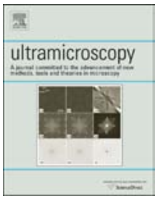
Ultramicroscopy 148 (2015) 1–9

Contents lists available at ScienceDirect

Ultramicroscopy

journal homepage: www.elsevier.com/locate/ultramic



Comparison between magnetic force microscopy and electron back-scatter diffraction for ferrite quantification in type 321 stainless steel

A.D. Warren [a,*], R.L. Harniman [b], A.M. Collins [b,c], S.A. Davis [b], C.M. Younes [a], P.E.J. Flewitt [a,d], T.B. Scott [a]

[a] *Interface Analysis Centre, HH Wills Laboratory, University of Bristol, Bristol BS8 1FD, UK*
[b] *School of Chemistry, University of Bristol, Bristol BS8 1 TS, UK*
[c] *Bristol Centre for Functional Nanomaterials, Nanoscience and Quantum Information Centre, University of Bristol, Bristol BS8 1FD, UK*
[d] *School of Physics, HH Wills Laboratory, University of Bristol, Bristol BS8 1FD, UK*

ARTICLE INFO

Article history:
Received 8 November 2013
Received in revised form
4 August 2014
Accepted 8 August 2014
Available online 23 August 2014

Keywords:
Ferrite
Austenite
Electron backscatter diffraction
Magnetic force microscopy
Type 321 stainless steel

ABSTRACT

Several analytical techniques that are currently available can be used to determine the spatial distribution and amount of austenite, ferrite and precipitate phases in steels. The application of magnetic force microscopy, in particular, to study the local microstructure of stainless steels is beneficial due to the selectivity of this technique for detection of ferromagnetic phases. In the comparison of Magnetic Force Microscopy and Electron Back-Scatter Diffraction for the morphological mapping and quantification of ferrite, the degree of sub-surface measurement has been found to be critical. Through the use of surface shielding, it has been possible to show that Magnetic Force Microscopy has a measurement depth of 105–140 nm. A comparison of the two techniques together with the depth of measurement capabilities are discussed.

© 2014 Elsevier B.V. All rights reserved.

1. Introduction

During exposure at high temperatures it has been observed that microstructural changes including second phase precipitation occurs in austenitic stainless steels [1]. Such changes can exert a negative influence on the chemical, physical and mechanical properties of these materials [1,2]. Although low volumes of ferrite are regarded as beneficial under certain circumstances (*e.g.* in welds), higher volume fractions can facilitate cracking and decreased corrosion resistance [3–6]. Correspondingly, it is important to have reliable analysis techniques for identification and quantification of the volume fraction and distribution of the various phases present. There are many characterisation techniques which can be used to monitor the evolution of phases, all with their own benefits and limitations [7–9]. Arguably, the most effective techniques are those which offer a unique identification, mapping and quantification facility, such as orientation mapping based on electron backscatter diffraction EBSD analysis. However many of the techniques make measurements at different length scales.

Magnetic force microscopy (MFM) is a variant of scanning probe microscopy [10], which utilises the interaction of a susceptible probe-tip with the magnetic domains of the sample to (i) measure the magnetic force and (ii) generate a map of the magnetic domains at the near surface of the material [11]. In addition, this technique is capable of mapping the surface topography of the material using the probe tip for standard tapping mode atomic force microscopy (AFM). MFM has found a widespread use in the analysis of magnetic recording materials [12] and has become well established for fault-finding and quality assurance testing in electronic data storage media [10,11]. Applied to the analysis of stainless steels, MFM potentially offers enhanced resolution at an increased mapping speed compared to EBSD, and has been used to map the distribution of ferromagnetic delta-ferrite (δ) and paramagnetic austenite (γ) in duplex steels [13–15]. Gadelrab et al. briefly postulated that the technique might be capable of detecting sub-surface ferrite after observing weak magnetic signals in a region of austenite [15].

The present study applies MFM to an aged Type 321 stainless steel plate, and directly compares maps recorded by both EBSD and MFM for the same region to establish the quantities

---

* Corresponding author.
E-mail address: Xander.Warren@bristol.ac.uk (A.D. Warren).

http://dx.doi.org/10.1016/j.ultramic.2014.08.006
0304-3991/© 2014 Elsevier B.V. All rights reserved.

of ferrite determined by both methods. The depth sensitivity for sub-surface ferrite detection is quantified in Section 3and is crucial to measuring the amount of ferrite in the Type 321 stainless steel.

2. Experimental procedure

The material used for this study was a Type 321 stainless steel plate. The specimen was cut normal to the plane of rolling and parallel to the direction of rolling. The plate had been aged at 650 °C for 99,930 h, followed by 8760 h at 750 °C and air cooled. The base material selected for the depth of measurement determination was a piece of 99.99% pure α-Fe. The chemical composition of the Type 321 plate metal is given in Table 1.

Specimens were cut to size using a rotary slow speed cutter and mechanically polished down to a 0.25 μm surface finish. This was followed by final polishing using colloidal silica to achieve a surface-roughness of 15 nm root mean square.

