University of Wollongong, Wollongong, NSW, 2519,  
10 Australia

11 \*Correspondence author email address: elenap@uow.edu.au.  
12

13 **Abstract:** In a low-alloyed multi-phase transformation-induced plasticity steel, solute  
14 carbon content in polygonal ferrite, bainitic ferrite and martensite was characterized  
15 using site-specific atom probe tomography. Selected area diffraction patterns were  
16 obtained using transmission electron microscopy (TEM), and the geometric distortion  
17 thereof was determined. The results showed that the lattice distortion increased in a  
18 sequence of polygonal ferrite, lath-like bainitic ferrite and martensite. This increasing  
19 distortion corresponded to an increase in carbon content of the phase.  
20  
21

22 In steels, during cooling from the austenitization temperature, polygonal ferrite,  
23 bainite and martensite can form sequentially. It is well known that polygonal ferrite  
24 forms via a diffusional mechanism at a slow cooling rate from the austenitization  
25 temperature, according to the equilibrium Fe-C phase diagram, leading to a  
26 body-centered cubic (bcc) structure [1]. Under rapid cooling conditions from the  
27 austenitization temperature, martensite is formed. This occurs via a displacive  
28 mechanism, in which all atoms move some fractions of an interatomic distance,  
29 resulting in a body-centered tetragonal structure in medium-high carbon steels [2]. At  
30 intermediate cooling rates, bainite forms in a temperature range between those at  
31 which polygonal ferrite and martensite form. This has led to a decades-old debate as  
32 to whether or not the bainite formation mechanism is diffusion-controlled [3] or  
33 displacive-controlled [4].  
34

35 Recently, the carbon content in defect-free lath bainitic ferrite (BF) has been  
36 measured using atom probe tomography (APT) and has been found to be higher than  
37 para-equilibrium [5-9]. This demonstrates the assumption that this phase contains  
38 excess carbon due to trapping of carbon at defects is not correct [5]. Tetragonality  
39 (distortion) of the bcc lattice occurs commonly in high carbon martensite where an  
40 ordered structure of solute carbon atoms results from the shear mechanism of phase  
41 transformation manifesting in carbon super-saturation compared to the equilibrium  
42 state of ferrite [10-14]. It was also suggested to be the reason for the observed carbon

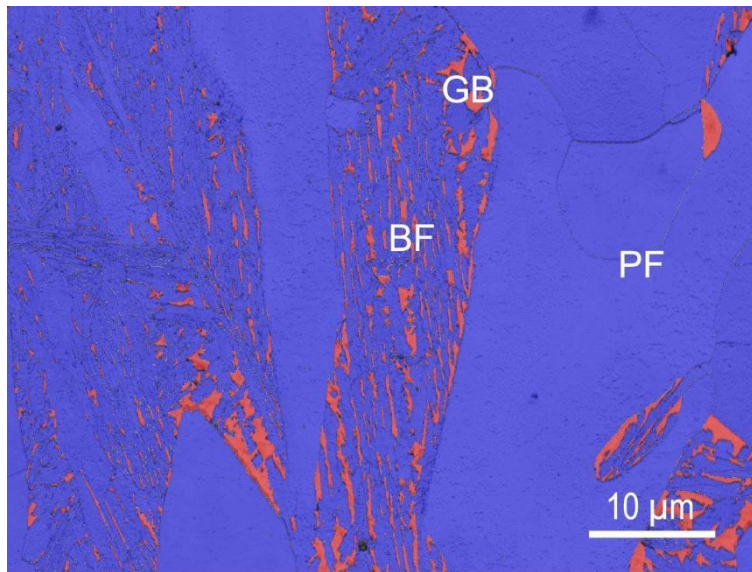
43 super-saturation in BF laths [15]. In-situ synchrotron, neutron and X-ray diffraction  
 44 (XRD) obtained from a range of different steels after suitable ageing (as listed in  
 45 Table 1) showed that the resulting BF laths had tetragonality. In addition, a decrease  
 46 in tetragonality of BF laths occurred with increasing holding time of bainitic steels as  
 47 a result of the carbon diffusing from the BF lath to the neighboring austenite [6, 17].

