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Electrical and mechanical characterization of two-phase alloys by means of scanning probe microscopy in dynamic impedance spectroscopy mode

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ABSTRACT

Dynamic impedance spectroscopy, evaluated for measuring non-stationary systems, was used in combination with scanning probe microscope. Using this approach, localized impedance measurements in the AFM contact mode could be carried out. Additionally, impedance–force curves were made at each phase of investigated materials to illustrate the relation between impedance and the force applied to a probe. Therefore, correlation of electrical and mechanical properties with particular phases of investigated two-phase alloys was made possible. The materials used in this study were spheroidal graphite cast iron and 2205 duplex stainless steel, both materials with clearly defined phases having significantly different properties.

1. Introduction

A.C. impedance techniques are commonly used in many research areas. Generally they are employed to characterize macroscopic surfaces or bulk properties of different materials. Thus, obtained results are averaged over the area or volume of an examined sample. In order to register localized impedance spectra on different grains, phases and inclusions on the surface, important developments have been made. Two main approaches are available: a micro-cell technique [1–5] and scanning probe microscopy (SPM) [6–9]. The micro-cell technique is well-established and used for *in-situ* examinations of metals in electrolyte. On the other hand, impedance measurements performed by means of scanning probe microscopy are mainly *ex-situ* evaluations. Therefore, impedance measured with micro-cell is of electrochemical kind, whereas SPM-based impedance is of electrical type.

The time required to register the full impedance spectrum is relatively long, since in both techniques impedance spectra are made point by point [10–15]. If the localized impedance measurements are required, single frequency is utilized in order to minimize the time of scanning [9,16,7]. The probe moves across the surface while the time at each point needs to be relatively short. Unfortunately, the single frequency measurement is less informative than the one, where full impedance spectrum is obtained. To make a compromise between full spectra and single frequency measurements, dynamic impedance spectroscopy (DIS) technique was proposed and applied to atomic force microscopy [17]. In DIS technique, the voltage perturbation signal consists of several frequencies, which are generated simultaneously. The response signal is registered and decomposed to individual components with Short Time Fourier Transformation (STFT). Details of dynamic impedance spectroscopy methodology, which originally applied to electrochemical phenomena, are described in a series of publications [18–21]. Due to the advantage of performing analysis under non-stationary conditions, DIS has already prove its worth in a variety of electrochemical and corrosion studies [22–26].

Evolution of the scanning probe microscopy (SPM) techniques, including atomic force microscopy (AFM), made it possible to investigate certain aspects of materials characterization, which had been troublesome earlier [27–31]. Further scientific development allow to combine the AFM with the localized impedance spectroscopy measurements [32–36]. Thus, it was possible to investigate electrochemical and corrosion phenomena in detail [37,38].

The aim of this work involves differentiation between mechanical and electrical properties of two-phase alloys with significantly various particular phases by means of dynamic impedance spectroscopy in the AFM contact mode. Therefore, it was possible to

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register relative localized impedance changes along with changeable force applied to the probe. Thus, electrical and mechanical characterization of two-phase alloys could be discussed.

2. Materials and methods

Spheroidal graphite cast iron and 2205 duplex stainless steel were investigated as the classical diphase alloys with phases of different mechanical and electrical properties. Moreover, these materials were chosen for their significantly different combination of phases: ferrite (interstitial solid solution of carbon in Fe-α) and graphite concerning spheroidal cast iron, and ferrite and austenite (interstitial solid solution of carbon in Fe-γ) regarding 2205 duplex stainless steel.

The specimens were ground on fine-grade silicon carbide abrasive paper up to 2500 grade. Then, they were polished with commercially available polishing paste, dedicated to steels and finally cleaned and degreased with acetone in ultrasonic bath.

The DIS was used in this investigation for the localized impedance measurements, as well as to conduct impedance–force curves at spheroidal graphite and the ferrite matrix in the case of spheroidal cast iron, and at ferrite and austenite respectively in the case of 2205 stainless steel. Description of the whole experimental system, equipment used and principles of measurement can be found elsewhere [39]. Fig. 1 presents a scheme of employed measurement set-up.

An advantage of the DIS over a conventional impedance measurement, where perturbation signal changes frequency by frequency, is that more periods of each frequency are being registered. The more of full periods of each frequency are registered, the more precisely the impedance could be measured. For example, the time required to obtain a spectrum at 15 frequencies ranging from 70 Hz to 4.01 kHz is only 0.1 s for the DIS. Seven periods of the lowest frequency are registered at this time and the higher frequency, the more periods are registered. A conventional way of generation and acquisition of only seven periods of each of 15 frequencies would take approximately 0.32 s.

Registered topography images were 60 × 60 μm in size in the case of spheroidal graphite cast iron and 40 × 40 μm in size in the case of 2205 duplex stainless steel. Impedance–force curves were registered at 16 points at each phase of the examined two-phase alloys.

3. Results

Topography images of the spheroidal graphite cast iron and 2205 duplex stainless steel registered in the contact mode of the AFM are presented in Figs. 2 and 3 respectively. Grids visible on each depicted AFM topography image indicate the investigated surface of particular phase, where impedance–force measurements were carried out in the AFM contact mode.

