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# Analyzing the scale of the bainitic ferrite plates by XRD, SEM and TEM

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ABSTRACT

Since the major strengthening mechanisms in nanocrystalline bainitic steels arise from the exceptionally small size of the bainitc ferrite plate, accurate determination of this parameter is fundamental for quantitative relating the microstructure to the mechanical properties. In this work, the thickness of the bainitic ferrite subunits obtained by different bainitic heat treatments was determined in two steels, with carbon contents of 0.3 and 0.7 wt.%, from SEM and TEM micrographs. As these measurements were made on 2D images taken from random sections, the method includes some stereological correction factors to obtain accurate information. Finally, the determined thicknesses of bainitic ferrite plates were compared with the crystallite size calculated from the analysis of X-ray diffraction peak broadening. Although in some case the values obtained for crystallite size and plate thickness can be similar, this study confirms that indeed they are two different parameters.

## 1. Introduction

Microstructures consisting of a matrix of bainitic ferrite with a second phase dispersion of other constituents, such as retained austenite or M-A (martensite-austenite) constituent have been developed by optimizing heat treatment schedules and chemical composition in Si rich steels [1–4]. It has been proposed that like in the case of martensite, the growth of bainite is displacive, and thus it can be described by an invariant plane strain (IPS). The associated strain has two components, a large shear deformation with a value of about 0.46 for nanostrutured bainite and in the range of 0.22–0.26 for sub-micron bainite [5,6]. The second component is a smaller dilatational strain (~0.03). The associated strain is so large that the transformation product is in the form of a thin plate, which minimizes the strain energy when the plate is elastically accommodated. As the large strains associated with the transformation cannot be sustained only elastically by the parent austenite, those are relaxed by the plastic deformation of austenite [5,7]. Under such scenario, generated dislocations are inherited by the product phase, bainitic ferrite, limiting the final thickness of the formed bainitic ferrite plate [8,9]. But, there are other important factor controlling the scale of the microstructure, thus, a strong austenite or a large driving force would also lead to a refinement of the bainitic ferrite plates, the former because there is a larger resistance to interface motion and the latter because an increased nucleation rate leads to microstructural refinement [10,11]. The magnitude of the mentioned parameters, dislocation density, austenite strength and driving force, increases as temperature decreases, explaining the observed refinement of bainitic microstructures when the transformation temperature is lowered [7].

At this point it is necessary to clarify the different morphologies that bainite might present. One of the most recent and comprehensive classifications was given by Zajac et al. [12,13], which may be applied for both low-carbon and high-carbon bainite. Thus, bainite is divided into groups according to bainitic ferrite morphology and the type and distribution of second phases: granular bainite with irregular bainitic ferrite grains containing globular island shaped second phases (either retained austenite or martensite); upper bainite with several lath-like bainitic ferrite crystals parallel to each other within sheaves and separated by a second phase (either cementite or M-A constituent); and lower bainite with bainitic ferrite plates containing a fine dispersion of precipitated carbide particles inside. It is important to remark that in granular bainite microstructures, the relatively coarse grains of ferrite present a well-developed substructure. In fact, they consist of sheaves of parallel plate-like subgrains of bainitic ferrite with very thin regions of austenite between the sub-units because of the low carbon concentration of the steels involved [14].

Carbide precipitation during bainitic transformation can be suppressed by suitable alloying element. It has been demonstrated that instead of the classical structure of bainitic ferrite laths separated by carbide crystals, a microstructure consisting of bainitic ferrite subunits interwoven with thin films of untransformed retained austenite can be obtained by alloying the steel with about 1.5 wt.% of Si [7].

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Like in the case of martensite, sub-units of bainitic ferrite would change its morphology from lath-like to plate-like with increasing carbon content, low transformation temperatures or in strong austenite [7].

It is well documented that most of the strength of this microstructure relies on the scale of the bainitic ferrite [1–4] and it is the plate thickness rather than the length which primarily determines the mean free slip distance [15]. Therefore, accurate determination of plate thickness is fundamental for quantitative relating the microstructure to the mechanical properties. This paper deals with the determination of the bainitic ferrite plate thickness by means of SEM and TEM. For this purpose, two very different, in terms of the scale, bainitic microstructures were selected, one in the nano range the other submicron. As plate thickness determination from TEM or SEM micrographs can be tedious and time consuming, it has been recognized in previous works that the crystallite size determination from XRD peak profile analysis was an alternative [16]. But a direct comparison between the sizes obtained using an image analysis procedure with the crystallite size calculated from XRD patterns showed that indeed crystallite and plate size refer to two very different concepts.

## 2. Materials and Experimental Procedures

For the purpose of this work, two bainitic steels with chemical compositions given in Table 1 were used. They will be referred according to the scale of their final bainitic microstructure, i.e. as Nano and Sub for a nanostructured and submicron bainite, respectively. Both alloys were selected for having very different C contents, which also implies very distinctive Bs and Ms transformation temperatures, and also final scale of the bainitic microstructure, see Table 1. A common feature in both alloys is a high Si content, 1.5 wt.%, suffice to avoid cementite precipitation during bainitic transformation [7]. Other elements as Mn and Cr are added for hardenability purposes, Mo to prevent temper embrittlement due to P [17], and Co and Al were added to the Sub steel grade with the solely purpose of accelerating the rate of transformation [18,19].

