Journal of Alloys and Compounds 404–406 (2005) 626–629

Journal of ALLOYS AND COMPOUNDS

www.elsevier.com/locate/jallcom

Magnetic properties of austeno-ferritic stainless steel after cathodic hydrogen charging

M.J. Wozniak*, A. Glowacka, J.A. Kozubowski

Warsaw University of Technology, Faculty of Materials Sciences and Engineering, Woloska 141, 02-507 Warsaw, Poland

Received 1 June 2004; received in revised form 24 November 2004; accepted 14 January 2005
Available online 18 July 2005

Abstract

The influence of cathodic hydrogen charging on the magnetic properties of austeno-ferritic stainless steel was investigated. The measurements using atomic force microscope working in magnetic force microscopy mode were performed. The changes of the magnetic properties of the steel were observed. The magnetic phase in the form of laths has been revealed.

Keywords: Metals; Magnetic measurements; Magnetic force microscopy; Atomic force microscopy

1. Introduction

AFM can measure not only topography but also physical properties such as: surface conductivity, static charge distribution, friction, magnetic fields, elastic moduli and hardness. Magnetic fields measurements using AFM technique is called magnetic force microscopy (MFM). The hydrogen charging induced phase transformation in austeno-ferritic duplex stainless steel were investigated using MFM mode.

2. Magnetic contrast generation

The components of atomic force microscope are shown on Fig. 1.

The position of the probe is usually measured with a laser photodiode and photodetector (Figs. 1 and 2). Two types of images are collected simultaneously during MFM measurements. A first scan line is collected in Tapping Mode (main scan) (Fig. 2a), whereas a second line is collected in Lift Mode (interleave scan) (Fig. 2b).

During the main scan performed in Tapping Mode [1] (intermittent contact mode) the topographic data are collected. The tip of the microscope is sensitive to a short range forces such as van der Waals forces [2]. The movement of the tip is controlled by feedback loop system (Fig. 1). During the interleave scan performed in Lift Mode [3] (non contact mode of AFM) the feedback system which controls movement of the tip (Fig. 1) is turned off and the probe is lifted to preselected height above the surface of the sample which is called the lift scan height (LSH) (Fig. 2b). The LSH can be changed from few nanometers to few hundreds nanometers. In the LSH range 30–300 nm the tip is more sensitive to far field forces such as magnetic or electrostatic forces than to short range molecular forces [2]. In response to a magnetic field coming from the sample surface, the magnetic field of the tip causes the cantilever (Fig. 1) bending upwards and downwards (only the cantilever not the whole probe) (Fig. 3). The whole probe still is lifted to lift scan height (Fig. 2). In the magnetic force microscopy (working in Lift Mode) the influence of magnetic forces is monitored by observing changes in resonance frequency of the cantilever. In the absence of magnetic forces, the cantilever of the tip, has a resonant frequency f_0. Magnetic forces cause a shift Δf in the resonant frequency, which is proportional to a force gradient, difference between negative

* Corresponding author. Tel: +48 22 660 8745; fax: +48 22 660 8514.
E-mail address: wozmi@inmat.pw.edu.pl (M.J. Wozniak).

0925-8388/$ – see front matter © 2005 Elsevier B.V. All rights reserved.
doi:10.1016/j.jallcom.2005.01.123

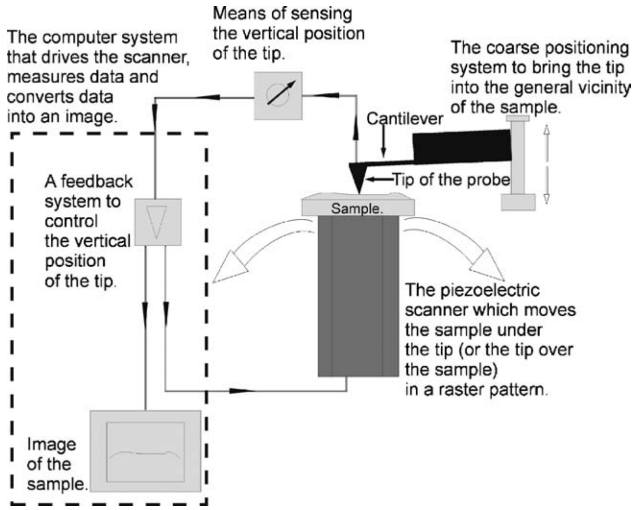

Fig. 1. Scheme of the atomic force microscope.

