Materials Science & Engineering A 659 (2016) 47–54

Contents lists available at ScienceDirect

Materials Science & Engineering A

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

Fatigue damage in coarse-grained lean duplex stainless steels

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ARTICLE INFO

Article history:
Received 21 August 2015
Received in revised form
3 February 2016
Accepted 4 February 2016
Available online 18 February 2016

Keywords:
Duplex stainless steel
Grain coarsening
Fatigue
Crack nucleation and growth
Microstructure

ABSTRACT

The present investigation is focused on assessing the effect of a thermal treatment for grain coarsening on the low cycle fatigue damage evolution in two types of Lean Duplex Stainless Steels (LDSSs). The dislocation structure developed during cycling is observed by transmission electron microscopy (TEM). Additionally, a detailed analysis of short crack initiated and grown during low cycle fatigue (LCF) is performed by means of optical and scanning electron (SEM) microscopy in combination with automated electron back-scattered diffraction (EBSD) technique. Though in both coarse-grained LDSSs the short cracks nucleate in the ferrite phase, in each steels its origin is different. The embrittlement caused by the Cr_2N precipitation and the plastic activity sustained by each phase can explain this difference. The propagation behavior of the short cracks present two alternative growing mechanisms: the crack grows along a favorable slip plane with high Schmid Factor (SF) or the crack alternates between two slip systems. In both cases, the crack follows the path with the smallest tilt angle (β) at a grain boundary.

1. Introduction

The degradation of the material resulting from cyclic stress/strain called fatigue damage comprises several stages. The early stage of this process is the concentration of cyclic plastic strain formed during the dislocation structure evolution. Slip bands, inclusions, precipitates, grain boundaries and twin boundaries could exert this local plastic concentration. In these sites the first microcracks nucleates. The microcracks form, grow and/or coalesce generating a macrocrack which propagates up to the fracture of the material. The period of short crack initiation and growth determines in most cases the fatigue life of a specimen. The main feature for those short cracks is that their nucleation and propagation rate is strongly influenced by the microstructure. Particularly, in multiphase alloys there are a large number of parameters which can influence fatigue damage, such as chemical composition, microstructural morphology and the local plastic activity of each phase [1,2].

Duplex stainless steels (DSSs) are two-phase austenitic (γ)-ferritic (α) alloys with principal alloying elements chromium, nickel and molybdenum. Thanks to the attractive combination of mechanical properties and corrosion resistance, DSSs are widely used in different industries as; petrochemical, pulp and paper, chemical tankers and architecture. The fluctuating alloying element prices (especially nickel and molybdenum) during the last decade has accelerated the development of more economic DSSs named Lean DSS (LDSSs). In LDSSs, the expensive nickel is partly substituted by nitrogen and manganese without degradation of corrosion and mechanical properties [3,4]. The usual manufacture process of DSSs, includes alternative steps of rolling and annealing, resulting in a lamellar microstructure of both phases with fine grains. The fatigue damage can be studied to its early stages by observation of the dislocation structure, by following the evolving surface relief and later by observing short crack nucleation and growth [5,6]. Therefore, in order to simplify the microcrack observation in DSSs during fatigue the grains are usually coarsened by a heat treatment of at 1250 °C followed by slow cooling to 1050 °C followed by a water-quenched [7–11]. This grain coarsening heat treatment (GCT) not only can change the morphology of the individual phases but also other microstructural modifications can take place. In this respect, it should be taken into account that precipitation of nitrides occurs after rapid cooling from high temperatures in ferritic steels, as in the ferritic phase of DSSs [12–14]. During this rapid cooling there is insufficient time for diffusion of nitrogen into austenite and the ferritic phase becomes supersaturated with nitrogen. Thus, intragranular chromium nitrides precipitate in ferrite with detrimental effects on material properties [13,14].

Additionally, it is also important to consider that depending on the microstructure and chemical composition of the austenitic phase of DSSs strain-induced martensitic transformation may occur [15,16]. Chiu et al. [17] suggest that the strain-induced martensitic transformation in metastable austenitic stainless steels

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http://dx.doi.org/10.1016/j.msea.2016.02.012
0921-5093/

have a beneficial effect on the fatigue resistance. The formation of strain-induced martensite in austenite of low stacking-fault energy (SFE) is closely related to shear bands, which are planar defects associated with the overlapping of stacking faults [18]. It is worth noting that the martensite transformation is enhanced by a lower SFE. In this sense, Jun and Choi [19] studied the correlation between the SFE and the austenite grain size in an Fe-Mn binary system. They showed that the SFE decreases rapidly with the increase of the austenite grain size in the range of $13-35\ \mu m$, over which SFE comes in a saturation region up to $185\ \mu m$. Recently, Saeed-Akbari et al. [20] evalued the grain size dependency of SFE in High-Manganese Steels. They found that SFE decreases as the grain size increases for austenite grain size in the range of $5-50\ \mu m$.

Thus, the results of the fatigue damage in GCT-DSSs should be carefully analyzed before performing a direct extrapolation to those expected in as-received DSSs. Therefore, this work intends to highlight the influence of GCT on the fatigue damage of two different LDSSs.

