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Characterization on stress-strain behavior of ferrite and austenite in a 2205 duplex stainless steel based on nanoindentation and finite element method



Ping Tao^{a,b}, Jian-ming Gong^{a,b,*}, Yan-fei Wang^c, Yong Jiang^{a,b}, Yang Li^{a,b}, Wei-wei Cen^{a,b}

^a School of Mechanical and Power Engineering, Nanjing Tech University, Nanjing 211816, China

^b Key Laboratory of Design and Manufacture of Extreme Pressure Equipment, Jiangsu Province, China

^c School of Chemical Engineering and Technology, China University of Mining and Technology, Xuzhou 221116, China

ARTICLE INFO	A B S T R A C T		
Keywords:	The stress-strain behavior of ferrite and austenite in a commercial 2205 duplex stainless steel was investigated by		
Nanoindentation	using nanoindentation test and microstructure-based finite element method (FEM). Results showed that the		
Duplex stainless steel	optimum load range for measuring phase properties in nanoindentation test was from 3000 uN to 7000 uN. The		
Microstructure	ferrite has slightly higher average elastic modulus than austenite, while austenite has higher average nano-		
Stress-strain curves FEM	hardness than ferrite. Representative stress-strain curves of ferrite and austenite were determined by means of		
	the power-law hardening and empirical relationship. Based on FEM, the differences in distribution and portion of		
	stress-strain in local phases were visualized, and the overall flow curve of the sample 2205 was extracted, which		
	was in good agreement with the obtained results from uniaxial tensile experiment.		

Introduction

Duplex stainless steels (DSS), consisting of nearly equal volume fraction of α -ferrite phase and γ -austenite phase, possess higher strength and excellent resistance against chloride corrosion, as compared with the austenitic and ferritic stainless steels [1,2]. Nowadays, DSS have been widely used in marine environments, desalination plants and nuclear industries [3–5]. Despite the favorable properties, those steels still have the risk of failure due to environmental cracking [6–8].

Over the past few decades, studies on the method for characterization of the dual phase in DSS were focused on the combined approach of quantitative metallography and microhardness measurement [9-11]. However, this method is limited to measuring mechanical response of the local phase. The main weakness is that the substrate effect cannot be eliminated due to the alternate grains in microstructure. Most researchers have built simulation models by means of theoretical parameters or materials with mechanical properties similar to DSS [12,13]. Although many experiments and theoretical models have been developed for studies of DSS, but to date, the stress-strain curves for individual phase have not been clarified, as there is no steel having the similar chemical composition and the same grain size of the constituent phases in DSS. In addition, it is expected that ferrite and austenite respond differently to an applied load during a manufacturing or working process. Although some researchers have proposed several stereological parameters to describe the microstructure and phase distribution,

complete understanding of the macroscopic stress-strain behavior in DSS is not possible without knowledge of its local phase features. Correct prediction and control of the environment-assisted cracking of the steel require a localized description of its phase properties, with consideration of the interaction between its constituents and its geometrical arrangements [14,15]. However, the desired results cannot be directly obtained from macroscopic tensile tests or microscopic measurements.

Nanoindentation test [16-26] could be a way of investigation in this respect. Nanoindentation experiments have been used as a powerful tool for studying the mechanical behavior of wide-range materials on different scales. Its load and displacement resolution reach nanometer scale, which can provide further information on the local elasto-plastic performance in dual phase materials with minimal influence of geometric effects [16,17]. Campos et al. [18] studied the interface between ferrite and austenite grains in DSS prepared by mixing ferritic-austenitic stainless steel grade powders to identify the influence of alloying elements and sintering conditions on the mechanical properties. Wang et al. [19] concluded that the measured mechanical properties, i.e. modulus and hardness, were significantly affected by surface treatment method. Ahn et al. [20] combined nanoindentation with Electron Backscattered Diffraction to assess the nanohardness of ferrite both in static and dynamic transformations in super-cooled austenite. Furthermore, Seok et al. [21] suggested a procedure for predicting the flow curves of dual phase steels by using nanoindentation experiments

* Corresponding author at: School of Mechanical and Power Engineering, Nanjing Tech University, Nanjing 211816, China. *E-mail address*: gongjm@njtech.edu.cn (J.-m. Gong).

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performed with spherical indenters. Cheng et al. [22,23] proposed a three-step inverse fitting calculation method based on the tests with Berkovich indenter to predict the stress-strain behavior of constituent phases for quenching and partitioning steels. It has also been found that, in a nanoindentation test, the applied load in characterizing properties of local phases is greatly dependent on the grain size [24–26]. Hence, the nanoindentation testing load cannot be arbitrary for measuring the local phase properties.

Nevertheless, it is not sufficient to study the cracking behavior only by determining the stress-strain curves of ferrite and austenite in nanoindentation test, but the irregular distribution of the complicated phases in the microstructure should also be taken into account. Recently, finite element method (FEM) simulation based on microstructures observed by optical microscopy or scanning electron microscopy is an increasingly relevant approach to deeper insights into deformation behavior, such as stress and strain distribution and failure initiation in dual phase steels [13,27]. Since the bulk behavior of the steel is significantly dependent on the phase morphology and distribution, FEM modeling can bridge the gap for a further understanding of the stress-strain distribution and portion at the local phases. Thus the objectives of this study are as follows: first to find out the optimum load range for nanoindentation investigation of phase properties in DSS; then, to determine the average stress-strain curves of ferrite and austenite as well as predicting the macroscopic flow stress-strain behavior of DSS in FEM models; finally, to visualize the effect of the mixed microstructure on stress and strain distribution in local phases.

