



# Quantitative assessment of local misorientations and pitting corrosion behaviour of pearlitic steel using electron backscattered diffraction and microcapillary techniques



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## ARTICLE INFO

### Article history:

Received 12 May 2015

Received in revised form 31 July 2015

Accepted 2 August 2015

Available online 5 August 2015

### Keywords:

Misorientation

Pearlitic steel

Corrosion

EBSD

Local electrochemistry

## ABSTRACT

The influence of the grain tolerance angle (GTA) on the microstructural parameters of pearlitic steel is investigated using electron backscattered diffraction. The corrosion behavior of this alloy is then evaluated from local polarisation curves (electrochemical microcell technique) and potentiostatic pulse testing. These results are analyzed according to the specimen microstructure (grain boundaries and grain orientation spread, GOS). It is shown that there is a critical value of the GOS (around  $10^\circ$ ) leading to significant decrease of the pitting potential. The influence of the GTA value on the analysis of electrochemical results is also discussed.

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## 1. Introduction

Several studies adopting a multi-physics approach at the microscale reveal that in the absence of metallurgical heterogeneities, gradients in surface properties play an important role in pit initiation and propagation. It was demonstrated [1] that stress gradients calculated using a thermal-mechanical simulation are the key-parameter in pit initiation along the austenite/ferrite interface of duplex stainless steels. Pit initiation was evaluated from local corrosion tests (using microcapillary-based techniques) under potentiostatic control. After long-term aging in sodium chloride solution, it was shown [2] that scattering in the pitting potential values is connected with the heterogeneous distribution of chloride adsorbed in the passive film. This study was conducted by coupling microcapillary-based techniques and secondary ion mass spectroscopy. It was also revealed that this distribution is related to the residual stress field generated in grains during surface preparation. The surface residual stress field was measured by means of micro-X-ray diffraction technique.

The role of grain boundary characteristics and crystallographic texture on stress corrosion cracking and fatigue corrosion cracking mechanisms was widely investigated using electron backscattered diffraction [3–8]. By contrast, only a few papers concern pitting corrosion of metallic alloys. The influence of grain stored energy on the intergranular corrosion in AA2024-T351 was investigated in [9]. The influence of the grain orientation spread (GOS) on the pitting corrosion in duplex stainless steels [10] and in martensitic stainless steels [11] was also studied. In these cases, crystallographic orientation gradients were assessed from electron backscattered diffraction measurements at the microscale. To our knowledge, no other paper reports the use of electron backscattered diffraction to establish relationships between gradients in surface properties and pitting corrosion in metallic alloys. However, electron backscattered diffraction is a powerful and non-destructive technique that can provide useful information.

The definition of grains in electron backscattered diffraction is different from the conventional metallographic techniques [12]. The algorithm used to construct grains enforces that any two neighboring points of the electron backscattered diffraction scan which differ in orientation less than a specified grain tolerance angle (GTA) value belong to the same “grain” [13]. Thus, the choice of GTA has a significant effect on the obtained microstructure (grain size and morphology). The choice of GTA can also affect the GOS and the

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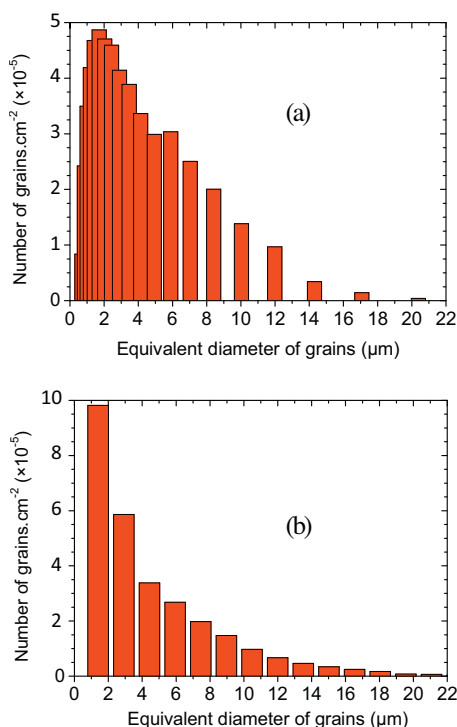


Fig. 1. Grain size distribution calculated for (a) GTA = 5° and (b) GTA = 15°.

kernel average misorientation values. These parameters are key-parameters to understand pit initiation and propagation in metallic alloys [10,11]. As GTA gets larger, grains delimited by low angle boundaries are encompassed with the larger deformed grains, and the spread of orientation in these larger grains (GOS) increases [14].

In the present paper, the influence of the GTA value on the microstructural parameters of pearlitic steel is first investigated using electron backscattered diffraction. The corrosion behavior of this alloy is then discussed according to the obtained GOS values. The corrosion behavior is determined from local polarisation curves plotted using the electrochemical microcell technique and very small capillaries (diameter of 20 μm) and potentiostatic pulse testing (PPT).

