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Letter to the Editor

Magnetic detection of sigma phase in duplex stainless steel UNS S31803

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ABSTRACT

Duplex stainless steels are high strength and corrosion resistant steels extensively used in the chemical and petrochemical industry. The best mechanical properties and corrosion resistance are obtained with a microstructure composed by equal parts of ferrite and austenite and free from tertiary phases. Sigma phase is one of these deleterious tertiary phases. In the present work different amounts of sigma phase were precipitated by heat treatments in a UNS S31803 stainless steel. Some specimens were cold rolled before sigma phase precipitation in order to evaluate the effect of deformation on the magnetic measurements. The amount of sigma phase was precisely determined by microscopy and image analysis for each heat treatment condition. The effects of sigma phase on the steel properties were investigated, confirming the detrimental effects of very small percentages on corrosion resistance and toughness. Two magnetic methods were used to detect sigma phase: magnetization saturation measurements in a Vibrating Sample Magnetometer and ferritoscope testing. Both methods were found to be sensitive to small percentages of sigma phase in the microstructure.

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1. Introduction

Duplex stainless steels present excellent mechanical and corrosion resistance properties when the microstructures are composed only by austenite (γ) and ferrite (δ) in approximately equal amounts [1,2]. Undesirable tertiary phases such as sigma (σ) and Chi (χ) may precipitate during hot forming or welding operations [3]. Spinodal decomposition of the ferrite phase into small particles of Cr-rich α' (α'_{Cr}) and Fe-rich matrix (α_{Fe}) may also occur during prolonged aging in the 350–550 °C range. This reaction is faster at 475 °C and causes hardening, embrittlement and corrosion decay [1,4].

Previous works [3,5,6] have shown that sigma phase precipitation in duplex stainless steels may occur between 600 and 1000 °C. In most steels the aging in the 800–900 °C interval gives the faster kinetics of precipitation. Cr-rich particles may also precipitate by spinodal decomposition of ferrite.

Sigma phase precipitates from ferrite in γ/δ interfaces or in δ/δ grain boundaries [7]. For most temperatures ferrite decomposes into austenite (γ_2) and sigma (σ) as an eutectoide reaction ($\delta \rightarrow \sigma + \gamma_2$) [3,8,9]. A small percentage of Chi (χ) phase is also prone to precipitate in Mo-rich duplex and superduplex steel by a $\delta \rightarrow \chi$ reaction, in the 700–900 °C range [7,8,10]. According to Michalska and Sozanska [11] χ-phase is metastable because is replaced by σ with the increase of aging time. In fact, these two phases are hardly separated by optical microscopy, but can be distinguished by scanning electron microscopy with polished specimens observed in the backscattered mode [8].

The precipitation of σ and χ phases by $\delta \rightarrow \sigma + \gamma_2$, $\delta \rightarrow \sigma$ or $\delta \rightarrow \chi \rightarrow \sigma$ reactions, must decrease the magnetic properties of the steel because ferrite is ferromagnetic and χ, σ and γ_2 are paramagnetic phases.

Some previous works have also proved the applicability of magnetic measurements on the detection of spinodal decomposition of the ferritic phase in duplex stainless steels [4,12]. The magnetic susceptibility was found to decrease during prolonged thermal aging due to α'_{Cr} precipitation [12,13]. Based on the strong variation of magnetic properties with the microstructural changes, Lo et al. [13] and Shek et al. [14] have shown interesting results about the use of duplex stainless steels as temperature indicators.

In the present study the formation of small quantities sigma phase in a wrought DSS UNS S31803 was investigated by two magnetic methods. Corrosion resistance and mechanical properties changes were also measured for some conditions.

2. Experimental

A 4.0 mm thick plate of solution treated duplex stainless steel UNS S31803 with composition shown in Table 1 was cut into pieces of $60.0 \times 30.0 \times 4.0\ mm^3$. These specimens were heat treated at 800 °C for 5, 10, 15, 20 and 30 min. Another group of

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Table 1
Chemical composition of the UNS S31803 steel.

Chemical analysis (%wt)							
Cr	Ni	Mn	Mo	C	S	P	N
22.3	5.44	0.92	2.44	0.02	0.004	0.010	0.160

Table 2
Heat treatments and specimens identification.

Heat treatment	Un-deformed	Deformed
–	ST	CR
800 °C–5 min	ST-800-5	CR-800-5
800 °C–10 min	ST-800-10	CR-800-10
800 °C–15 min	ST-800-15	CR-800-15
800 °C–20 min	ST-800-20	CR-800-20
800 °C–30 min	ST-800-30	CR-800-30
1050 °C–furnace cooled	ST-1050-F	–

specimens were cold rolled from 4.00 to 2.25 mm (true deformation 0.575) before the aging at 800 °C for the same periods of time. One of the un-deformed specimens was heated at 1050 °C for 40 min and cooled in furnace. This slow cooling was practiced to allow sigma phase precipitation via continuous cooling. Table 2 shows the identification of all specimens produced in this study.

Each specimen was tested with the ferritoscope from Helmut–Fischer®, a commercial device used to measure the ferrite or martensite phases in austenitic and duplex stainless steels [15,16]. Before measurements the ferritoscope was calibrated with the duplex standard samples. Ten measurements were performed in each specimen.