Experiment and finite element model

Material

The Material employed for this investigation was a commercial 2205 DSS produced by OUTOKUMPU with typical chemical composition (wt%:0.015C, 0.4Si, 1.41Mn, 0.001S, 0.027P, 22.45Cr, 5.69Ni, 3.14Mo, 0.27Cu, 0.2Co, 0.176N). It was received in the form of a 4 mm thickness plate and had been solution-annealed at 1393 K then quenched in forced air and water. The ferrite content of the sample was 46% according to the supplier's test results.

The chemical composition of the main alloying elements in ferrite and austenite was measured by using Energy Dispersive X-ray Spectroscopy (EDS) operated at 15 kV. As listed in Table 1, ferrite has a higher content of Cr and Mo, while austenite has a higher content of Ni. The optical microscope (OM) and scanning electron microscope (SEM) micrographs of the as-received 2205 DSS are shown in Fig. 1. As a result of rolling, a heavily banded microstructure in two directions with austenitic islands scatters a lot with typical values in the range about $20-100 \,\mu$ m in the longitudinal (rolling) direction.

Experimental procedures

Uniaxial tensile tests were carried out on a Hydraulic Servo 4830 machine at strain rate $\dot{\epsilon} \sim 10^{-3} \, \text{s}^{-1}$ at ambient temperature. Tensile specimens with a gauge size of $25 \times 10 \times 2 \, \text{mm}^3$ were machined from the plate with a tensile axis parallel to the rolling direction. The surface of specimens was ground with successive grades of emery paper up to 2000 grit, polished with paste, washed with deionized water and dried.

Nanoindentation measurements were performed on Hysitron

 Table 1

 Chemical composition of ferrite and austenite phase measured by EDS.

Fe	Cr	Ni	Мо
64.96	22.534	4.35	4.32
66.34	20.98	5.97	3.10
65.65	21.76	5.16	3.71
	Fe 64.96 66.34 65.65	Fe Cr 64.96 22.534 66.34 20.98 65.65 21.76	Fe Cr Ni 64.96 22.534 4.35 66.34 20.98 5.97 65.65 21.76 5.16

Triboindenter TI-Premier using a Berkovich indenter tip. Surface preparation of specimens of size \emptyset 15 × 4 mm for testing was ground to 2000 grit, followed by mechanical polishing with 3 µm and 1 µm waterbased diamond suspensions. Then, the electro-polishing was employed to get rid of the work-hardening microscopic layer caused by mechanical polishing. The electrolyte during the experiment was composed of 14 vol% H₃PO₄, 18 vol% H₂SO₄, 53 vol% C₃H₅(OH)₃ and 15 vol% H₂O [28], all of which were chemical purity. Single indentation in the range of 1000 µN to 12,000 µN with loading and unloading times set to 30 s and maximum load hold time to 5 s, respectively. The distance between adjacent indentations was set to be 7.5 µm, large enough to avoid mutual influence. Five separate areas were selected for the experiments and at least five different indentation points in each phase were applied at each load level.

Finite element model

A section of 0.1 mm \times 0.1 mm area was chosen from microstructure and imported into the finite program ABAQUS to build the numerical model with the same orientation and phase fraction in the duplex microstructure, as shown in Fig. 2. The FEM model was meshed with 95,584 elements. Homogenous boundary conditions at each side were used to analyze the phase-specific stress and strain distribution. Symmetric boundary conditions were applied to the left side and bottom side of the models and the load in the form of displacements was applied to the right side of the models in the x-direction. The Poisson's ratio is 0.3 and homogeneous mechanical properties were assumed for each phase in all directions. Representative stress-strain relationships of ferrite and austenite derived from nanoindentation experiments were put in FEM models.

Results and discussion

Determination of phase properties in 2205 DSS

Hardness and elastic modulus

Prior to the indentation tests on the specimen, the contact area function was calibrated by an indirect method, where a 10 by 10 indent array at various contact displacements (varying loads from 10,000 μ N down to 500 μ N) was performed in a fused quartz of known elastic modulus [29]. The load function used was a load control function with a 5 s load time, 2 s hold time and a 5 s unload time. The measured contact area function A_c is expressed as:

$$A_{c} = C_{0}h_{c}^{2} + C_{1}h_{c} + C_{2}h_{c}^{\frac{1}{2}} + C_{3}h_{c}^{\frac{1}{4}} + C_{4}h_{c}^{\frac{1}{8}} + C_{5}h_{c}^{\frac{1}{16}}$$
(1)

According to the Kick's law [30], the values of load should be proportional to the square of the values of indentation displacement in the loading segment:

$$P = Ch^2 \tag{2}$$

where *C* is the loading curvature, *P* and *h* are the indentation load and displacement.

Typical load versus displacement curves of ferrite and austenite at various loads captured in nanoindentation tests from $1000 \,\mu$ N to $12,000 \,\mu$ N are shown in Fig. 3a. Also the in-situ Scanning Probe Microscope (SPM) images of each phase at the load of $5000 \,\mu$ N in the nanoindentation tests are presented in Fig. 3b. By linear fitting of indentation load \sqrt{P} and displacement *h*, parameters of average loading curvature of ferrite (C_{α}) and austenite (C_{γ}) can be extracted. As shown in Fig. 4, C_{α} is lower than C_{γ} which implies that ferrite is easier to be penetrated than austenite. This can also be confirmed by their residual indentation morphologies in SPM of each phase in Fig. 3b. Clearly, the plastic deformation and residual form of ferrite are more obvious than those of austenite. The nanohardness and elastic modulus were determined by the method proposed by Oliver-Pharr, as given in Eqs.

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