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Ferrite and austenite phase identification in duplex stainless steel using SPM techniques

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ABSTRACT

It can be challenging to properly identify the phases in electro-polished duplex stainless steel using optical microscopy or other characterization techniques. This letter describes magnetic force microscopy to properly identify the phases in electropolished duplex stainless steel. The results are also confirmed with the current sensing atomic force and scanning Kelvin probe force microscopy. The difference in topography heights between the ferrite and austenite phases is attributed to the different etching rates during electropolishing, although these phases have different mechanical properties. The current in the austenite is much higher compared with the ferrite, thus current sensing atomic force microscopy can also be used to properly identify the phases.

1. Introduction

Duplex stainless steels offer both high mechanical strength and superior corrosion resistance, attributed to the microstructure consisting of the ferrite (α) and the austenite (γ) phases [1–3]. However, it is hard to correctly identify the two phases. The common methods for characterization of duplex stainless steel are quantitative metallography and microhardness measurements [4]. Traditionally, etching has been used for identification and characterization of the ferrite and austenite phases in duplex stainless steel, because the etching process leads to a slight preferential dissolution of a certain phase, namely the topography height difference between the ferrite and austenite appears after etching (Fig. 1a and b). However, it is difficult to identify the corresponding phase in etched duplex stainless steel, and thus the ferrite and austenite phases are occasionally mislabeled. In order to unequivocally identify the two phases, scanning probe microscopy (SPM)-based technique, namely, magnetic force microscopy (MFM), has been used, since MFM can distinguish ferromagnetic ferrite from paramagnetic austenite with high spatial resolution [5–10]. The present work aims to properly distinguish and identify the ferrite and austenite phases present in duplex stainless steel using various SPM techniques. These SPM-bashed techniques are MFM, scanning Kelvin probe microscopy (SKPFM) and current-sensing atomic force microscopy (CSAFM) measurements.

2. Experiment

The material used for experiments is conventional 2507 duplex stainless steel, described in previous reports [11–14]. The specimens were wet ground with SiC paper up to 2000 grit, and then mechanically polished with diamond paste to 1.5 μm. Once the mechanical polishing was finished, the final electrochemical polishing was performed in a mixed solution of HNO_3:H_2O = 1:1 for 20 s at 1.2 V applied voltage. This allowed distinguishing the ferrite and austenite phases. The specimens were ultrasonically cleaned in ethanol and dried in N_2 gas flow.

MFM and SKPFM measurements were carried out before electrochemical polishing using a dimension Nanoscope V, from Bruker Instruments Inc. All measurements were conducted in air at room temperature. After electrochemical polishing, specimens were kept for 24 h in air, allowing the formation of a native passive layer. Then CSAFM measurements were conducted on the specimen with air-formed film using Agilent 5500 AFM (Agilent Technologies, USA) operated in the current sensing mode. Nanosensor PPP-MFMR hard magnetic material coated probes were used in the MFM measurements (2.8 N/m force constant, 45–115 kHz resonant frequency), while DPE14/AIBS conductive Pt-coated silicon tips (5.7 N/m force constant and 50 nm tip radius) were used for the SKPFM and CSAFM measurements. Nanoindentation tests were performed with the Hysitron Triboindenter equipped with the Berkovich indenter top to measure the nanohardness of the two phases.

3. Results and discussion

Fig. 1c shows the MFM image of the duplex stainless steel specimen, where the magnetic domains distribution can be clearly observed. The ferrite matrix has a striped appearance in the MFM image due to the presence of magnetic domains, surrounding the austenite phase, which appears uniform due to its paramagnetic properties. Hence, MFM images can properly distinguish and identify corresponding phases in duplex stainless steel.

With the aid of the MFM image, the ferrite and austenite phases can also be easily identified in the SKPFM image of the same region (Fig. 1d) as the MFM image shown in Fig. 1c. Ferrite matrix exhibits lower Volta potential (darker color), while austenite has a higher potential (lighter color), which is in good agreement with previous reports on SKPFM measurements

