Ultramicroscopy 146 (2014) 17–26

Contents lists available at ScienceDirect

Ultramicroscopy

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

Determination of the easy axes of small ferromagnetic precipitates in a bulk material by combined magnetic force microscopy and electron backscatter diffraction techniques

L. Batista [a,*], U. Rabe [a,b], S. Hirsekorn [a]

[a] Fraunhofer Institute for Non-destructive Testing (IZFP), Campus E3 1, 66123 Saarbrücken, Germany
[b] University of the Saarland, LZPQ, 66123 Saarbrücken, Germany

ARTICLE INFO

Article history:
Received 5 March 2014
Received in revised form
7 May 2014
Accepted 9 May 2014
Available online 17 May 2014

Keywords:
Magnetic force microscopy (MFM)
Electron backscatter diffraction (EBSD)
Magnetic easy axis
Magnetic domains
Precipitate
Microstructure

ABSTRACT

A method to determine the magnetic easy axes of micro- and nanoscopic ferromagnetic precipitates embedded in a bulk material is proposed and applied to globular cementite (Fe_3C) embedded in a ferrite matrix. The method combines magnetic force microscopy (MFM) with electron backscattered diffraction (EBSD) measurements. Magnetic domain structures in globular and in lamellar cementite precipitates in unalloyed pearlitic steels were imaged using MFM. The domain structure of the precipitates was analyzed in dependency of their size, shape and crystallographic orientation. It was found that the magnetic moments of the cementite precipitates are highly geared to their crystalline axes. The combined MFM and EBSD studies allow the conclusion that the cementite easy direction of magnetization is the long [010] axis. For fine lamellae cementite the determination of their crystallographic orientations using electron diffraction techniques is very difficult. With the previous knowledge of the behavior of the domain structure in globular cementite, the crystalline orientations of the fine lamellae cementite can be estimated by simply observing the magnetic microstructures and the topographic profiles.

1. Introduction

Ferromagnetic materials exhibit intrinsic 'easy' and 'hard' magnetization directions, i.e. the energy required to magnetize a crystal depends on the direction of the applied field relative to the crystal axes. Magnetic anisotropy is an important property [1] and has therefore been exploited in the design of most magnetic materials of commercial importance, including e.g. grain-oriented electrical steels [2,3] or thin films for ultra-high density magnetic recording [4,5]. On the other hand, the magnetic micro- and nanostructure of the grains or phases of structural materials such as steels are of interest because the magnetic properties can be exploited for non-destructive testing.

The magnetic easy axis is usually found by measuring the magnetic anisotropy of single crystals using techniques like superconducting quantum interference device (SQUID) [6], torsion oscillating magnetometry (TOM) [7], alternating field gradient magnetometry (AFGM) [8], vibrating sample magnetometry (VSM) [9], or ferromagnetic resonance (FMR) [10]. The signal intensity of all these techniques is proportional to the total magnetic moment and hence to the volume of the sample. However, some ferromagnetic compounds, as e.g. Fe_3C, Fe_3Al, γ'-Fe_4N, are encountered as a second phase embedded in a polycrystalline matrix material. Techniques which are sensitive to the whole sample volume are not suitable to determine the magnetic easy axes of such phases separately. On the other hand, such second phases are usually not available as larger single crystal specimens that are pure, void-free, homogeneous, texture-free, and stoichiometric. Therefore techniques are required which provide local magnetic and crystallographic information in bulk materials.

The magneto-optic Kerr effect (MOKE) technique [11,12] is a magnetic domain imaging method in which the contrast is obtained by the interaction between magnetic fields and polarized light. In addition, the MOKE technique provides local magnetization curves, however, its spatial resolution is limited by the wavelength of the used laser (a few hundred nanometers) [13]. A much higher spatial resolution can be achieved using methods based on electron microscopy. Scanning electron microscopy with

* Corresponding authors.
E-mail address: leonardo.batista@izfp.fraunhofer.de (L. Batista).

http://dx.doi.org/10.1016/j.ultramic.2014.05.003
0304-3991/

polarization analysis (SEMPA) can directly detect the sample magnetization component with a spatial resolution of about 20 nm [14]. The major limitation in application of SEMPA is the fact that the measurements must be performed in ultra-high vacuum on a well prepared clean conducting surface. Lorentz electron microscopy (LEM) [15] is based on the deflection of electron beams caused by the Lorentz force in transmission electron microscopy. Using the LEM technique, Keh and Johnson [16] studied the domain structure on cementite thin foils of approximately 200 nm thickness. Even though modern aberration-corrected microscopes achieve spatial resolution in the order of 1 nm, the transmission electron microscopes are expensive, and the LEM technique is limited to thin foils which are transparent to the electrons. Especially in the case of multiphase materials, differences in the etching rates of the different phases impede smooth thin foil preparation.

In contrast to the techniques discussed above, magnetic force microscopy (MFM) [17–19] is suitable for measurements on thick (bulk) samples and can therefore be used to study micro- and nanoscopic embedded magnetic materials in a matrix by measuring simultaneously the topography and the magnetic microstructure with a relatively easy sample preparation. MFM is a scanning probe technique based on sensing the long-range magnetostatic interaction between the sample surface and a microfabricated tip with nm radius of curvature. A lateral resolution of about 10–20 nm can be achieved with optimized imaging conditions.

