Complementary use of transmission electron microscopy and atom probe tomography for the examination of plastic accommodation in nanocrystalline bainitic steels

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A displacive transformation involves the motion of a glissile interface. As in work-hardening, its motion can be halted by defects, such as dislocations, stacking faults or twins in the austenite. The defects are created when the shape deformation accompanying bainite growth is accommodated by plastic relaxation of the surrounding austenite. The growing plate stops when it collides with the austenite grain boundary. Because transformation from strong austenite leads to fine plates, alloys can be designed such that the bainite transformation is suppressed to low temperatures (125-350ºC) leading to a nano-scale bainitic microstructure. Complementary high resolution transmission electron microscopy and atom probe tomography have provided new experimental evidence on the accommodation of transformation strain, subjects critically relevant to understand the atomic mechanism controlling bainitic ferrite growth.

Introduction

A new generation of steels known as NANOBAIN is now under developed which on transformation at low temperature (125-350ºC) lead to a nano-scale microstructure [1]. It consists of slender crystals of ferrite (α), whose scale is similar to that of carbon nanotubes, and retained austenite (γ). Plastic relaxation in the austenite adjacent to the ferrite may control the final size of the bainitic ferrite plates [2]. The defects generated in this process resist the advance of the bainitic ferrite-austenite interface, the defects density being highest for lower transformation temperature [3]. The plastic relaxation of the shape change was examined a long time ago using in-situ hot-stage transmission electron microscopy (TEM) and pre-polished samples of austenite transforming to bainite. Observations revealed that the growth of bainite is accompanied by the formation of dislocations in and around the bainite [4]. Direct observations of the austenite/ferrite interface provided qualitatively that the bainitic ferrite contained a higher dislocation density than that of the retained austenite [5]. Dislocation tangles were also observed associated with the austenite/ferrite interfaces. The retained austenite was also found to contain planar faults, often with one dominant fault plane. When the fault plane was approximately normal to the foil plane, the faults could be seen to terminate at slip steps in the austenite/ferrite interface indicative of accommodation slip on {111}γ planes during displacive transformation. Sandvik and Nevalainen [6] observed that the austenite adjacent to the bainitic ferrite contains fine twins, which divide the austenite into small blocks. The twin density was highest in the austenite between parallel, closely spaced ferrite plates and increased with decreasing transformation temperature.

In this work, the plastic relaxation of the shape deformation accompanying low temperature bainite has been examined by high resolution TEM (HR-TEM) and atom probe tomography (APT). Results have provided new experimental evidence on the accommodation of transformation strain accompanying bainitic ferrite growth.

Experimental Procedure
The chemical composition of the studied steels is given in Table 1. The alloys were supplied after casting, rolling and a homogenization heat treatment at 1200°C for 48 h. Specimens were austenitized and then isothermally transformed at 200°C or 300°C for different times before quenching into water. Details on the processing, heat treatments and preliminary microstructural characterization of this new generation of steels are reported elsewhere [1,7]. TEM specimens were sliced from 3 mm in diameter rods of the heat treated material, mechanically thinned to 0.06 mm, and then twin-jet electropolished to perforation using a mixture of 5% perchloric acid, 25% glycerol, and 70% ethanol at 10 – 0°C at 45V. The samples were examined on a Philips Tecnai F30 field emission gun TEM (FEG TEM) operated at 300 keV.

APT specimens were cut from the heat treated material and electropolished by the standard double layer and micropolishing methods [8]. APT analyses were performed in the ORNL local electrode atom probe (LEAP), which was operated in voltage pulsed mode with a specimen temperature of 60 K, a pulse repetition rate of 200 kHz, and a pulse fraction of 0.2. The carbon content segregated on defect features i.e. dislocations (linear features) and twins (plane features) was estimated from APT using concentration profiles in a selected volume including the defect feature or proximity histograms across that feature. Error bars for APT data represent the statistical scatter in the composition profiles.

Results and Discussion

The microstructure of this new generation of steels consists of 20-60 nm thick plates of bainitic ferrite and 20-40% austenite depending on the transformation temperature [1,7]. Retained austenite is enriched in carbon to 5-12 at. % C depending on its morphology and size. Nanoscale austenite films that were entrapped between neighbouring subunits of bainitic ferrite have a higher carbon content than the sub-micron blocks of residual austenite located between the sheaves of bainite [9]. A TEM micrograph of the microstructure formed at 200°C for 5 days in NB1 steel where the two different austenite morphologies are visible is shown in Fig.1a. In addition, a high level of carbon (~0.5 at. %), well above that expected from para-equilibrium with austenite (~0.12 at. %), is in solid solution in bainitic ferrite [10]. This super-saturation was attributed to the trapping of carbon at the dislocations in the bainitic ferrite [1].