and positive magnetic force acting on the tip [3]. The attractive magnetic forces cause the negative frequency shifts whereas the repulsive magnetic forces cause the positive frequency shifts. The shift in the resonant frequency is transferred by the microscope software into an image of the magnetic interaction. The tips used for MFM are coated with a ferromagnetic thin film. The resolution of magnetic image obtained with commercially available tips is about 50 nm [5].

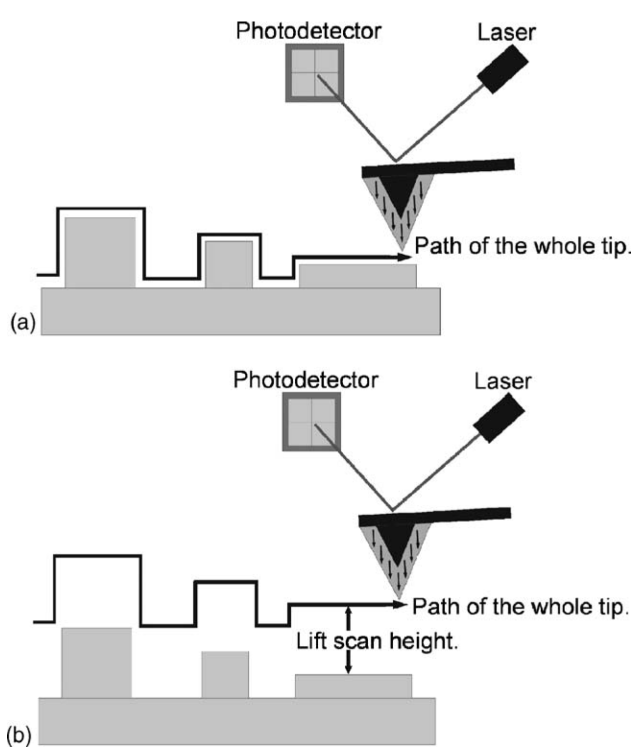

Fig. 2. (a) Main scan, when the topographic data are collected. (b) Interleave scan, when the magnetic interaction data are collected.

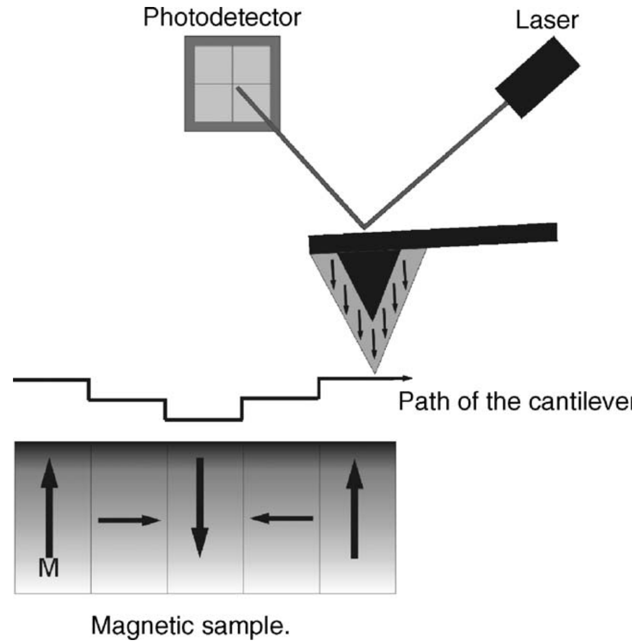

Fig. 3. Interaction between the magnetic tip and magnetic sample.