Small discs with 3.5 mm diameter and 0.2 mm thickness were cut and machined from the specimens for measurement of the saturation magnetization in a Vibrating Sample Magnetometer. Saturation magnetization was taken in an applied field of 2 T.

The amount of sigma phase of each sample was quantified by image analysis from optical micrographs. The specimens were prepared by mechanical polishing and electrolytic etch in a 10% KOH solution. Voltage and time applied were 3 V and 15 s. The electrolytic etching with 10% KOH solution reveals σ phase as brown particles which can be quantified by image analysis. It is possible that some χ phase was also formed and quantified as σ phase. However, it was not the objective of this work to separate these two phases, because they have similar effects on steel properties. The volume fractions of σ were quantified with Image Tools software [17]. Ten fields were quantified for each condition. The error associated to each measurement was estimated by the standard deviation of the samples assuming a normal distribution and 99% of confidence.

The effects of small quantities of σ on the mechanical properties and pitting corrosion resistance were quantified in the un-deformed specimens. After ferritoscope measurement they were cut and machined to produce two reduced size Charpy impact specimens with dimensions $55 \times 10 \times 2.5\ mm^3$, according to the ASTM E-23 standard [18]. Charpy tests were carried out at room temperature ($RT = 25$ °C) in a universal impact machine with maximum capacity 300 J. The estimated error associated to impact tests is ± 1 J. After the impact tests specimens with dimensions $15 \times 10 \times 2.5\ mm^3$ were cut for corrosion tests.

The pitting corrosion resistance of specimens aged at 800 °C and the one treated at 1050 °C and furnace cooled were evaluated by anodic polarization tests in 3.5% NaCl solution, at room temperature and 40 °C. The tests were conducted in a conventional three-electrode cell using a Pt foil as the auxiliary electrode, and a saturated calomel electrode (SCE) as the reference one. The recommendations of the ASTM G61 standard [19] were followed. The working electrode was constructed using the duplex stainless steels specimens embedded in epoxy resin with encapsulated stainless steel wire bonded for electric contact. The tests were initiated after nearly steady-state open circuit potential (E_{oc}) had developed (about 30 min). After that, a potential sweep came through the anodic direction at 1 mV s^{-1} until the current density of 1 mA/cm^2 was reached. Then the scan was reversed to the cathodic direction up to the E_{oc} value. Prior to each experiment, the working electrodes were polished with grid 400 emery papers, degreased with alcohol and cleaned in water. The working solution was 3.5% NaCl (artificial sea water). The corrosion behavior was evaluated by the absolute value of pitting potential (E_{pit}).

3. Results and discussion

Fig. 1(a–c) show the microstructures of specimens aged at 800 °C for 10, 15 and 30 min, respectively. Fig. 1(d) shows the specimen treated at 1050 °C and furnace cooled. Fig. 2(a–b) show the specimens deformed and aged at 800 °C for 10 and 15 min, respectively. In these figures σ phase appears dark and ferrite remain almost un-attacked. A comparison between Figs. 1 and 2(a–b) clearly shows the increase of σ phase precipitation with cold deformation prior aging. Table 3 shows the results of sigma phase quantification by image analysis, expressed in volumetric percentages.

Fig. 3 shows the variation of toughness and hardness with %σ in specimens aged at 800 °C without previous deformation. Small contents of σ phase promote considerable decrease of toughness without notable influence on the hardness. For instance, the precipitation of 1.3%σ decreased the impact toughness from 32 J (specimen ST) to 24 J (specimen ST-800-10). Similar results were obtained by Nilsson [20], Gun [7] and Chen [5] in duplex and superduplex steels.

Figs. 4 and 5 show the polarization curves for specimens ST and ST-800-5, respectively. In the solution treated specimen the pitting potential (E_{pit}) is at least 1.0 V_{SCE} for both test temperatures (RT and 40 °C), which means that the critical pitting temperature (CPT) is higher than 40 °C. On the other hand, the specimen ST-800-5, which contains only 0.20%σ, presents a pronounced decrease of E_{pit} when the temperature is raised from RT to 40 °C. The precipitation of such a low amount of σ phase caused the decrease of the CPT to temperatures below 40 °C. Table 4 shows the E_{pit} values of all un-deformed specimens. At room temperature a considerable decrease of the pitting potential was only observed in the specimen ST-800-30, which presents (10.9 ± 1.6)% of σ phase. The critical pitting temperature (CPT) of this specimen is lower than 25 °C.

Fig. 6 shows the variations of the magnetization saturation (m_s) and ferritoscope reading (F) with the amount of σ phase for deformed and un-deformed specimens. Error bars were not included in this figure for a better comprehension. First analyzing the results of the specimen ST and CR specimens, the magnetization saturation found in ST (56.6 Am^2/kg) corresponds to a ferrite (δ) content equal 42.6%, using the relation ($C_\delta = m_s/133.0$) proposed in [21]. The ferritoscope reading of specimen ST indicates a ferrite content slightly different: (38.3 ± 0.4)%. The results of specimen CR show a larger difference. The magnetization saturation measured in CR was 57.8 Am^2/kg, which corresponds to 43.5% δ. In fact, cold deformation does not increase the ferrite content, but may promote some bcc martensite (α') formation by a $\gamma \rightarrow \alpha'$ reaction.

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