The shape deformation associated with a displacive transformation of austenite in steel can be described as an invariant plane strain with a relatively large shear component. Christian [11] demonstrated that when the shape strain is elastically accommodated, the strain energy scales with the plate aspect ratio (thickness/length). This is why all of the displacive transformation products in steel such as Widmanstätten ferrite, bainitic ferrite, and martensite, occur in the form of thin plates. The need to minimise strain energy dictates a thin plate, but this also leads to a minimisation of the volume of transformation per plate. Therefore, a plate will tend to adopt the largest aspect ratio consistent with the available free energy change driving the transformation. In ideal circumstances, where the transformation interface remains glissile throughout, and where there is no friction opposing the motion of the interface, thermoelastic equilibrium occurs [12]. The aspect ratio of the plate adjusts so that the strain energy is equal to the driving force.

The thermoelastic equilibrium has been widely demonstrated for martensite [12], but it has not been straightforward for bainite. One reason for this is that bainite grows in the form of sheaves, which are groups of connected platelets which grow in parallel formations. The spacing between the platelets is to a large extent controlled by the carbon diffusion field associated with each platelet [2], therefore, the increase in platelet thickness is restricted by the diffusion fields and by the presence of adjacent platelets (i.e., soft and hard impingement effects).

A further complication is that the bainite transformation occurs at higher temperatures than those of martensite, where the austenite is mechanically weaker. The shape deformation therefore causes plastic deformation, and the resulting debris from dislocations eventually blocks the transformation interface, which loses coherency. Dislocation debris associated with the austenite/ferrite interfaces is evident in Fig. 1b. Consequently, platelets of bainitic ferrite are arrested in their growth even
when their size is much smaller than the austenite grain size. In this scenario, the plates are expected to become thicker at high temperatures because the yield strength of the austenite will then be lower.

**Dislocation structure in nanocrystalline bainitic steels**

This dislocation structure of bainite is often attributed to the shear transformation mechanism. However, such a mechanism would not lead to dislocations in the ferrite if the shape deformation were elastically accommodated. As mentioned above, thermoelasticity in martensite and shape memory alloys depends on the elastic accommodation of the shape deformation and the movement of any interfaces must occur without the creation of defects. It is only if the shape deformation is accompanied by plastic relaxation, that the dislocations associated with this plastic strain are inherited by the product phase.

The average dislocation density in ferrite and austenite of these novel microstructures was determined by TEM in NB1 steel transformed at 300°C for 3 days to be $\left(0.51 \pm 0.27\right) \times 10^{15} \text{ m}^{-2}$ and $\left(0.18 \pm 0.02\right) \times 10^{15} \text{ m}^{-2}$, respectively [13]. These values are above those reported for conventional bainite, $1.7-4.0 \times 10^{14} \text{ m}^{-2}$ [14] and, in general terms, similar to those measured for martensitic microstructures [15]. A progressive increase in the dislocation density in ferrite was also observed as the austenite/ferrite interface is approached [13]. This increase is related to the plastic deformation occurring in the surrounding austenite to accommodate the transformation strain as growth progresses and the following inheritance of those dislocations by the expansion of the growing bainitic ferrite plate [13].

Complementary APT results reveal carbon trapping at dislocations in the vicinity of a ferrite–austenite interface as shown in Fig. 2. The distribution of the carbon atoms in the analysis volume is not uniform and carbon-rich and carbon-depleted regions are clearly distinguishable. The carbon-enriched region at the top right of the atom map (Fig. 2a) represents austenite and the low carbon regions indicate the ferrite phase. Finally, the linear features with significant levels of carbon are dislocations in the vicinity of a ferrite–austenite interface. The 10 at. % C isoconcentration surface shown in Fig. 2b outlines the carbon-enriched region around a dislocation. It is evident from the proximity histograms across the dislocation in Fig. 2.c that dislocations only trap the carbon atoms, as originally suggested by Kalish and Cohen [16]. As a result of the large distortion caused by the supersaturated carbon in the ferrite lattice, there is an attracting interaction between carbon atoms and the stress fields of dislocations [17]. Carbon tends to diffuse to sites close to dislocations in order to reduce its chemical potential.