3. Experimental

The digital instruments multi mode nanoscope IIIa operating in intermitten contact mode (Tapping Mode) and non contact mode (Lift Mode) was used to investigate the topographic and magnetic features of samples of austenitic-ferritic duplex stainless steel before and after cathodic hydrogen charging. The MESP-HM Veeco silicon probes, covered with a thin layer of the cobalt alloy 150 nm in thickness, were employed for the magnetic characterization of the material. The resonance frequency of these probes is in the range of 60–100 kHz. The cantilever length is 225 μm and nominal tip radius of curvature 25–50 nm. Before each sets of measurements the probe was magnetized by a strong permanent magnet, with the field aligned along the tip axis (direction perpendicular to the sample surface), and the standard magnetic sample (magnetic recording tape) from Digital Instruments was imaged to ensure the status of the magnetic tip. The lift scan height of the probe during the interleave scan was set equal to 100 nm. The material used in the current study was Cr23–Ni5–Mo3 austenitic-ferritic duplex stainless steel (0.026C, 1.57Mn, 5.43Ni, 22.94Cr, 2.75Mo wt.%). The investigated steel contains the same volume fractions of austenite and ferrite phases. Samples in the form of bars (8 mm in diameter) were annealed at 1000 °C in an argon atmosphere for 1 h and subsequently quenched in water. Then, the samples were machined to reduce their diameter to 3 mm and next sliced to 0,2 mm thick discs using a wire saw. Finally, they were polished to thin foils by two-sided electrolytic thinning. Hydrogen charging of the thin foils was carried out electrolytically at room temperature, during 2 h time, in a 0,1 M H_2SO_4 aqueous solution with an addition of 10 mg/l

