Materials Science & Engineering A 666 (2016) 269–279

Contents lists available at ScienceDirect

Materials Science & Engineering A

journal homepage: www.elsevier.com/locate/msea

Towards understanding the effect of deformation mode on stress corrosion cracking susceptibility of grade 2205 duplex stainless steel

C. Örnek*, D.L. Engelberg

Materials Performance Centre & Corrosion and Protection Centre, School of Materials, The University of Manchester, Sackville Street, Manchester M13 9PL, United Kingdom

ARTICLE INFO

Article history:
Received 17 February 2016
Received in revised form
20 April 2016
Accepted 21 April 2016
Available online 22 April 2016

Keywords:
Duplex stainless steel
Atmospheric corrosion
Stress corrosion cracking
Electron backscatter diffraction (EBSD)
X-Ray diffraction (XRD)
Scanning electron microscopy (SEM)

ABSTRACT

The effect of bending, rolling, and tensile deformation on stress and strain development in grade 2205 duplex stainless steel has been investigated using x-ray diffraction (XRD) and electron backscatter diffraction (EBSD) analyses. The deformed microstructures were assessed for their stress corrosion cracking (SCC) susceptibility, with highest microstructure propensity observed after bending deformation. Strain localisation occurred in the austenite, independent of applied deformation mode. Cold rolling and bending also resulted in stress development in the austenite, with the ferrite also indicating significantly increased stresses after tensile straining. The austenite phase became more susceptible towards SCC, whereas the ferrite seemed to be more prone towards selective dissolution. Rolling deformation enhanced the propensity to localised corrosion.

1. Introduction

Duplex stainless steel (DSS) has been increasingly implemented as a structural material in a wide range of application such as nuclear, petrochemical, medical, and automotive. Over the last decade, an increasing use of DSS's was recorded and a growing demand has been forecast [1]. These steels provide good mechanical properties with excellent resistance to corrosion and SCC [2]. The introduction of cold work is routine practice in industry, and it is now accepted that the degree of deformation significantly changes microstructure, mechanical, as well as corrosion properties.

For manufacturing DSS components into complex shapes and designs, rolling, shaping, and drawing is typically performed, which ultimately results in microstructure deformation with strain and stress partitioning in the duplex microstructure. The latter is based on the mismatch of mechanical deformation behaviour between ferrite and austenite, which in turn can affect microstructure corrosion and SCC susceptibility [3]. The knowledge about microstructure and processing history has crucial importance in understanding and predicting corrosion and SCC phenomena.

The duplex microstructure is complex; it consists, usually, of a balanced ratio of ferrite (δ) and austenite (γ) [4]. There is often residual strain present in the microstructure due to different thermal expansion coefficients of ferrite and austenite, which causes heterogeneous strain fields during rapid cooling processes [5,6]. Furthermore, the grains of forged duplex alloys are non-equiaxed and elongated in the rolling or milling direction, leading to a texture-like banded structure with discontinuous island-like austenite grains embedded in a ferrite matrix [7]. The population of microstructural features, including the distribution of existing phases, grain boundaries with their characters, geometries, and morphologies, and stress and strain states are important to assess the performance of the material with exposure to demanding environments.

Grade 2205 DSS is currently implemented as a storage container material for some of the UK's intermediate-level radioactive waste (ILW) streams [8,9]. Chloride-bearing particulates can deposit on container surfaces during storage, which can become wet within a wider range of relative humidity (RH) forming highly aggressive thin-film electrolytes, leading to localised corrosion. This phenomenon has become known as atmospheric corrosion [10–14]. $MgCl_2$ is one of the most detrimental salts which can form up to 12 M chloride electrolyte at its deliquescence RH at room temperature and, therefore, atmospheric corrosion may be a potential challenge for the integrity of ILW containers. In the presence of residual stress, a transition to atmospheric-induced stress corrosion cracking (AISCC) may further endanger the durability of ILW containers [10–14]. The internal surface of the container can reach temperatures up to 50 °C, with the containers subjected to cold deformation and welding processes during fabrication [15–17].

* Corresponding author.
E-mail addresses: cem.oernek@manchester.ac.uk, cem_oernek@hotmail.de (C. Örnek), dirk.engelberg@manchester.ac.uk (D.L. Engelberg).

http://dx.doi.org/10.1016/j.msea.2016.04.062

In this work the effect of cold work has been investigated under conditions relevant to the storage of ILW. Rolling, bending, and tensile deformations were applied on plate and sheet grade 2205 DSS materials, and the microstructure development characterised by EBSD, with XRD being implemented to measure the stress evolution in ferrite and austenite. The microstructure after cold deformation was exposed to droplets of $MgCl_2$ in a temperature- and humidity-controlled environment, and the susceptibility to atmospheric corrosion and AISCC was investigated. The implications of these observations are discussed in light of microstructure optimisation for DSS application in demanding environment.

2. Experimental

2.1. Materials used

Sheet and plate grade 2205 DSS, supplied by *Rolled Alloys UK*, with a composition given in Table 1, was used in this work. Both materials were solution-annealed by the manufacturer at 1050 °C for 1 h, and denoted as 'as-received'. The thickness of the plate and sheet materials was 10 mm and 2 mm, respectively. Tensile, bending, and rolling deformation was introduced and the effect of microstructure development and susceptibility to AISCC was investigated.