48  
 49 **Table 1. The heat treatment for different steels.**

Chemical compositions, wt. %	Heat treatment for bainitic transformation	Reference
Fe-0.84C-2.26Mn-1.78Si-1.55Co-1.47Cr-0.25Mo-0.11V-0.11Cu	200 °C for 10 days	[16]
Fe-0.7C-1.4Si-1.3Mn-1.0Cr-0.2Mo-0.1Ni	220 °C for 1 day	[6]
Fe-0.8C-1.6Si-2.0Mn-1.0Cr-0.2Mo-4.0Co-1.7Al	250 °C for 0.5 day	[6]
Fe-1.04C-3.89Si-1.97Mn-0.24Mo-1.43Al	300 °C for 1 day	[17]

50  
 51 The tetragonality of BF laths in bainitic steels has received significant attention [6, 8,  
 52 16, 17]. However, limited data are available for bcc phases in multi-phase steels. In  
 53 addition, it is very important that advanced technologies should be employed in  
 54 tetragonality characterization as new scientific information could be found because of  
 55 improved accuracy. The present paper studies the tetragonality in bcc phases  
 56 (polygonal ferrite, BF laths and martensite) in a low-alloyed multi-phase  
 57 transformation-induced plasticity (TRIP) steel using high-resolution transmission  
 58 electron microscopy (TEM). The relationship between tetragonality and solute carbon  
 59 content fits very well to the recently published data using high-resolution XRD.

60  
 61  
 62 A low-alloyed multi-phase TRIP steel (Fe-0.17C-1.61Mn-1.52Si-0.20Cr, wt. %) was  
 63 produced using a laboratory simulated strip casting process, which included an  
 64 isothermal holding at 400 °C for 900 s [18]. The microstructure consisted of 55%  
 65 polygonal ferrite, 4.5 % retained austenite (RA), carbide-free bainite (BF, granular  
 66 bainite (GB)), and small amounts of martensite and Widmanstätten ferrite (WF) as  
 67 shown in a representative electron backscattering diffraction (EBSD) map in Figure 1.  
 68 Examples of martensite and WF were reported in previous studies [7, 18].



69

70 Fig.1—A representative EBSD phase map showing BCC phases in blue and FCC austenite in red.

71 *BF* is bainitic ferrite, *GB* is granular bainite and *PF* is polygonal ferrite.

72

73 Site-specific tips for APT were micro-machined based on EBSD maps. APT  
 74 experiments were carried out using a local electrode atom probe (LEAP 4000 HR,  
 75 Cameca Instruments). The details have been published in Ref. [7]. It is emphasized  
 76 here that the chemical compositions of the BCC phases were calculated from the  
 77 volumes containing no clusters, precipitates or solute segregation.

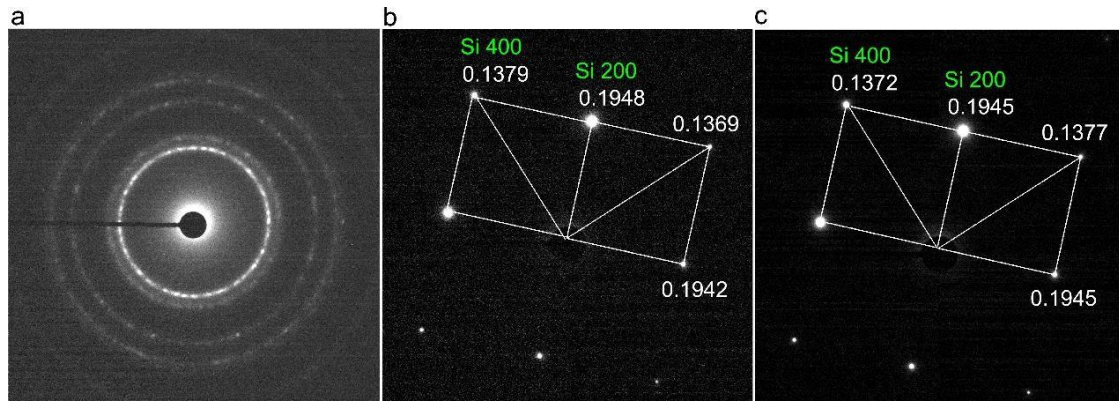
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79 Foils for TEM characterization were prepared using focused ion beam (FIB) milled  
 80 (Helios NanoLab G3 CX dual-beam, FEI) lift-outs with the help of EBSD phase maps  
 81 to identify suitable lift-out locations. Foils were characterized using a JEOL ARM  
 82 200F instrument operated at 200 kV. Selected area diffraction (SAD) patterns were  
 83 acquired at a camera length of 30 cm. Camera lengths had been calibrated previously  
 84 using a polycrystalline gold reference specimen. Electron diffraction patterns are  
 85 subject to an intrinsic distortion, as a result of lens distortions, mechanical  
 86 misalignments and aberrations. A typical manufacturer's installation is that this  
 87 distortion should be less than 1%. The correction of this distortion (to reveal the true  
 88 extent of any tetragonality) was carried out as described below.