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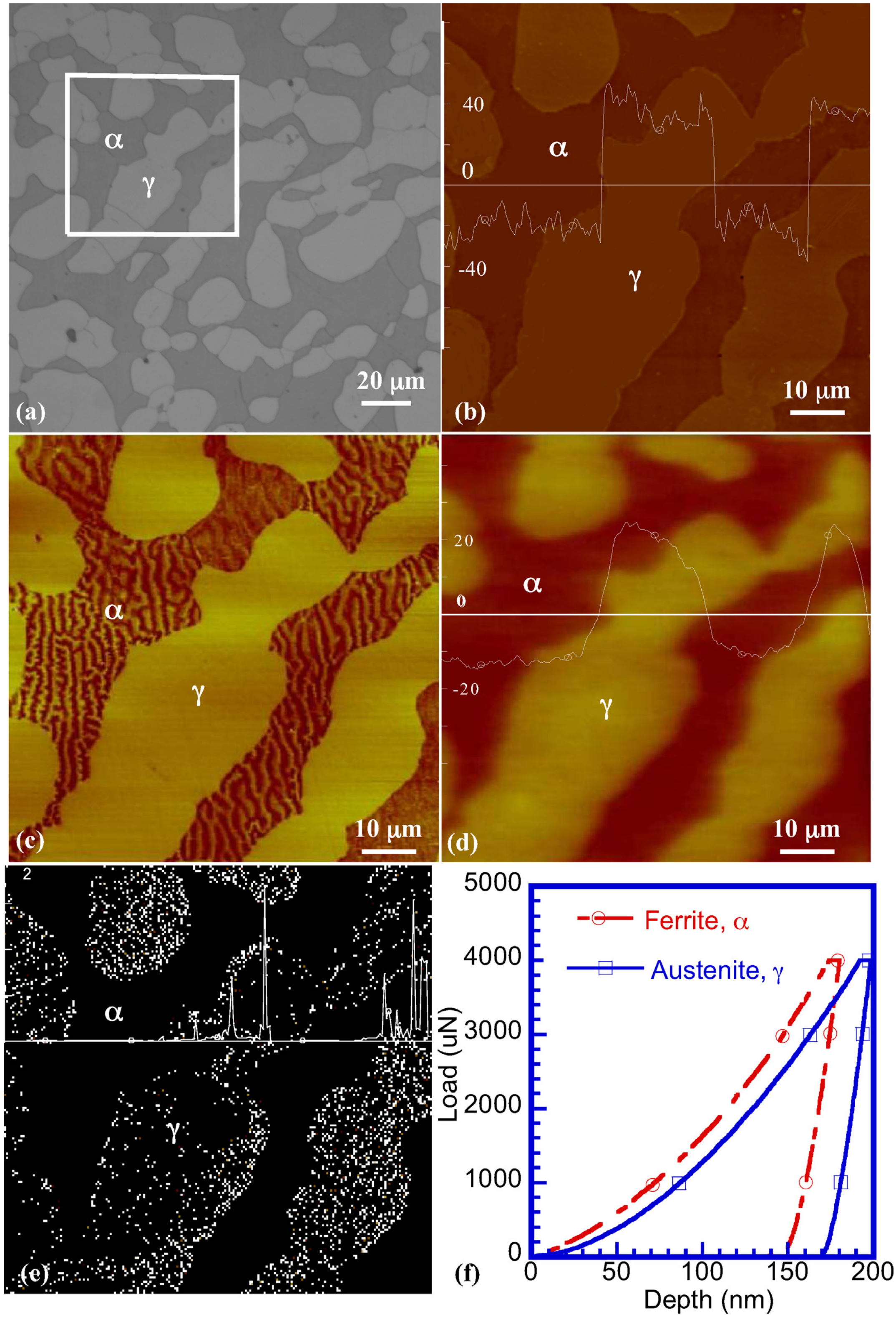

Fig. 1. (a) Optical image of electrochemically etched duplex stainless steel surface; (b) AFM topography with the line profile in nm of the same surface area marked in (a); (c) MFM image on the same area as (b); (d) SKPFM map with Volta potential profile line; (e) CSAFM current map with the current profile in A, and (f) nanoindentation load-displacement curves obtained from the ferrite and austenite phases.

of duplex stainless steel [5–13]. This may be a result of high of nickel content in austenite, which has higher Volta potential, compared with ferrite enriched with chromium [7,12]. Once MFM data has been obtained, ferrite matrix and austenite phase can be easily identified in electropolished samples from the optical micrographs and the AFM topography by comparing MFM, optical, AFM and SKPFM images of the same area, shown in Fig. 1. AFM topography with the line profile (Fig. 1b) of the same surface area, marked in Fig. 1a, shows that the ferrite matrix is about 50 nm lower (darker color) than the austenite (brighter color), indicating that the preferential dissolution occurs in the ferrite phase during the etching process, namely the ferrite is more chemically active than austenite in the HNO_3 etching solution. These results are also consistent with the SKPFM measurements (Fig. 1d), and previous reports on the corrosion behavior of duplex stainless steels [4–8,12].

Moreover, CSAFM current map with the line profile indicates that the air-formed passive film on the austenite has a higher current than on the ferrite, as seen in Fig. 1e. This is because ferrite has more chromium than austenite, which is more easily passivated, and thus the chromium oxide is undoubtedly preferentially formed on the ferrite surface in air [15]. However, the obtained results are different compared with the Souier et al.'s work [16], in which the ferrite topography is reported higher than austenite and the ferrite current is higher than the austenite. This was explained by the hardness difference of the two phases, i.e. the ferrite being harder than the austenite [16]. The nanoindentation measurements reveal that the hardness of the ferrite is higher than that of austenite, which is obvious from the load–displacement curves shown in Fig. 1f. The average hardness value obtained by nanoindentation for ferrite is 4.41 ± 0.44 GPa and 3.57 ± 0.52 GPa for austenite, measured at nine different locations for each phase. This result is in agreement with the previous report that the ferrite is harder than austenite [17].

There is little detail is given in Ref. [16] about the sample chemical mechanical polishing: "final chemo-mechanical polishing was performed using colloidal silica particles in a basic solution (pH 9, particle average diameter: 40 nm)" [16]. Although most likely, we used different chemical etching solution than Souier et al. [16], it does not change the fact that the austenite current is much higher than ferrite. Thus, it is believed that the phases in reference [16] might be mislabeled. The current-sensing atomic force microscopy can be used to properly identify the phases, since the austenite phase always has higher current compared with the ferrite phase for the same tip bias.

4. Conclusions

MFM was used to properly distinguish and identify the ferrite and austenite phases in electropolished duplex stainless steel. Matrix ferrite and austenite phase are easily identified in the MFM image, where ferromagnetic ferrite shows a striped appearance due to the presence of magnetic domains, while paramagnetic austenite exhibits a uniform non-magnetic structure. These results were confirmed with SKPFM, CSAFM and nanoindentation measurements. The difference in topography heights between the ferrite and austenite phases is attributed to the different etching rates during electropolishing, although there is difference in the hardness values of the two phases measured by nanoindentation. CSAFM can also be used to properly identify the phases, since the austenite current is always much higher than the ferrite current.

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