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### Short communication

## Electrochemical behavior and compositions of passive films formed on the constituent phases of duplex stainless steel without coupling

## Yi Wang, Xuequn Cheng \*, Xiaogang Li

Corrosion and Protection Center, University of Science and Technology Beijing, Beijing 100083, China

#### ARTICLE INFO

#### ABSTRACT

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Keywords: Passive film Duplex stainless steel Cyclic volammetry XPS Selective dissolution The electrochemical behavior and compositions of passive films formed on the constituent phases ( $\alpha$ -phase,  $\gamma$ -phase) of 2205 duplex stainless steel (DSS) without coupling in neutral 3.5% NaCl solution were investigated, utilizing single-phase samples prepared by selective dissolution. The results demonstrate that, compared to  $\gamma$ -phase, Fe species in passive film of  $\alpha$ -phase are more stable due to the higher content of Mo within  $\alpha$ -phase. The superior stability of Cr (III) species in the passive layer of  $\gamma$ -phase may result from the higher content of Ni in  $\gamma$ -phase, which leads to the higher content of Cr, Cr/Fe ratio, and better passive layer. The beneficial effect of coupling of two phases on the passive behavior of 2205 DSS is also found.

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#### 1. Introduction

The properties of passive film provide the key to the high resistance of stainless steels to corrosion [1]. For duplex stainless steel, a heterogeneous passive film is formed on both phases [2] due to the differences in chemical composition between austenite and ferrite. Specifically, Ni and N are partitioned to the austenite; Cr and Mo are enhanced in ferrite [3]. Therefore, the behavior of passive layer on single-phase of DSS is of great importance in terms of the dominant effect of weaker phase on the whole performance [4].

However, the researches of multiphase materials were difficult [5]. In the early days, researchers [5,6] studied the passivity of single-phase of DSS by using alloys having compositions similar to austenite and ferrite phases. But those alloys did not possess the exact element content of the composed phases of DSS and thus the passive properties of single-phase cannot be precisely presented. Recently, new tools, such as X-ray photoelectron spectroscopy (XPS) [7] and scanning Kelvin probe force microscopy (SKPFM) [8,9], provide direct methods to study the passive layer of single-phase. Unfortunately, the passive behavior of single-phase would be affected by the coupling of two phases during the formation of passive films.

In the present work, two single-phase samples, having almost exact compositions of both phases of 2205 DSS, were prepared by selective dissolution according to W.-T. Tsai [10]. Subsequently, the electrochemical behavior and compositions related to passive films of two phases were presented and compared.

#### 2. Experimental

The used 2205 DSS steel sheet with thickness of 5 cm was solution heat treated at 1200 °C for 4 h, followed by water quenching. The specimens were prepared with size of  $10 \times 5$  mm, with copper wire connected to the rear surface and was mounted in resin with 0.5 cm<sup>2</sup> exposure to solution. The composition (in wt. %) of the sample was as followed: Si 0.59, Mn 1.2, P 0.029, Mo 2.62, Cr 22.57, Ni 4.63, C 0.029, S 0.0043, N 0.13, and Fe balance. Before selective dissolution, samples were wet ground with 400~2000# SiC papers, polished using diamond paste, and ultrasonically cleaned in distilled water, ethanol, and acetone.

A mixed 2 M H<sub>2</sub>SO<sub>4</sub> + 0.5 M HCl solution was applied for selective dissolution [10] with a standard three-electrode cell. The reference electrode was saturated calomel electrode (SCE), and a platinum foil was served as the counter electrode. The respective characteristic peak potentials, -245 mV<sub>SCE</sub> and -320 mV<sub>SCE</sub> shown in Fig. 1a (showing a good coincidence with the result of W.-T. Tsai [10]), were selected for potentiostatic etching. Etching was for 10 h to ensure almost exclusive  $\alpha$ -phase or  $\gamma$ -phase was exposed on the surface, respectively. Fig. 1(b)–(f) showed the preparation process of single-phase sample. After selective dissolution, samples were smeared with epoxy resin and put into a vacuum chamber to completely expel the air in the cavities. After 24 h, the epoxy resin was carefully removed by grinding with 2000# SiC papers, leaving exclusive phase exposed on the surface.

The morphology and characteristics of single-phase sample were explored by scanning electron microscopy (SEM) together with energy dispersive spectroscopy (EDS) (FEI Quanta 250) and magnetic force microscopy (MFM) (Bruker Nano Scope V). The compositions of oxide films on  $\alpha$ -phase and  $\gamma$ -phase were investigated by X-ray photoelectron







<sup>\*</sup> Corresponding author. Tel.: +86 10 62333931; fax: +86 10 62334005. *E-mail address:* chengxuequn@ustb.edu.cn (X. Cheng).



Fig. 1. Schematic diagrams showing the preparation of single-phase sample. (a) characteristic peak potentials of α-phase and γ-phase, (b)-(f) specific steps for preparation.

spectroscopy (XPS) (Thermo Escalab 250), before which single-phase samples were immersed in neutral 3.5% NaCl solution under open circuit potential (OCP) for 12 h.

Quasi steady-state polarization and cyclic voltammetry (CV) tests were performed to characterize the behavior of passive films of two

phases using an AUTOLAB PGSTAT 302N. After removing air-formed oxide by reduction at -1.4 V for 3 min, electrochemical experiments were carried out at room temperature in neutral 3.5% NaCl solution. The reference electrode was an SCE, and a platinum foil was served as the counter electrode. All potentials were measured against an SCE.



Fig. 2. (a) SEM and (b) MFM images of  $\gamma\text{-phase}$  and  $\alpha\text{-phase}$  samples.

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