The average carbon level of the Cottrell atmosphere in Fig. 2 was estimated to be $14.0 \pm 2.2$ at. % C. This value is higher than the prediction of Cochardt et al. [18] of a saturation carbon concentration of 6–7 at. % in the region of one Burgers vector around the core. Wilde et al. [19] also observed a maximum carbon concentration within a dislocation atmosphere of 8 at. % C from an atom-probe analysis of a quenched and room temperature aged Fe–C alloy. The average carbon level of different Cottrell atmospheres detected by APT in NB1 steel is listed in Table 2. The range of APT values for the amount of carbon trapped at dislocations is remarkable. Carbon content values range from 5.8 to 14.0 at. % without an apparent correlation with the transformation time.

Cottrell and Bilby [17], developed the theory of segregation of carbon atoms to form atmospheres around dislocations to describe yielding and strain aging phenomena in low carbon steel. It was showed that the relief of stresses round a dislocation by foreign atoms in solid solution can cause an equilibrium atmosphere to form. A carbon atom solution in iron interacts with a dislocation and diffuses to it. The total area, which can supply carbon atoms to a dislocation in the interval of time from $t$ to $t+\Delta t$, is:

$$A,$$  

(1)
where $V_{\text{dis}}=2(\pi/2)^{1/3}(AD/kT)^{2/3}$, $D$ is the diffusion coefficient of carbon, $k$ the Boltzmann constant, $T$ the absolute temperature, and $A$ is a parameter defining the magnitude of the interaction between the dislocation and the carbon atom \[17\]. They neglected the effects of the saturation of dislocations and the variation of carbon concentration during aging and calculated the degree of the atmosphere formation by:

\[
\frac{-}{}, \quad (2)
\]

where $N(t)$ is the number of carbon atoms diffusing to dislocations in a unit volume, $N_{0\theta}$ is the initial dislocation density, $n_0$ the initial concentration of carbon in solid solution and $\lambda$ is the slip distance of the dislocation.

More recently, Zhao et al. \[20\] described the formation of Cottrell atmosphere in an ultra-low carbon steel assuming that the segregation rate of carbon atoms to dislocations is proportional to the local concentration of the active carbon atoms, $n(t)$, and the density of the unpinned dislocations, $L(t)$:

\[
\frac{-}{}, \quad (3)
\]

The evolution of the carbon content in solid solution in ferrite was determined by APT during transformation at 200 ºC in NB1 steel \[21\]. Results indicated that, as the formation of bainitic ferrite progresses, the super-saturated ferrite is gradually decarburized (Table 2). The density of the dislocations as function of transformation time has not been determined in bainitic microstructures, but it is reasonable to assume that it will increase as bainitic ferrite grows. Based on this theory, the trend of the segregation of carbon atoms to dislocations reported in Table 2 as function of time may not be so obvious.

Energy calculations in dislocation theory are often made in the approximation of a dislocation line tension. The dislocation is considered to be a smooth flexible string with a line tension. The self-energy of a segment of dislocation line depends on its interaction with the other portions of the line, and hence depends on the dislocation configuration, so that the effective line tension is difficult to define with much accuracy. Hirth and Lothe \[22\] provided explicit expressions for the elastic energy of any configuration made up of straight segments and developed line tensions appropriate for the various cases from the more general theory. Their theory reflected the fact that a dislocation segment interacts with all other segments of a dislocation in complex configuration. Qualitatively, the stress field of a given dislocation is largely cancelled out by the stress fields of the reminder of the complex array of dislocations outside a region, approximately cylindrical with radius the interdislocation spacing, bounding the dislocation. Consistently with the line-tension concept, Pereloma et al. \[23\] found by APT that the extent of solute segregation to a dislocation depends on its position relative to other defects. Their results indicated that the sizes of the atmospheres are larger and the maximum levels of carbon are higher on dislocations located far apart compared to those in tangles or array arrangements. This observation suggested that, taking all other conditions to be the same in ferrite, the extent and level of carbon segregation is dependent on the overall carbon content and the amount of solute available in the local vicinity. If the dislocation atmosphere can consume the solute from a larger volume, then the atmosphere is larger, more homogeneous, and has a higher carbon content. In addition, the carbon concentration in the atmosphere will depend on the type of dislocation. Cochardt et al. \[18\] pointed out that the atmosphere around a screw dislocation can bind more than twice the number of carbon atoms associated with an edge atmosphere. This is because segregation takes place all around a screw dislocation but only “below” the slip plane of an edge dislocation. Unfortunately, it is not possible to determine the type of dislocation observed by APT, as only the shape of and the solute levels at the observed atmosphere is revealed by this technique.
Baird [24] pointed out that the binding energy of interstitial atoms to dislocations is higher than the binding energies of cementite precipitates. Kalish and Cohen [16] proposed a dislocation trapping model in which carbon prefers to be located at dislocations rather than in ε-carbide or cementite precipitates. In addition, Cochardt et al. [18] stated that the maximum interaction energy at a distance of one Burgers vector from both edge and screw dislocations is 0.75 eV, whereas the binding energy of carbon in cementite is only 0.5 eV [24]. This suggests that, within a distance of one Burgers vector, carbon atoms are more stable in a dislocation atmosphere than in cementite. Extensive TEM examination of these nanocrystalline steels failed to reveal carbide particles inside the bainitic ferrite [1]. This is indeed an interesting observation, as at these temperatures, steels with such high carbon levels (Table 1) would transform to a lower bainitic microstructure. The difference between upper and lower bainite comes from competition between the rate at which carbides can precipitate from ferrite and the rate at which carbon is partitioned from supersaturated ferrite into austenite [25]. The precipitation of cementite from lower bainite can occur at temperatures below 125 ºC in time periods that are too short to allow any substitutional diffusion of iron atoms. The long range diffusion of carbon atoms is of course necessary, but because carbon resides in interstitial solution, it can be very mobile at temperatures as low as -60 ºC [26]. It is believed that the solute segregation to defects observed in these novel steels plays an important role in carbon redistribution during bainite transformation. Segregation to dislocations is expected to bind and hence prevent or hinder the carbon atoms from diffusing out of the ferrite lattice and from precipitating within the ferrite [10]. This explains the high level of carbon that exists in the bainitic ferrite after transformation in nanocrystalline steels [21].