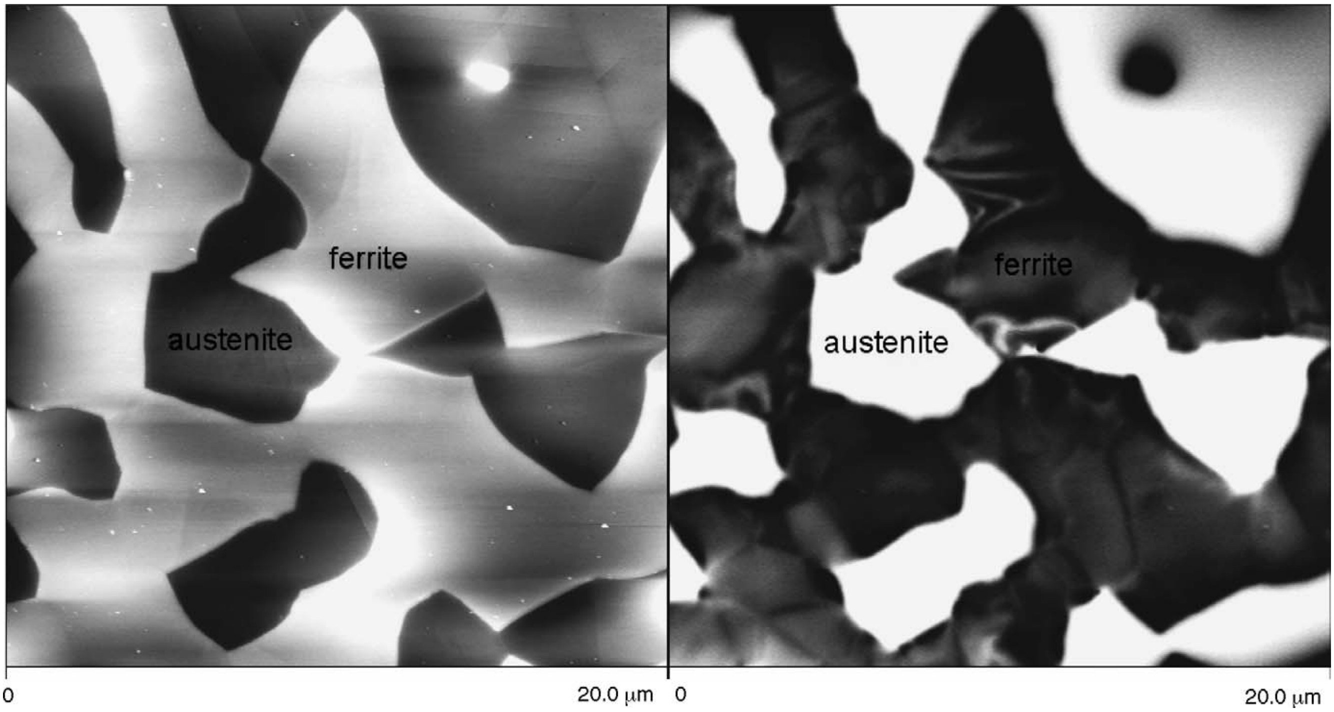

Fig. 4. Initial duplex steel microstructure before hydrogen charging. Topographic (left side) and magnetic image (right side).

of a hydrogen entry promoter (As_2O_3). A current density of 20 mA/cm^2 was applied between the specimen and a platinum anode.

4. Results and discussion

Hydrogen charging results in changing of the mechanical properties of steel by transformation of the microstructure similar to the changes induced by cold working [6]. In austenitic steels the phase transformations: $\gamma \to \alpha$' and $\gamma \to \varepsilon$ were observed. The γ phase (austenite) is a paramagnetic, face center cubic (fcc), the α-ferrite is a ferromagnetic, body center cubic (bcc) phase, the α' martensite is a ferromagnetic, body center cubic (bcc) phase, whereas ε martensite is a paramagnetic, hexagonal close packed (hcp) phase. The different magnetic properties of austenite, ferrite and both martensites phases are the source of the magnetic contrast, which gives a possibility of using magnetic force microscopy (MFM) for investigation of phases transformations in this kind of steel. Fig. 4 shows the initial microstructure, before hydrogen charging. Both phases ferrite and austenite are clearly visible at the topographic image (left side of the picture) and the magnetic image (right side of the picture). In the topographic image the γ grains are concave because γ phase was etched during the sample preparation. In the magnetic image the magnetic domain structure grains of the α phase is clearly visible because α phase is a ferromagnetic. The grains of ferrite exert an attractive magnetic forces—dark region on the magnetic image (negative frequency shifts). The paramagnetic γ phase does not give any changes in the magnetic contrast [2].

Fig. 5 shows changes in the structure of the investigated steel caused by cathodic hydrogen charging process. In comparison to the initial state, both ferrite and austenite phases have changed. Inside grains of the paramagnetic austenite phase there appear new magnetic laths which are clearly visible in the magnetic interaction image (right side of the Fig. 5). These magnetic laths exert an attractive magnetic forces (negative frequency shifts or dark region on magnetic image). The magnetic character of the phase which appeared in austenite, and our further works [6,7] allow us to conclude that is the ferromagnetic martensite α' phase . The magnetic domain structure of the ferrite phase has changed (Fig. 5 right side). It is now much more fine in comparison with initial structure, what can be induced by hydrogen effects [6–9].

5. Conclusion

The hydrogen induced phase transformation in Cr23-Ni5-Mo3 austenitic-ferritic duplex stainless steel were observed. In grains of the austenite phase, laths of the α' martensite have appeared. The similar use of the AFM (Tapping Mode) and MFM (magnetic force microscopy, Lift Mode) gives a unique chance to observe changes in topographic features and magnetic domain structure, on the same area, simultaneously. The MFM technique provided insights into changes of structure of duplex steel, which have been hardly possible from other technique.