89

90 A polycrystalline gold on carbon specimen provided ring diffraction patterns (Figure  
 91 2(a)). Ellipse fitting analysis (EFA) was used to measure the distortion [19]. This  
 92 showed an elliptical distortion of  $0.91 \pm 0.26\%$  (distortion is defined as  $(1 - (\text{minor axis}/\text{major axis})) \times 100\%$ ) at an angle of 160 degrees with respect to the horizontal, ie.  
 93 a vector pointing to approximately 10 o'clock would be parallel to the major axis of  
 94 the ellipse. The elliptical distortion was corrected by warping the pattern, leading to a  
 95 decrease in elliptical distortion to  $0.26 \pm 0.03\%$ . For verification, single crystal patterns  
 96 of silicon with the beam parallel to the [001] direction (Figure 2(b)) were obtained  
 97 under identical conditions to those used for polycrystalline gold. A Si  $\langle 400 \rangle$  vector  
 98

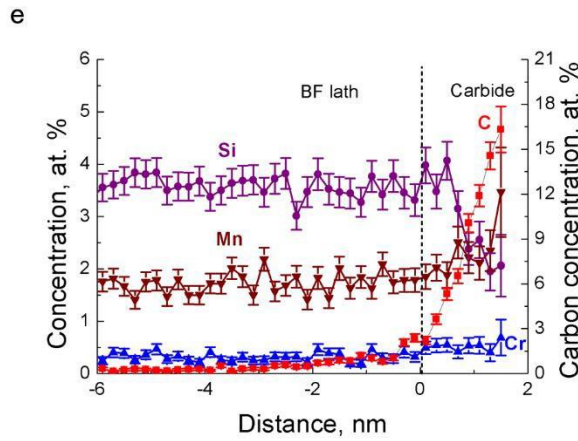
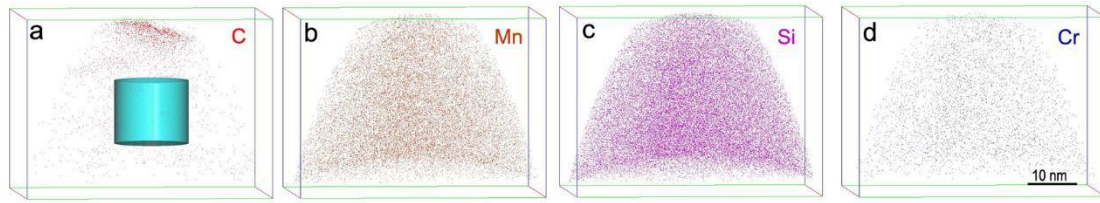
99 lay close to the major axis of distortion. The measured distortion in the as-recorded  
 100 pattern was obtained by comparing the length of this Si <400> vector with its  
 101 orthogonal counterpart. This gave a value of 0.73%. The Si [001] pattern (Figure 2(b))  
 102 was warped using the distortion parameters obtained from the gold pattern (Figure  
 103 2(a)), and the overall distortion was reduced to 0.36% (Figure 2(c)). Thus, this was  
 104 generally consistent with that measured in polycrystalline gold, which demonstrated  
 105 the reliability of this correction method.



106  
 107 Fig. 2—(a) Selected area diffraction pattern from polycrystalline gold; (b) uncorrected and (c)  
 108 corrected selected area diffraction patterns from single crystal of silicon with the beam parallel to  
 109 [001]. The values are measured lattice spacing in Å.