Exemplary localized impedance spectra representing individual points of grids depicted in Fig. 2 (topography image of the spheroidal graphite cast iron) and Fig. 3 (topography image of 2205 duplex stainless steel) are presented in Figs. 4 and 5. It should be emphasized that the impedance spectra depicted in Fig. 4a represents the localized impedance measurements conducted at the graphite spheroid, while the spectra presented in Fig. 4b relate to the ferrite matrix of the investigated cast iron. Furthermore, the impedance spectra illustrated in Fig. 5a represents the localized impedance measurements conducted at the austenite phase, while the spectra presented in Fig. 5b relate to the ferrite phase of the investigated duplex stainless steel. It should be also emphasized that recorded localized impedance changes are strictly of relative character.

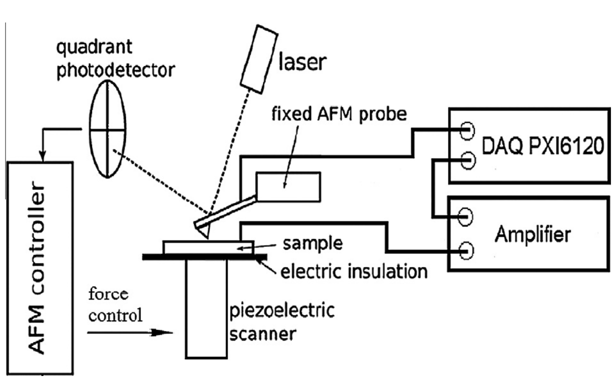

Fig. 1. Scheme of applied set-up used to dynamic impedance spectroscopy measurements in the AFM contact mode.

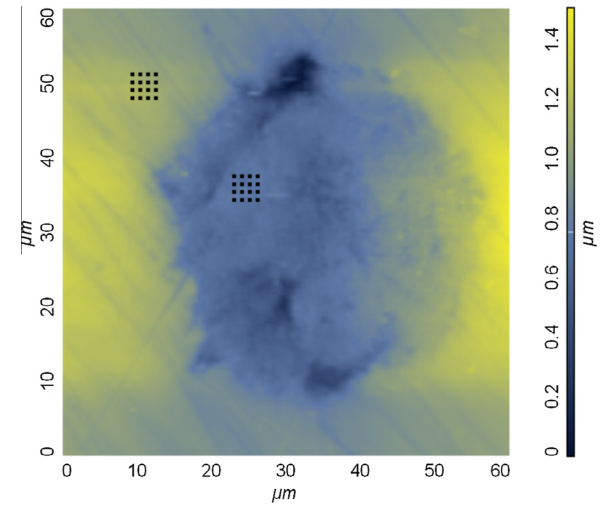

Fig. 2. Topography image of 60 × 60 μm area for spheroidal graphite cast iron. Grids indicate the investigated surface of particular phase for which examples of impedance spectra are presented in Fig. 4.

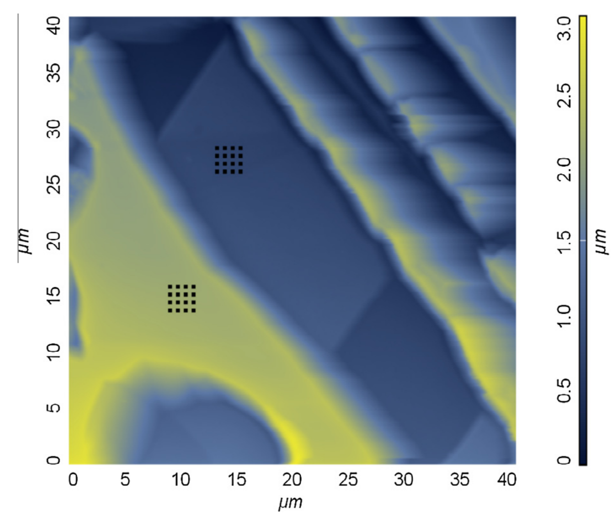

Fig. 3. Topography image of 40 × 40 μm area for 2205 duplex stainless steel. Grids indicate the investigated surface of particular phase for which examples of impedance spectra are presented in Fig. 5.

The impedance–force curves were performed 16 times at several graphite spheroids and 16 times at the ferrite matrix. The same procedure was performed at austenite and ferrite phases of 2205 duplex stainless steel. Each of 16 curves recorded for graphite and for the matrix, as well as each of 16 curves registered for the austenite phase and the ferrite phase, was fitted to a linear equation and respective averages were made. Average curves and their equations for both phases of the spheroidal graphite cast iron and 2205 duplex stainless steel are presented in Figs. 6 and 7 correspondingly.

4. Discussion

Graphite spheroid can be observed in the middle of the topography image presented in Fig. 2 among ferrite matrix. On the other hand, Fig. 3 illustrates the AFM topography image of the austenite phase (yellow[1]) and the ferrite phase (dark blue).

[1] For interpretation of color in Fig. 3, the reader is referred to the web version of this article.