In this paper we examine the magnetic domains of micro- and nanoscopic ferromagnetic precipitates embedded in a bulk material using MFM. Local crystallographic information of the microstructure is provided by the electron backscatter diffraction (EBSD) technique, the recent developments of which allow a spatial resolution of about 10 nm [20]. The correlation between the magnetic and crystallographic microstructure is used to determine the magnetic easy axis of globular cementite precipitates in a ferrite matrix.

2. Experiments

Two different unalloyed steels were examined in this study, Fe–0.8% C containing lamellar cementite, and Fe–1.5% C containing globular cementite embedded in a ferrite matrix, respectively. Scanning electron microscopy (SEM) images (Fig. 1) show the microstructure of the samples. Lamellar pearlite composed of alternating plates of ferrite (soft α-Fe) and cementite (hard Fe_3C) is a typical microstructure of Fe–0.8% C (Fig. 1b). Globular cementite precipitates (Fig. 1a) were obtained by heat treating an unalloyed pearlitic Fe–1.5% C sample in vacuum at 720 °C during 20 h and slowly cooling at the rate of 10 °C/h in the furnace. The surfaces of all samples were mechanically polished and afterwards vacuum annealed at 600 °C for 4 h in order to remove residual stresses. Two micro-hardness indents were placed within the center region of the specimens as a reference to ensure that the EBSD and MFM measurements were taken in the same area. Directly before the measurements the specimens were demagnetized and etched using Nital (95% ethanol + 5% nitric acid). The demagnetization process was done by applying alternating fields of slowly decreasing amplitude.

Electron backscatter diffraction maps were obtained on a JEOL JSM-7000F scanning electron microscope (Jeol Ltd., Tokyo, Japan) equipped with an EDAX Trident EBSD analysis system (EDAX Inc., Mahwah, USA). An acceleration voltage of 15 kV and an emission current of around 100 mA were used for all scans. Data was recorded and analyzed using the EDAX/TSL OIM software package [20]. The step size was between 20 and 100 nm for all EBSD maps reported here.

Magnetic force microscopy measurements were performed under ambient conditions using a commercial MFM instrument (Nanoscope III® multimode, Bruker AXS Inc. (formerly Digital Instruments/Veeco), Madison, WI, USA). The topographic and magnetic images were obtained using the two-pass (Tapping/lift® mode) technique. Within the first pass the surface profile is recorded in the intermittent contact mode (tapping mode) [21]. Magnetic forces are mapped in the second pass whereas the magnetic sensor tip scans the previously measured topographic profile at an adjusted distance (lift-height) in the range of 10–100 nm above the surface. The cantilever is excited to forced vibration at a frequency close to its first flexural resonance. The gradient of the magnetic tip–sample interaction forces shifts the resonance frequency of the cantilever. As this frequency shift is usually small compared to the half-width of the resonance, the magnetic image is obtained by measuring the phase shift at the frequency of excitation as a function of position.

3. Results and discussion

3.1. Choice of a suitable MFM probe and general observations

It is well known that the contrast of MFM images depends on the imaging parameters and on the choice of the sensor tip, because the local tip–sample interaction forces are a result of the magnetic fields and moments of the tip and the sample [19]. During MFM imaging, the tip stray field may cause reversible and irreversible changes in the local magnetic state of the sample and vice-versa [22–24]. Therefore, it is very important to choose the appropriate kind of magnetic probe for each particular experiment.

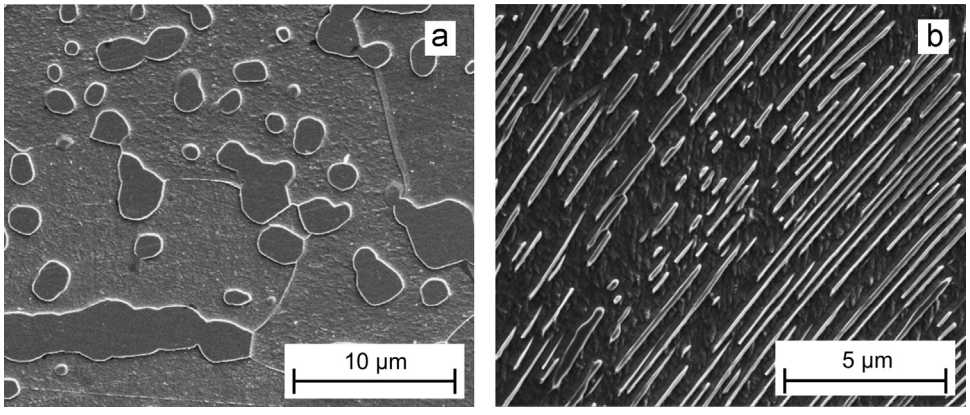

Fig. 1. SEM images showing the microstructure of the investigated unalloyed pearlitic steel samples: (a) Fe–1.5% C with globular cementite and (b) Fe–0.8% C with lamellar cementite.