*Accommodation twinning in nanocrystalline bainitic steels*

Plastic relaxation of the shape change occurring in the adjacent austenite can also take the form of accommodation twinning [2], so called in order to distinguish these mechanical twins from the transformation twins observed in martensite due to the lattice invariant strain [27]. Accommodation twinning is apparent in TEM micrographs of NB2 steel transformed at 200ºC for 10 days shown in Fig. 3. The bright field image in Fig. 3b illustrates the austenite in contact with a bainitic ferrite plate exhibiting, apart from dislocations debris associated with the austenite/ferrite interface, extensive twinning. These twins are lenticular in shape with a thickness of approximately 2 to 10 nm. The corresponding diffraction pattern of Fig 3.c confirms that the bainitic ferrite obeys the Kurdjumov-Sachs orientation relationship with the austenite and inheriting twins from austenite. Zhang and Kelly [28] obtained similar results for a nanocrystalline bainitic steel (0.8C–1.6Si–2.0Mn–1.0Al–0.2Mo–1.0Cr–1.5Co–0.002P in wt-%) using a convergent beam Kikuchi line diffraction technique. Nanoscale twinning austenite was also observed on NB3 steel after isothermal transformation at 200ºC for 10 days and tempering at 400ºC for 30 min. [29]. The trace of the twinning was displaced by an angle of 9-12º after crossing the austenite/ferrite interface verifying that pre-existing twin caused by the shape strain of bainitic transformation was displaced by the shape strain of another plate of transforming bainitic ferrite [30].

A HR-TEM image of a nanoscale twin formed at the austenite/bainitic ferrite interface holding nearly the Kurdjumov-Sachs (K-S) orientation relationship is shown in Fig. 4a. HR-TEM images of mechanical twins often contains Moiré fringes parallel to the twinning plane because it is often embedded in the austenite matrix due to its lenticular shape. The lattice shear from \{111\}_γ to \{101\}_α with a shear angle of 10.5º is identified at the interface in HR-TEM image shown in Fig. 4a. Scheme in Fig. 4b illustrates that the lattice deformation results in a 10.5º displacement from austenite to ferrite under the K-S orientation relationship. This angle is comparable to the observed for twinning at the interface. Moreover, Fig. 4a and 4b show the twinning shear to be identified as a \{111\}<111> mode. It was suggested that these defects are indicative of accommodation slip on \{111\}_γ planes [5]. Kang et al. also found \{111\}_γ defects during bainite transformation by in-situ TEM observations using specimen heating holder [31].
It is known that impurities such as phosphorus, calcium and silicon can segregate to incoherent twin boundaries with a high free volume [32–34]. The concentration profile in Fig. 5 shows a very fine-scale modulation in the carbon atom map of a sample transformed at 200 °C for 2 days. These carbon-enriched planes, whose thickness compares well with that of the twins illustrated in Fig. 3b, were identified as twin boundaries in retained austenite. Their average carbon content was estimated to be 9.6±0.8 at.%. The formation of nanoscale twins requires significant amount of carbon in austenite together with large shape strains. These requirements and the observed crystallographic features are consistent with a displacive transformation mechanism for bainite [5].

