



ELSEVIER

Contents lists available at ScienceDirect

## Journal of Magnetism and Magnetic Materials

journal homepage: [www.elsevier.com/locate/jmmm](http://www.elsevier.com/locate/jmmm)

## Phase quantification in nanobainite via magnetic measurements and X-ray diffraction

W. Solano-Alvarez<sup>a,\*</sup>, H.F.G. Abreu<sup>b</sup>, M.R. da Silva<sup>c</sup>, M.J. Peet<sup>a</sup><sup>a</sup> Department of Materials Science and Metallurgy, University of Cambridge, UK<sup>b</sup> Departamento de Engenharia Metalúrgica e de Materiais, Universidade Federal do Ceará, Fortaleza, Brazil<sup>c</sup> Instituto de Física e Química, Universidade Federal de Itajubá, Itajubá, Minas Gerais, Brazil

## ARTICLE INFO

## Article history:

Received 9 October 2014

Received in revised form

12 November 2014

Accepted 13 November 2014

Available online 15 November 2014

## Keywords:

Nanostructured bainite

Phase quantification

Saturation magnetization

X-ray diffraction

## ABSTRACT

Accurate phase quantification of nanostructured bainitic steel is of importance because of the nature of its percolating structure that controls many of its mechanical properties. X-ray diffraction is the technique of choice for such analysis, but magnetic methods can be more rapid and less sensitive to defect structures. In this study, the phase volume fractions measured using both of these techniques for the specific mixtures associated with nanostructured bainite have been compared and contrasted. An expression which relates the volume fraction and the saturation magnetization is obtained and its form is found to be consistent with previous work done on duplex stainless steels and TRIP steels. The fitting constants used in many of such analyses vary significantly so an attempt is made to rationalize the differences by considering the factors that determine the intrinsic saturation magnetization of ferrite.

© 2014 Elsevier B.V. All rights reserved.

## 1. Introduction

Nanostructured bainite contains an extremely fine mixture of ferrite and austenite. The ability of the austenite to percolate the steel dictates amongst other factors, the ductility [1] and its resistance to the diffusion of hydrogen through the two-phase mixture [2]. Given the specific austenite morphology characteristic of nanostructured bainite, there is a critical threshold of austenite fraction below which the material fractures during tensile loading [3], and the austenite also ceases to be effective as a barrier to hydrogen ingress [2].

There are many methods to determine the retained austenite content [4–8], each of which has limitations that have been discussed extensively [9,10]. Diffraction using X-rays or neutrons has the advantage of rigor in the analysis of data and remains the method of choice for phase fraction analysis. However, magnetic methods to determine volume fractions can be rapid to implement even though they may require calibration. One difficulty is that the calibration constants seem to vary widely as a function of the alloy composition and microstructure [11–16]. In the present work we investigate X-ray diffraction and magnetic detection of retained austenite in nanostructured bainitic steel, and attempt to clarify the nature of the calibration function required for the magnetic

technique. Given that there are now several commercial applications and numerous research programmes associated with this structure, it would be useful to have an easy method to characterize the retained austenite content with confidence.

## 2. Experimental methods

## 2.1. Material

All alloys used were cast as 25–50 kg ingots, which were then reduced 50% via hot forging. The chemical compositions are presented in Table 1.

## 2.2. Heat treatments

The heat treatment of small samples cut via electrical-discharge machining included homogenization at 1200 °C for 2 days and furnace cooling, austenitization in the  $\gamma$  region (900–1000 °C) for 15 min and air cooling, bainitic transformation at different temperatures and times (Table 2) before being quenched in water at room temperature, and in some cases tempering also for different times and temperatures. For the two heat treatments prior to the bainitic transformation, samples were sealed in quartz tubes back-filled with argon or in a vacuum furnace. Cooling slowly through the 700–550 °C range, following homogenization, was used to ensure pearlite formation and avoid martensite, which would induce quench cracks [17]. Extensive characterization by

\* Corresponding author. Tel.: +44 1223 334336.

E-mail addresses: [ws298@cam.ac.uk](mailto:ws298@cam.ac.uk), [wilberths@hotmail.com](mailto:wilberths@hotmail.com) (W. Solano-Alvarez).

**Table 1**  
Chemical composition, wt%, of the alloys used.

Alloy	C	Si	Mn	Cr	Mo	Ni	V	Co	P	S	Al
A	0.79	1.59	1.94	1.33	0.30	0.02	0.11	–	< 0.005	–	–
B	0.80	1.59	2.01	1.00	0.24	–	–	1.51	0.002	0.002	–
C	0.79	1.56	1.98	1.01	0.24	–	–	1.51	0.002	0.002	1.01
D	0.78	1.60	2.02	1.01	0.24	–	–	3.87	0.002	0.002	1.37

X-ray diffraction and microscopy in these and similar conditions has been reported previously [18–22].