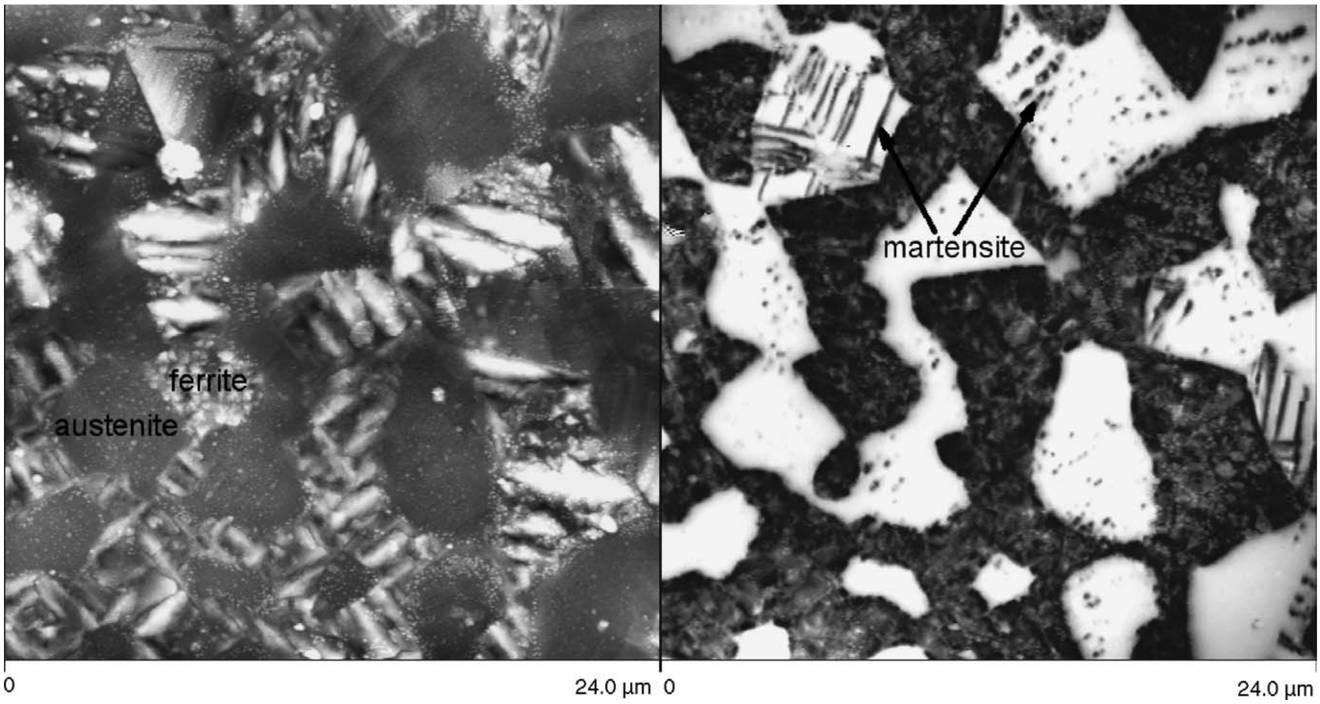

Fig. 5. Duplex steel microstructure after hydrogen charging.

Acknowledgment

Authors wish to thank Polish Committee of Scientific Researches (KBN) and Warsaw University of Technology for supporting this work.

References

[1] X. Chen, M.C. Davies, C.J. Roberts, Ultramicroscopy 75 (1998) 171–181.
[2] A. Dias, M.S. Andrade, Appl. Surf. Sci. 161 (2000) 109–114.
[3] Multi Mode SPM Instruction Manual version 4.31ce, Digital Instruments Veeco Metrology Group 1996–1999.
[4] B.R.A. Neves, M.S. Andrade, Appl. Phys. Lett. 74 (1999) 2090.
[5] J.M. Garcia, A. Thiaville, J. Miltat, J. Magn. Magn. Mater. 249 (2002) 163–169.
[6] A. Glowacka, W.A. Swiatnicki, E. Jezierska, in: Conf. Proc.: XII International Conference EMS'2005, Kazimierz, Poland, 2005.
[7] A. Glowacka, M.J. Wozniak, W.A. Swiatnicki, J. Alloys Compd., in press.
[8] A. Glowacka, W.A. Swiatnicki, Mater. Chem. Phys. 81 (2003) 496.
[9] A. Glowacka, W.A. Swiatnicki, J. Alloys Compd. 356-357 (2003).