Subsequent EBSD analysis was performed in a Zeiss EVO MA10 scanning electron microscope (SEM) fitted with a LaB_6 electron source and a high-speed camera (DigiView 3). A spacial resolution better than 20 nm was achieved for the local orientation measurements [16,17]. The EBSD scans were performed by operating the SEM at 30 kV, with the specimen tilted by 70° to the horizontal. EBSD maps were acquired using a step size of either 0.4 μm or 1 μm. Orientation image mapping (OIM) data collection software (Ametek, Utah, USA), was used to record and analyse the EBSD data. All maps shown were subjected to confidence-index thresholding to remove any data present with a confidence-index value of less than 5%. The volume fraction of phases present in EBSD data is determined by the number of pixels (and thus diffraction patterns) which are present within the mapped area.

The AFM system used for magnetic analysis was a Bruker Multimode instrument fitted with a Nanoscope V controller and a Picoforce extender, fitted with a Budget Sensors Multi75M-G tip (Innovative Solutions Bulgaria Ltd.) of 'high' coercivity. These tips had a Co alloy magnetic coating, 85 ± 5 nm thick at a distance of approximately 300 nm away from the tip, which thinned to 50 ± 10 nm thick for the majority of the tip body. The manufacturer specified tip radius was < 60 nm. The optimum settings for imaging were determined to be a lift scan height of 50 nm with a drive amplitude based noise reduction. The system has a potential spatial resolution of ~ 10 nm [18]. A step size of 170 nm was utilised for this study with the scan rate optimised at 0.54 Hz and 1024 lines, to give a compromise between retrace path recovery, resolution and speed. It should be noted that no pre-imaging magnetisation was performed on the sample to align the magnetic domains and therefore enhance the response of the ferrite. A sufficient magnetic response was achieved with the specimen in the as-received condition. The AFM/MFM results were analysed using Gwyddion 2.30 [19] open source scanning probe microscopy analysis software (http://gwyddion.net/; Czech Metrology Institute, Brno, Czech Republic). Standard processing for MFM images involved three point levelling, data levelling by mean plane subtraction, line correction by comparison to height median and in some cases either horizontal scar removal or defect interpolation tools. Automated thresholding was based on height, and thus was used with the intersection merge mode.

In order to relocate the same areas between the two analysis techniques fiducial markers (square trenches) were prepared using focused gallium ion beam milling (FEI FIB201 Workstation). The dark rectangle to the right of several of the Figs. 5–7 shown in this paper is the FIB milled fiducial marker. The same ion beam system was used to mill the cross-sections as part of the depth of measurement investigations. Although conventionally a platinum strap would be used to preserve the top surface of the specimen, this was not adopted so that it would be easier to align the images with those obtained from other techniques. The platinum straps (1.5×7.5 μm^2 and 5×10 μm^2) utilised in the second part of this investigation were deposited with thicknesses ranging from 40 to 200 nm, using a FEI Helios NanoLab 600i Workstation.

3. Data processing and quantification

3.1. MFM signal response

EBSD signals are widely acknowledged as originating from the near surface of a specimen [20,21], with a depth resolution of 7.9 nm for Cu using a 40 nA beam with a 30 kV accelerating voltage [22] and a depth resolution for a AL-TRIP (mixed austenite and ferrite structure) steel estimated as 5–10 nm [23]. Although depth resolution of EBSD is dependent on the material density, as well as the electron beam current and accelerating voltage, a key factor is the channelling of electrons along lattice planes. Depending on the relative orientation of the crystal lattice and the incident beam, this can significantly increase the penetration, so that a depth resolution of up to 38 nm has been reported for Cu due to this contribution [22]. Where two grains are sampled by EBSD, diffraction occurs simultaneously leading to overlapping patterns. Where one pattern is brighter than the other, modern EBSD software is capable of distinguishing between the patterns.

Depth resolution in MFM is contributed by the amount of material that the stray magnetic field (especially the component perpendicular to the sample surface) from sub-surface ferrite grains can penetrate and still remain detectable by the MFM probe tip. When ferrite grains are small, they appear to contain a single magnetic domain. However for larger grains several domains can be present. If these are at, or close to the specimen surface, the ferrite grains would be expected to form flux closure domains [24,25]. These are magnetic domains that are orientated at right angles to the direction of the main magnetic domain within the grain. This allows the field lines to form closed loops [24–26] thus reducing the magnetostatic energy [24–26]. In situations where there are multiple neighbouring ferrite grains (and thus magnetic domains), it has been demonstrated that flux closure paths are achieved due to sufficient number of suitable domain orientations being present [24]. It has been noted experimentally [24] that flux closure domains often form less intense magnetic poles along the domain boundaries. As such, from a practical perspective, the flux closure domains reduce the magnitude of the stray field and thus the volume of material penetrated. It was recognised that despite this effect, MFM has the potential to sample a depth which exceeds that of EBSD, so that sub-surface ferrite may be detected. Hence, a measurable response to parts of 'sub-surface' ferrite grains inclined to the surface may be developed. Fig. 1 shows a schematic representation of a ferrite grain, lying at a shallow

Table 1
Chemical composition (wt%) of Type 321 stainless steel.

C	Si	Mn	S	P	Ni	Cr	Mo	N	Al	Cu	Nb	Ti	V	Ce	Fe
0.06	0.45	1.82	< 0.001	0.025	9.34	17.12	0.33	0.015	0.049	0.27	< 0.01	0.34	0.06	–	Bulk

Download English Version:

https://daneshyari.com/en/article/8038208

Download Persian Version:

https://daneshyari.com/article/8038208

Daneshyari.com