110  
 111

112 As an example, Figure 3(a) shows a carbon atom map from a volume which included  
 113 enriched (carbide) and depleted (BF lath) regions, while Figure 3(b-d) show  
 114 homogeneous distribution of manganese, silicon and chromium. Chemical gradients  
 115 (Figure 3(e)) were revealed using proximity histograms at iso-concentration surfaces  
 116 of 3.0 at. % C, namely the proximate interface between BF lath and carbide. As  
 117 expected, carbon and manganese were enriched while silicon was depleted in the  
 118 carbide. The chemical composition of this BF lath was calculated in the homogeneous  
 119 region (cylinder in Figure 3(a)) without any visible segregation and clustering. Similar  
 120 measurements were undertaken to determine the compositions of polygonal ferrite,  
 121 BF lath and martensite from numerous locations. Here, the statistic carbon content for  
 122 each phase is listed in Table 2, which shows an increased sequence of carbon content  
 123 from PF to BF lath to martensite.



124

125 Fig. 3—Atom maps of (a) carbon (the blue cylinder is a volume which shows no segregation or  
 126 clustering and from which the composition was determined), (b) manganese, (c) silicon and (d)  
 127 chromium; (e) proximity histogram across the interface between a BF lath and carbide at the  
 128 iso-concentration surface of 3.0 at. % C (the left y-axis corresponds to Si, Mn and Cr).

129

130

**Table 2. Summary of carbon content and corrected lattice distortion.**

	Carbon content, at. % (wt. %)*	Corrected distortion, %**
Polygonal ferrite	0.027±0.002 (0.0063±0.0005)	0.08±0.07
Bainitic ferrite lath	0.32±0.16 (0.075±0.038)	0.83±0.14
Martensite	2.7±0.5 (0.61±0.12)	1.16

131

\* determined from APT data; \*\* determined from SAD patterns.

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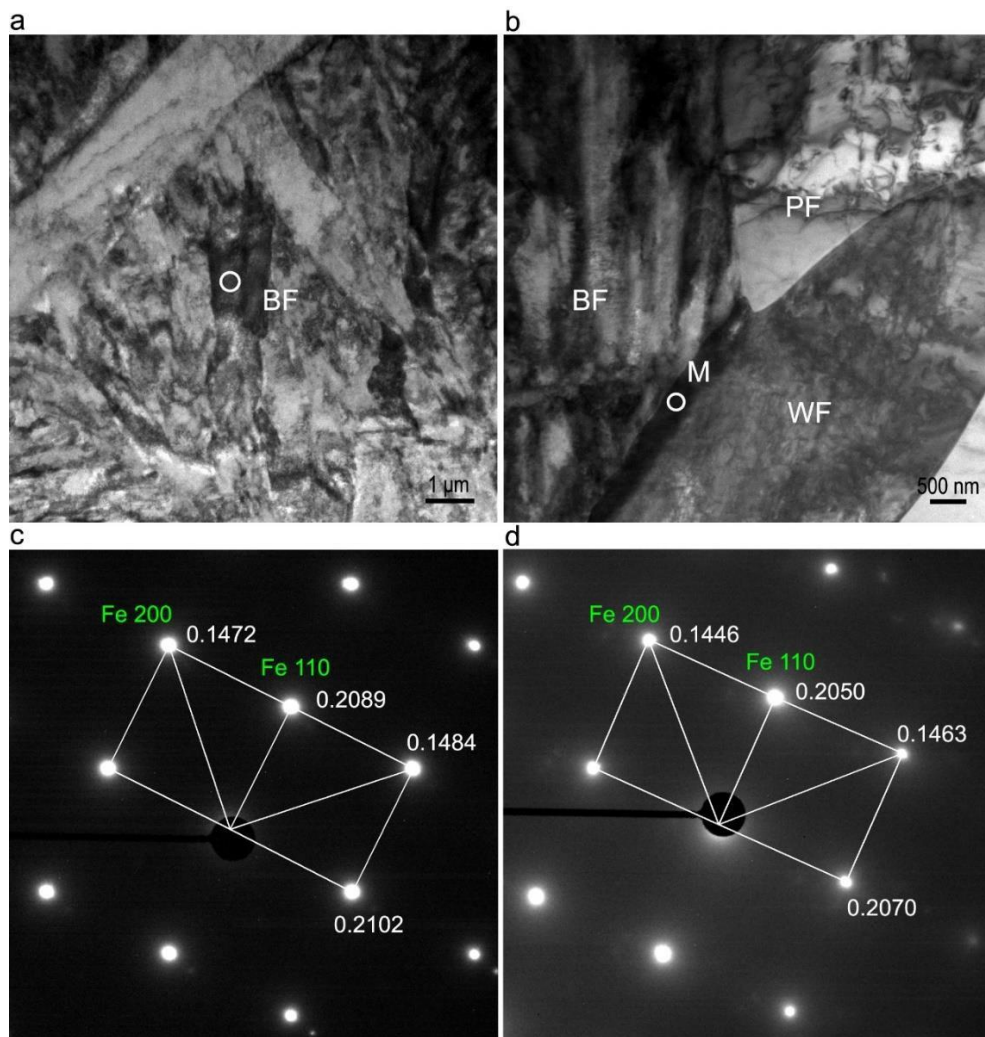
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Figure 4(a) and (c) show an example of a BF lath and its distortion-corrected SAD pattern, respectively; while Figure 4(b) shows martensite between WF and a bainitic packet, and its corrected SAD pattern was shown in Figure 4(d). The measured lattice distortion (according to *section 2.2*) is summarized in Table II. The polygonal ferrite had the lowest distortion of 0.08±0.07 %, which is effectively zero. After distortion correction of the calibration polycrystalline gold pattern, its residual distortion was 0.26±0.03%. Thus, a distortion of about 0.3% is probably the real limit of precision for this technique. The BF lath showed statistically significant lattice distortion (0.83±0.14 %) while the martensite showed the greatest degree of distortion (1.16 %). Garcia-Mateo *et al.* [6] used high-resolution TEM to measure lattice spacings in a Fe-1.0C-1.5Si-1.9Mn-1.3Cr-0.26Mo-0.1V (wt. %) bainitic steel isothermal held at 200 °C for 6 days. They found that along [010] and [001] directions the spacings were 0.286 and 0.289 nm, respectively, corresponding to a 1.05 % distortion. This is