## 2. Experimental procedure

Experiments were carried out on pearlitic steel rods. The chemical composition of this alloy (wt%) is: 0.69–0.73% C, <0.02% S, <70 ppm N, <0.02% P, 0.1–0.3% Si, 0.4–0.6% Mn, <0.05% Cr, <0.01% Mo, <0.05% Cu, <0.01% Al, bal. Fe. Samples were cut from rods and embedded in an epoxy resin. They were ground using emery papers (4000 grade) and polished with diamond pastes (down to 1 μm). Specific vibratory polishing using the VibroMet 2 vibratory polisher from was then carried out to remove the cold-worked layer formed during mechanical polishing with diamond pastes.

The grain orientation was determined using a field-emission scanning electron microscope (FE-SEM, JEOL JSM-7600F) coupled with the TSL, EDAX, OIM, XM4 electron backscattered diffraction system. This microscope operated at 20 kV and a probe current of 3 nA. The working distance was set between 17 and 23 mm. Electron backscattered diffraction measurements were done on a surface area of 450 × 380 μm<sup>2</sup> with a step of 0.25 μm and an integration time of 23 ms per point.

Local electrochemical investigations were performed using the electrochemical microcell technique [15–17] and capillaries with a diameter of 20 μm. The pitting potential values determined with

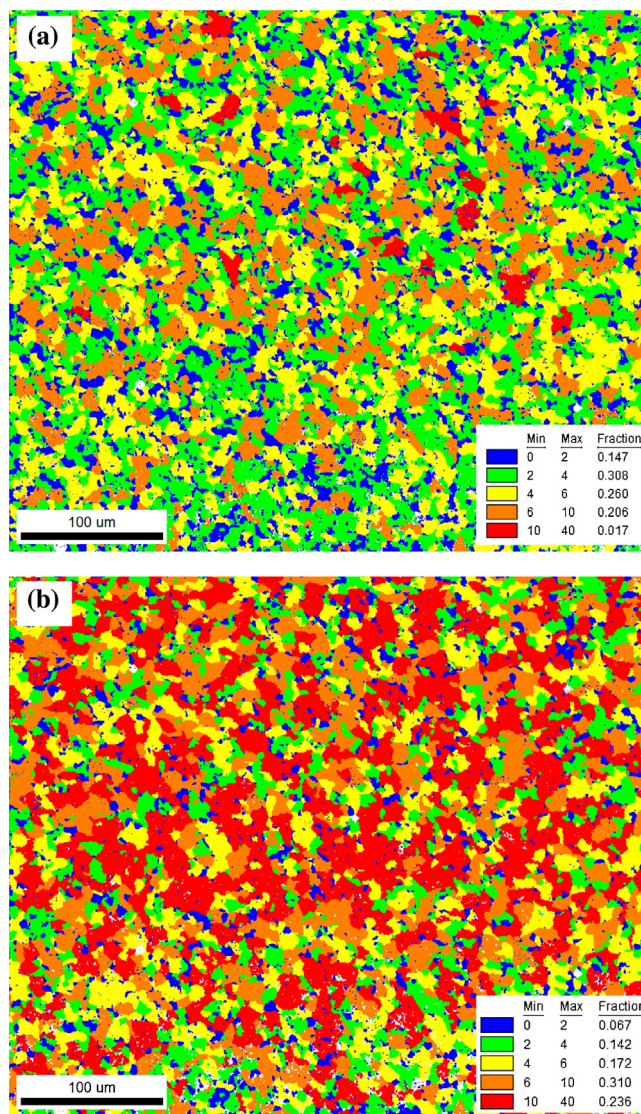


Fig. 2. GOS maps calculated for (a) GTA = 5° and (b) GTA = 15°.

such capillaries can be compared to those derived from classical measurements at the macroscale. Indeed, it has been shown [18] that the capillary geometry affects the electrochemical response (concentrations of species, potential) only when the pit radius is greater than the capillary radius/20. Therefore, in this study, the capillary geometry has influence on the electrochemical response when the pit radius is greater than 1 μm (i.e., not at the pitting potential where stable pits initiate). On the other hand, in the cathodic domain, the diffusive flux of species in the microcell is systematically greater than in the classical three-electrode cell, resulting in higher current densities using the microcell [19,20]. Therefore, comparing the cathodic branch of the polarisation curves derived from local and global scales is difficult. Comparing the corrosion potential values (where both anodic and cathodic reactions occur) at both scales is also difficult. Local electrochemical experiments were performed in 0.05 M NaCl (aerated solution) at 25 °C using a PGSTAT128 AUTOLAB potentiostat/galvanostat. All potentials were measured vs. Ag/AgCl (3 M KCl solution) and the counter electrode was made of platinum wire. Local polarisation curves were plotted at 1 mV s<sup>-1</sup>. The potential range was between –500 and –100 mV vs. Ag/AgCl. No prior polarisation was applied to the system.

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