Cold rolling with a thickness reduction of 20% and 40% was carried out on bars cut from the plate material with dimension of 10 mm (width) × 10 mm (thickness) × 80 mm (length). The latter was performed along the pre-existing rolling direction (RD) of the microstructure. Coupons in sizes of 10 mm by 10 mm were prepared from as-received and cold-rolled plate materials and used for metallographic assessment and corrosion testing.

Flat strip specimens with dimensions of 70 mm (length) × 20 mm (width) × 2 mm (thickness) were manufactured from the sheet material, with RD of the microstructure being parallel to the length of the specimens. A hole was drilled in both ends with a radius of 5.2 mm in order to fixate the specimen after bending. Bending deformation was carried out with a self-designed clevis-pin bending setup, as shown in Fig. 1, giving the sheet metal a U-shape. An *Instron 5569* testing machine was used in compression mode, with a load cell of 50kN and a loading rate of 10 mm/min to bend the specimens. The radius of the pin was 10 mm. After bending, the sample was fixated using a bolt and nut to keep the samples under elastic strain. A photograph of the U-bend specimen is shown in Fig. 1(d).

Flat tensile specimen were prepared from the sheet material with a gauge length of 50 mm, mechanically ground to 1200-grit emery paper, and electropolished using a 20 vol% perchloric acid and 80 vol% methanol with 20 V for 60 seconds at −35 °C. Tensile loading was performed using a self-designed tensile rig. A strain gauge was attached on the other side of the specimen to record the actual strain. One tensile specimen was loaded to 0.2% strain and another one was strained to 2% strain.

2.2. Microstructure characterisation

Metallographic examination was performed on all material conditions, which included as-received, 20% and 40% cold-rolled, 2% tensile-strained, and U-bend microstructures. The surface of specimens was prepared by grinding up to 4000-grit using SiC grinding papers, followed by mirror-finish to 0.1 μm with diamond paste, and finalised with an OP-S polishing (∼40 nm silica colloid particles) treatment for one hour. The U-bend specimens were analysed before and after bending in the fixated condition, and the tensile specimen was analysed before and after applied load. For SEM observations and EBSD measurements, the tensile specimen had to be removed from the tensile rig.

EBSD was used for material characterisation of plate and sheet materials by extracting grain size, grain boundary information, crystallographic phase fraction, and local misorientation (LMO), with the latter indicative of the distribution of plastic strain. An *FEI Quanta 650* scanning electron microscope (SEM) interfaced with a *Nordlys* EBSD detector from *Oxford Instruments* with *AZtec V2.2* software was used for data acquisition. A step size of 0.15–0.74 μm over a large area covering 1000s of grains with an accelerating voltage of 20 kV was used. Data post processing was carried out with HKL Channel 5 software. High-angle grain boundaries (HAGB's) were defined with misorientation ≥15° and low-angle grain boundaries (LAGB's) between >1° and <15°. The grain size was determined by the mean linear intercept method as the mean of the vertical and horizontal directions. The standard deviation for each grain size measurement was determined from the square root of the variance. LMO maps were generated by using a 3 × 3 binning and a 5° threshold for the sub-grain angle threshold. This analysis gives the average LMO for a misorientation below the pre-determined sub-grain angle threshold, and can be used to locate regions with higher concentrations of misorientation in microstructures. The latter is typically associated with local micro-deformation in the form of plastic strain, due to the presence of dislocations [18].

2.3. Atmospheric corrosion and AISCC tests

As-received samples, 10, 20, and 40% cold-rolled rectangular coupons, one tensile specimen with 2% applied load, and one U-bent specimen, all with 4000-grit surface finish, were exposed to controlled atmospheric environment using a KBF Binder humidity chamber. Tests were performed by depositing droplets containing $MgCl_2$ and a mixture of $FeCl_3 + MgCl_2$ (0.68: 1 mol ratio of $FeCl_3$: $MgCl_2$) onto the surface of each specimen, yielding a surface chloride coverage (initial deposition density) ranging from 145 μg/cm^2 to 1450 μg/cm^2. All samples were exposed to 50 °C and 30% RH and to 80 °C and 27% RH for 9–455 days. Further details of the test conditions are summarised in Table 2. An Eppendorf micropipette was used to dispense a 0.5 μl droplet volume producing an overall droplet radius of 1.7 mm. The effect of secondary spreading of the droplet was not considered, and all data are therefore referred to as initial deposition density. The exposure conditions chosen are close to the equilibrium deliquescence RH of $MgCl_2$, resulting in a concentrated $MgCl_2$ solution in the range of 12–15 M chloride [19–21].

After exposure, the specimens were rinsed with deionised water and the corrosion products removed by immersion in hot 10 wt% citric acid solution. The corrosion morphology was analysed with an *FEI Quanta 650 SEM*. Ferrite and austenite was distinguished by their channelling contrast difference and morphological appearance, giving ferrite a darker appearance.

Table 1
Chemical compositions (in wt.-%) of grade 2205 duplex stainless steel used.

Grade	C	Si	Mn	P	S	Cr	Ni	Mo	N	Nb	Cu	Co	Fe
Plate	0.016	0.40	1.50	0.021	0.001	22.40	5.80	3.20	0.180	–	–	–	bal.
Sheet	0.015	0.42	1.41	0.020	0.001	22.44	5.75	3.32	0.155	0.006	0.21	0.12	bal.