Cylindrical samples, 4 mm in diameter and 10 mm long, were heat treated in a Bahr 805D high-resolution dilatometer. After austenitisation, samples were cooled down to selected isothermal temperatures and held for different periods of time to ensure the end of the bainitic transformation, after which they were cooled down to room temperature.

The process parameters, i.e. the austenitisation temperature, cooling rate to the isothermal temperature, and the value of this same temperature, were adapted to the alloy chemical composition and to its hardenability, the latter to avoid the formation of ferrite and pearlite prior to bainite transformation, see Table 2 for further details.

Experimental determination of the Ms. temperatures by He quenching from the austenitisation temperature, assisted in the selection of the bainitic transformation temperatures, see Table 1. As mentioned, for most of the experiments, the selected isothermal holding times were those strictly necessary to ensure the end of the bainitic transformation. But, an experiment was kept longer than strictly necessary for reasons later clarified, this experiment is identified by the '+' symbol.

Microstructures present in the alloys after these heat treatments were characterized using both, field emission gun scanning electron microscopy (FEG-SEM) and transmission electron microscopy (TEM). Standard grinding and final polish with 1 μm diamond paste were used for SEM sample preparation. Microstructural features were developed by etching with 2% Nital. TEM specimens were sliced from 3-mm-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 °C at 45 V. The samples were examined on a TEM JEOL 2010 transmission electron microscope operated at 200 keV. Finally, the thickness of the bainitic ferrite subunits was measured directly from SEM and TEM micrographs using up to 100 measurements to provide acceptable accuracy.

**Table 1**
Chemical composition of the alloys in wt.%. Experimental martensite start temperature, Ms., and theoretical bainite start temperature, Bs, according to ref. [20], all in °C.

| | C | Si | Mn | Cr | Mo | Ni | Co | Al | Ms | Bs |
|---|---|---|---|---|---|---|---|---|---|---|
| Nano | 0.7 | 1.5 | 1.4 | 1.0 | 0.2 | 0.1 | | | 170 | 350 |
| Sub | 0.3 | 1.4 | 2.0 | 1.6 | 0.2 | 1.4 | 1.7 | 1.0 | 220 | 460 |

**Table 2**
Conditions for bainitic heat treatments. Tγ stands for the austenitisation temperature, C.R. is the applied cooling rate, and $T_{iso}$ and $t_{iso}$ stand for the isothermal heat treatment temperature and time, respectively. The symbol + denotes that the treatment was extended far more than strictly necessary to end the transformation.

| | Tγ (°C) | C.R. (°C/s) | $T_{iso}$ (°C) | $t_{iso}$ (h) |
|---|---|---|---|---|
| Nano-220(+) | 900 | 35 | 220 | 168 |
| Nano-220 | | | 220 | 24 |
| Nano-250 | | | 250 | 14 |
| Nano-300 | | | 300 | 5 |
| Nano-350 | | | 350 | 4.5 |
| Sub-325 | 925 | 50 | 325 | 2 |
| Sub-350 | | | 350 | 2 |
| Sub-375 | | | 375 | 2 |
| Sub-400 | | | 400 | 2 |

Specimens for XRD analysis were prepared by the described procedure, but in order to remove the deformed layer, where austenite may have transformed to martensite by TRIP effect, several cycles of etching and polishing were applied before a final polish with colloidal silica suspension (40 nm). XRD measurements were carried out with a Bruker AXS D8 diffractometer equipped with a Co X-ray tube, Goebel mirror optics and a LynxEye Linear Position Sensitive Detector for ultra-fast XRD measurements. A current of 30 mA and a voltage of 40 kV were employed as tube settings. The XRD data were collected over a 2θ range of 35–135° with a step size of 0.01°. In this study, the version 4.2 of Rietveld analysis program TOPAS (Bruker AXS) was used for quantification and calculation of structural parameters of both, retained austenite and bainitic ferrite. Line broadening effects due to the crystallite size and lattice strain were analyzed with the double-Voigt approach, in which both the crystallite size and strain comprise Lorentzian and Gaussian component convolution [21]. In order to eliminate the instrumental contribution to peak broadening, instrument functions were empirically parameterized from the profile shape analysis of a corundum sample measured under the same conditions.

Reported hardness measurements (HV20) corresponds to an average of at least 4 measurements.

## 3. Microstructure and Experimental Results

### 3.1. General Description

Regardless of the steel and heat treatment, the microstructure in all cases consisted in bainitic ferrite and retained austenite. TEM observations, as reported in previous works, failed to reveal cementite precipitation [9,22]. Examples of the microstructures at selected temperatures and magnifications can be found in Fig. 1, where both phases have been identified. XRD measurements come to support these results, showing that the predominant phase is ferrite, the remaining being retained austenite, Fig. 2 (a). As expected from the incomplete reaction phenomena ruling bainitic transformation [7], the maximum extent of transformation increases as transformation temperature decreases, i.e. ferrite fraction decreases as isothermal temperature increases. However it has to be noted that for Sub steel the tendency is not respected in the whole temperature range, as at 375 °C and 400 °C there is an increase in the amount of ferrite detected, Fig. 2 (a). It is important to note here that during transformation, austenite (γ) is enriched in carbon relative to the average composition of the steel, due to the partial partitioning of