146

147 slightly higher than 0.83 % measured in the present material, although their BF lath  
 148 had a higher carbon content (up to ~ 0.55 at. %) compared with  $0.32 \pm 0.16$  at. % in the  
 149 present work. A significant difference in the distortion between the BF lath (0.83 %)  
 150 and martensite (1.16 %) was found here. Although dislocations may influence this  
 151 distortion, measurements of dislocation densities of BF lath ( $10^{14} \sim 10^{15} \text{ m}^{-2}$ ) [7, 20]  
 152 and martensite ( $10^{14} \sim 10^{15} \text{ m}^{-2}$ ) [21] in similar steels to those studied here were very  
 153 similar. In addition, the substitutional alloying elements were comparable between BF  
 154 lath and martensite (such as c.f.  $1.85 \pm 0.11$  vs  $1.86 \pm 0.14$  at. % Mn) due to their quick  
 155 formations at low temperatures. These suggest that the observed difference in lattice  
 156 distortion is a structural effect.



157

158 Fig. 4—Bright field images of (a) BF lath and (b) martensite with corresponding  
 159 distortion-corrected SAD patterns (c) and (d) from the regions circled in (a) and (b) respectively.  
 160 The zone axis of (c, d) is  $[011]\alpha$ . The numbers in (c, d) are lattice spacings in Å. *BF* is bainitic  
 161 ferrite, *PF* is polygonal ferrite, *WF* is Widmanstätten ferrite and *M* is martensite.