2. Material and experimental procedure

2.1. Material

The investigated materials were two LDSSs, LDX 2101 (UNS S32101) and AL 2003 (UNS S S32003). Table 1 gives the chemical composition in weight percent of both LDSSs. These LDSSs were selected taken into account their different Ni contents. LDSS AL 2003 (UNS S32003) has a chemical composition similar to the standard SAF 2205 while LDX 2101 (UNS S32101) is more representative of LDSSs. These steels were received in longitudinally welded stainless steel pipes. The manufacturing process of the pipes includes a hot rolled stage and a subsequent welding of the tube. A thermal treatment at 1050 °C followed by a water quench was finally carried out to the tube. The steels supplied after this industrial process will be hereinafter designated as as-received (AR). In this condition [11,21], a lamellar structure of austenite and ferrite is distinguished in the rolling direction with no evidence of any additional secondary phases. A coarse grain structure of DSSs facilitates microcracks observation, so the AR materials were solution annealed 2 h at 1250 °C followed by slow cooling to 1050 °C (to regain identical fractions of austenite and ferrite) and subsequent water quenching. LDSSs with coarse grains will be referred as coarse grain thermal treated LDSSs (GCT – LDSSs).

2.2. Experimental procedure

Metallographic preparation of specimens included a standard mechanical grinding procedure and a two-step electrolytic etching, method documented to be successful for providing indirect evidence for the presence of nitrides and revealing the microstructure of the samples [13]. Nevertheless, grain boundaries are slightly visible using this etching procedure. Therefore, SEM electron backscattered diffraction (SEM-EBSD) technique was used as a quantitative characterization tool [22]. The use of this technique enabled us to determine the average grain size and volume fraction of each phase whereas the microhardness of both phases of each LDSS was also measured (Table 2). The Vickers indentations were performed with a load of 245.2 mN during 10 s For each sample a minimum of 10 measures were carried out. With these results thereafter, the corresponding medium value with its standard deviation was calculated.

From slabs taken parallel to the axis of the pipe, flat specimens were prepared by electro erosion with a 20 mm gauge length and a section of 30 mm^2. In order to obtain a smooth surface for the fatigue tests, all the specimens were initially ground and polished with sequentially finer grits. These specimens were used to obtain the cyclic stress–strain curves under fully reversed total strain control, applying a triangular waveform at a constant total strain rate of $\dot{\varepsilon}=2\times10^{-3}\ s^{-1}$, with total strain range of $\Delta\varepsilon_t=0.6\%$. This total strain value corresponds to a plastic strain range, measured from the hysteresis loop at midlife to fracture, of approximately $\Delta\varepsilon_p=0.2\%$. In order to observe the damage evolution during LCF tests, additional cyclic tests were conducted at room temperature under fully reversed plastic strain control, with a plastic strain range of $\Delta\varepsilon_p=0.2\%$. Under these conditions, tests were repeated five times so as to detect in each phase the surface relief associated with initial slip lines and the subsequent microcrack nucleation and growth. Specimens for these tests were further electrolytic-polished; using a solution of 10% perchloric acid in ethanol as electrolyte. This surface preparation allows the observation of the structure during tests and the acquisition of good quality electron back-scattered diffraction patterns (EBSD). Surface damage observations of a central sector of the specimens were performed by in situ microscopy before and during the LCF test using an optical system composed of a CCD camera JAI mod. CM-140MCL with a $50\times$ objective, focal length of 13 mm, depth of field of $\pm1\ \mu m$ and a $12\times$ ultra zoom device mounted on the fatigue test machine. After LCF tests a Scanning Electron Microscope (SEM) equipped with EBSD detector was used to determine the slip systems, their Schmid Factor (SF) and their angles relative to the tensile axis. On the other hand, the angles between the surface slip markings and the loading axis were measured. The comparison of these angles with the calculated ones permits to identify the activate slip systems and theirs corresponding SFs.

In order to analyze the dislocation structure before and after fatigue thin foils were observed by TEM operating at 100 kV.

Table 1
Chemical composition of LDSS AL 2003 and LDX 2101 in weight percent (wt%).

LDSS	C	Si	Mn	P	S	Ni	Cr	Mo	Cu	N
AL 2003	0.021	0.22	1.73	0.024	–	3.8	22	1.8	–	0.18
LDX 2101	0.026	0.63	4.9	0.021	0.001	1.53	21.53	0.28	0.33	0.22

3. Results and discussion

Table 2 summarizes the average grain size, volume fraction and hardness of each phase in GCT-LDX 2101 and GCT-AL 2003. Fig. 1 shows the microstructure of both LDSSs after the GCT. Whereas the microstructure of the AR LDSSs have been characterized by lamellar phases highly elongated in the rolling direction with fine grains [11,21], an isotropic microstructure with a considerable increase of the grain size in both phases is produced by GCT. This figure also shows small etching pits in the ferrite phase of both steels, corresponding to Cr_2N. During cooling from high temperatures, as occurs in GCT, the solubility of nitrogen is much higher in austenite than in ferrite. Therefore, if the cooling rate is high enough to prevent diffusion of nitrogen into austenite, the ferrite gets supersaturated with nitrogen and chromium nitrides are then formed [12]. In this sense, as it is seen in Fig. 1, the nitrogen neighbour to the phase boundaries has had time to diffuse into the austenite avoiding the precipitation of Cr_2N. In LDSSs, nickel is partially replaced by nitrogen as an austenite stabilizer. However, a high concentration of manganese is also added to ensure adequate nitrogen solubility [23]. A higher density of chromium nitrides precipitate in the ferrite phase of GCT-AL2003 than in GCT-LDX 2101, consistent with the lower solubility of nitrogen in GCT-AL2003. This result agrees with the microhardness