Conclusions

The plastic relaxation of the austenite accompanying bainite transformation at abnormally low temperatures has been examined by TEM and APT in a new generation of nanocrystalline steels. High resolution examination of these novel microstructures has revealed dislocations debris associated with the austenite/ferrite interface and nanoscale twins in the austenite in contact with bainitic ferrite plates. They are related to the plastic deformation occurring in the surrounding austenite to accommodate the transformation strain as bainitic ferrite growth progresses. In addition, APT revealed that a substantial quantity of carbon was trapped at dislocations and twins. Carbon trapping at defects prevents the decarburization of super-saturated ferrite and, to some extent, alters the carbide precipitation sequence during low temperature bainite formation.

Acknowledgement

Atom probe tomography research supported by ORNL’s Shared Research Equipment (SHaRE) User Facility, which is sponsored by the Office of Basic Energy Sciences, U.S. Department of Energy. The authors gratefully acknowledge the support of the Research Fund for Coal and Steel and the Spanish Ministry of Science and Innovation for funding this research under the contracts RFSR-CT-2008-00022 and MAT2007-63873, respectively. J. Cornide also acknowledges the Spanish Ministry of Science and Innovation for financial support in the form of a PhD research grant (FPI).

References

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<th>Mn</th>
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<td>wt%</td>
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<td>1.9</td>
<td>0.3</td>
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<tr>
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<td>1.8</td>
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<td>at. %</td>
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<td>1.9</td>
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Table 1. Chemical Composition of Nanocrystalline Bainitic Steels
Table 2. Average carbon level of the Cottrell atmosphere and in solid solution in ferrite (at-%) measured by APT in NB1 steel

<table>
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<th>Isothermal Heat Treatment</th>
<th>Carbon in Cottrell Atmosphere</th>
<th>Carbon in Solid Solution in Ferrite</th>
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<td>200 °C-2 days</td>
<td>7.4 ± 0.1</td>
<td>1.4 ± 1.6&lt;sup&gt;a&lt;/sup&gt;</td>
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<tr>
<td></td>
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<td>1.3 ± 0.4</td>
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<td>1.3 ± 0.4</td>
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<td></td>
<td></td>
<td>(1.4 ± 0.1)&lt;sup&gt;b&lt;/sup&gt;</td>
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<tr>
<td>200 °C-4 days</td>
<td>7.4 ± 2.6</td>
<td>0.5 ± 1.0</td>
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<td></td>
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<td>1.1 ± 0.4</td>
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<td>1.0 ± 1.3</td>
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<td></td>
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<td>(0.9 ± 0.2)</td>
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<td>200 °C-6 days</td>
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<td>1.0 ± 0.8</td>
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<td></td>
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<td>(0.8 ± 0.2)</td>
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<td>200 °C-10 days</td>
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<td>(0.6 ± 0.5)</td>
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</table>

<sup>a</sup> Error bars representing the statistical scatter in the APT composition profiles due to the number of ions in each slice of the selected volume of analysis.

<sup>b</sup> Values in parentheses are mean values of the APT measurements listed inside the same cell. Their corresponding error bars are standard deviations of the mean value representing the dispersion of a collection of APT measurements.
Figure 1.- Transmission electron micrographs of NB1 steel transformed at 200°C for (a) 5 days and (b) 4 days. α is bainitic ferrite, γ₀ is sub-micron block of austenite, γ₁ is nanoscale film of austenite and γ is austenite.
Figure 2.- (a) Carbon atom map, (b) carbon isoconcentration surfaces at 10 at.% C superimposed with the carbon atom map, and (c) proximity histograms across (b) a dislocation in the vicinity of the ferrite/austenite interface in bainitic ferrite after transformation at 200°C for 6 days in NB1 steel.
Figure 3.- Transmission electron micrographs of NB2 steel transformed at 200°C for 10 days: (a) general microstructure; (b) nanoscale twins in retained austenite; and (c) corresponding diffraction pattern. $\alpha$ is bainitic ferrite and $\gamma$ is austenite.
Figure 4.- (a) High resolution transmission electron micrograph of a nanoscale twin at the austenite/bainitic ferrite interface in NB2 steel transformed at 200°C for 10 days; (b) scheme of the lattice correspondence of austenite in projection and bainitic ferrite in projection.
Figure 5.- (a) Carbon atom map and (b) concentration profile showing carbon segregation about nanoscale twins in retained austenite after transformation at 200°C for 2 days in NB1 steel.