162

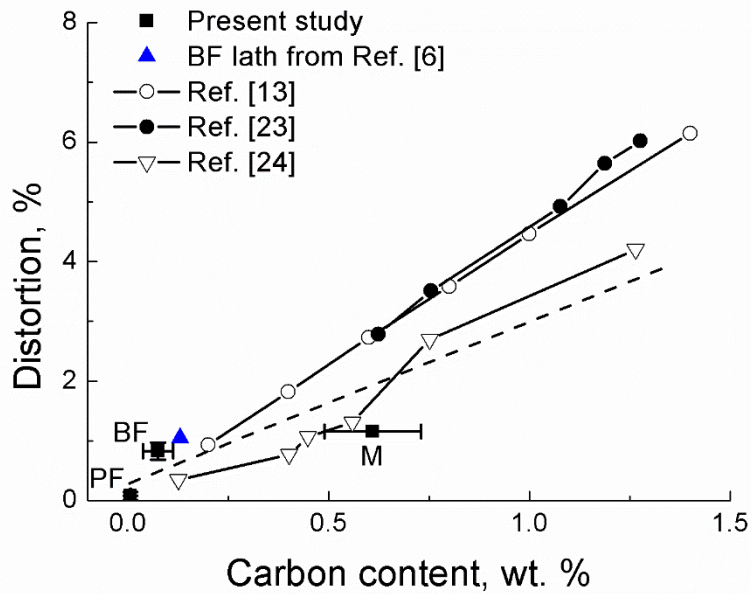
163

164 Polygonal ferrite forms in a diffusional process, and has a very low carbon solubility



165 (0.027±0.002 at. %). The measured lattice distortion of this BCC lattice (0.08±0.07%)  
166 was effectively zero. Both the BF lath and martensite formed in a low temperature  
167 regime, resulting in a high carbon content of 0.32±0.16 and 2.7±0.5 at. % (Table II),  
168 respectively. The large distortion (tetragonality) of these two phases (BF lath =  
169 0.83±0.14 % and martensite = 1.16 %) determined from SAD patterns arises from the  
170 high carbon concentrations – the degree of tetragonality increasing with carbon  
171 concentration [5, 17]. Due to low temperature transformation, the Bain strain [22]  
172 transfers carbon atoms in one octahedral interstice per iron atom in the face-centered  
173 cubic lattice to interstices in the BCC lattice of ferrite. Carbon would preferentially  
174 occupy just one of the three sub-lattices of octahedral sites rather than tetrahedral sites  
175 in the BCC lattice due to smaller solution energy in the former [15]. This phenomenon  
176 causes cube edge distortion and in turn tetragonality, due to the octahedral site being  
177 limited to accommodating only those atoms which are smaller than carbon. XRD  
178 analysis also demonstrated the tetragonality in BF lath and martensite, and that the  
179 degree of tetragonality varies with carbon content in the BF lath [6, 16, 18]. The linear  
180 relationship between the c/a ratio and carbon content has been established based on  
181 XRD data for martensite [13, 23, 24].

182  
183 Figure 5 shows the evolution of distortion with carbon content. The data for Fe-C  
184 martensite retrieved from Refs. [13, 23, 24] was also included, where distortion was  
185 calculated as (c-a)/a. The data from Refs. [13, 23] collected in 1950-1970s shows a  
186 larger distortion than the data from Ref. [24] published in 2017. This difference is  
187 probably ascribed to the improvement of X-ray diffractometer instrumentation and  
188 utilization of Rietveld refinement with related software. It means that advanced  
189 technologies are important in tetragonality characterization because they can provide  
190 excellent accuracy and in turn maybe offer new information and further  
191 reconsideration on this old topic. As shown by dashed line, the measured distortion in  
192 the present study has a reasonable fit with the newest data in Ref. [24]. The  
193 fluctuation along this dashed line is probably due to the experimental error. In  
194 addition, the variation between the data in the literature and the current work could be  
195 related to the difference in investigated materials compositions and processing.  
196 Alloying elements interactions with carbon could have occurred after completion of  
197 BF formation during subsequent holding at 400°C. Different alloying elements have  
198 different effects on lattice spacing. It has been reported that carbide-forming elements  
199 remove carbon atoms from their ordered positions and reduce the martensite lattice  
200 tetragonality, while non-carbide forming substitutional elements increase it due to  
201 their effect on carbon activity in iron [25].  
202



203

204 Fig. 5—The evolution of distortion in SAD pattern with carbon content. Solid lines are deduced  
 205 from the XRD data for martensite [13, 23, 24]. *PF* is polygonal ferrite, *BF* is bainitic ferrite lath  
 206 and *M* is martensite.

207

208

209 To summarise, site-specific APT and TEM/electron diffraction investigations on a  
 210 low-alloyed multiphase TRIP steel directly showed an increase in carbon content  
 211 (measured from a defect-free volume) and indirectly demonstrated an increase in  
 212 tetragonality, in a sequence of polygonal ferrite, BF lath and martensite. Similar to  
 213 martensite, the excess of carbon in the BF results in lattice tetragonality. Although  
 214 these data showed somewhat deviation from the linear relationship between  
 215 tetragonality and carbon content determined for martensite in 1950-1970s, they fitted  
 216 well to the data recently obtained using high-resolution XRD, indicating the  
 217 importance of advanced technologies for future research and reconsideration in  
 218 tetragonality.

219

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224

225 **References:**

226

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