The main three parameters that determine the quantity of the ferromagnetic ferrite present are the mass, chemical composition, and the heat treatment of the samples. Therefore, the magnetization was studied by altering only one of these three parameters at a time in order to separate out their individual contributions. For example, samples 6 and 7 have different compositions but constant heat treatment and approximately constant mass; samples 8, 9 and 10 have different mass but constant composition and heat treatment; samples 3 and 4 have different heat treatment but constant composition and approximately constant mass; and finally samples 1 and 2 have no variation, meaning constant heat treatment, composition, and very similar mass.

### 2.3. X-ray diffraction

Samples were polished to a 1  $\mu\text{m}$  finish, etched with 2% Nital and analyzed with X-ray diffraction (XRD) to measure the volume fractions of bainitic ferrite and retained austenite using a Philips PW1830 vertical diffractometer with unfiltered  $\text{CuK}\alpha$  radiation. Scans were performed with an acceleration voltage and current of 40 kV and 40 mA from 40 to 125°, with a step size of 0.05° and a dwell time of 26 s. A divergence slit of 0.5°, an anti-scatter slit of 0.5°, and a receiving slit of 0.2 mm were used to restrict the beam size and the counts obtained. The volume fractions of ferrite and austenite were derived using *HighScore Plus* and the Rietveld refinement method.

### 2.4. Transmission electron microscopy

Samples were cut into thin slices 250  $\mu\text{m}$  thick using a silicon carbide cutting wheel and ground down to 40–100  $\mu\text{m}$  using 1200 silicon carbide grinding paper. These thin discs of 0.3 mm in diameter were then electropolished using a twin jet polishing machine and a solution of 80% ethanol, 15% glycerol, and 5% perchloric acid with voltages between 20 and 40 V. Samples were observed in a JEOL 200 CX with an accelerating voltage of 200 kV.

### 2.5. Vibrating sample magnetometer

The magnetization and coercive field of the different samples were measured at room temperature using the TEM discs prior to electropolishing with a VSM EGG-PAR model 4500 with a maximum applied field of 600  $\text{kA m}^{-1}$  and total measuring time of 25 min. These conditions were chosen in order to be able to compare the results to XRD volume fractions and to other magnetic measurements in the literature, which were mostly performed at room temperature. This study concentrates only on dual phase systems, but it should be noted that for the tempered samples additional measurements could be performed above the Curie temperature of cementite (480 K) without affecting the structure in order to obtain information about the carbide phase fractions.

## 3. Results and discussion

Given the similarity in heat treatments and expected nanostructure, only some representative samples were studied using TEM. The images obtained are presented in Fig. 1. For the sake of brevity, samples from here onwards are referred to as  $X - T^{\text{isotrans}} - t^{\text{isotrans}} - T^{\text{temper}} - t^{\text{temper}} - Y$ , where  $X$  is the alloy type,  $T$  stands for the temperature in [°C],  $t$  is the time in hours [h], days [d], or years [y], and  $Y$  is the sample number in case of there being more than one with the same composition and heat treatment.

The stereologically corrected bainitic plate size of some untempered samples was determined using the mean width in the direction normal to the plate length. For samples 1 (A-200C-10d-1) and 2 (A-200C-10d-2) the mean thickness is  $39 \pm 1$  nm and for sample 5 (B-150C-1y) it is  $51 \pm 4$  nm [18].

The volume fraction of retained austenite obtained via Rietveld analysis of the complete XRD spectra (including overlapping peaks) is presented in Table 3 along with the values of saturation magnetization per unit mass obtained at the maximum applied field and the effective field corrected by the demagnetization factor corresponding to shape (thickness) of each sample [23]. The magnetization curves are presented in Fig. 2.

**Table 2**  
Heat treatments performed on different samples. The weight column corresponds to the final sample geometry of thin discs used for TEM and magnetic measurements.

Sample	Alloy	Weight/g	Isothermal transformation		Tempering	
			Temperature/°C	Time	Temperature/°C	Time
1	A	0.01135	200	10 days	–	–
2	A	0.01065	200	10 days	–	–
3	A	0.00965	200	10 days	400	1 h
4	A	0.0089	200	10 days	500	1 h
5	B	0.00405	150	1 year	–	–
6	B	0.00395	200	4 days	–	–
7	C	0.0036	200	4 days	–	–
8	D	0.0099	200	4 days	–	–
9	D	0.0064	200	4 days	–	–
10	D	0.01235	200	4 days	–	–

Download English Version:

<https://daneshyari.com/en/article/1799305>

Download Persian Version:

<https://daneshyari.com/article/1799305>

[Daneshyari.com](https://daneshyari.com)