Journal of Magnetism and Magnetic Materials 388 (2015) 96–100

Contents lists available at ScienceDirect

Journal of Magnetism and Magnetic Materials

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

Eddy current techniques for super duplex stainless steel characterization

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ARTICLE INFO

Article history:
Received 30 July 2014
Received in revised form
6 April 2015
Accepted 7 April 2015
Available online 8 April 2015

Keywords:
Eddy current testing
Super duplex stainless steel
Sigma phase

ABSTRACT

Super duplex stainless steel (SDSS) is *a* two-phase material where the microstructure consists of grains of ferrite (δ) and austenite (γ). SDSS exhibit an attractive combination of properties, such as: strength, toughness and stress corrosion cracking resistance. Nevertheless, SDSS attain these properties after a controlled solution heat treatment, leading to *a* similar volumetric fraction of δ and γ. Any further heat treatment, welding operation for example, can change the balance of the original phases, or may also lead to precipitation of a deleterious phase, such as sigma (σ). For these situations, the material corrosion resistance is severely impaired. In the present study, several SDSS samples with low σ phase content and non-balanced microstructure were intentionally obtained by thermally treating SDSS specimens. Electromagnetic techniques, conventional Eddy Current Testing (ECT) and Saturated Low Frequency Eddy Current (SLOFEC), were employed to characterize the SDSS samples. The results showed that ECT and SLOFEC are reliable techniques to evaluate σ phase presence in SDSS and can provide an estimation of the δ content.

1. Introduction

Super duplex stainless steel (SDSS) presents an attractive combination of mechanical properties and high corrosion resistance due the presence of two phases (δ and γ) in its microstructure [1,2], and, because of these relevant properties, this material has been widely used in the marine and petrochemical industries [3,4]. In order to attain the optimal combination of mechanical and corrosion properties, the steel grades in the duplex family are metallurgical designed to exhibit in the annealed condition a microstructure consisting of equal proportion of δ *e* γ phases [2,5–7]. Some Standards [8] allow δ/γ ratios from approximately 65/35 to 55/45. However, welding operations may lead to microstructural changes in the original base material changing the balance of δ and γ phases and/or causing the precipitation of deleterious phases due to steel exposure in temperatures ranges from 300 °C to 1000 °C [7,9]. The most harmful deleterious phase that can be originated in the material microstructure is σ phase, and because of the significantly higher volumetric fraction than other deleterious phases [2] it will receive greater attention in this paper. Sigma phase precipitation can cause chromium depletion in the adjacent regions, impairing dramatically the mechanical and corrosion resistance properties of the material [10].

The presence of σ phase causes changes in the electromagnetic properties of SDSS [10]. Ferrite is ferromagnetic, while γ is paramagnetic and σ phases non-magnetic. Thus an increase of σ phase percentage with the consequent decrease of the δ phase volumetric fraction, results in lower ferromagnetic material behavior [11]. Owing to these characteristics, electromagnetic techniques are an interesting non-destructive tool for the degradation evaluation caused by the presence of σ phase content in the SDSS. The main goal of this study is characterize the presence of low amount of σ phase using typical NDT tools such as conventional ECT and Saturated Low Frequency Eddy Current (SLOFEC).

2. Background and motivation

In some previous publications, Rebello et al. performed a detailed study about the magnetic behavior of duplex stainless steel for different microstructural conditions [12,13]. Vibrating Sample Magnetometer (VSM) measurements were performed in several samples and the results are shown in Fig. 1 [13]. Through the

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http://dx.doi.org/10.1016/j.jmmm.2015.04.034
0304-8853/

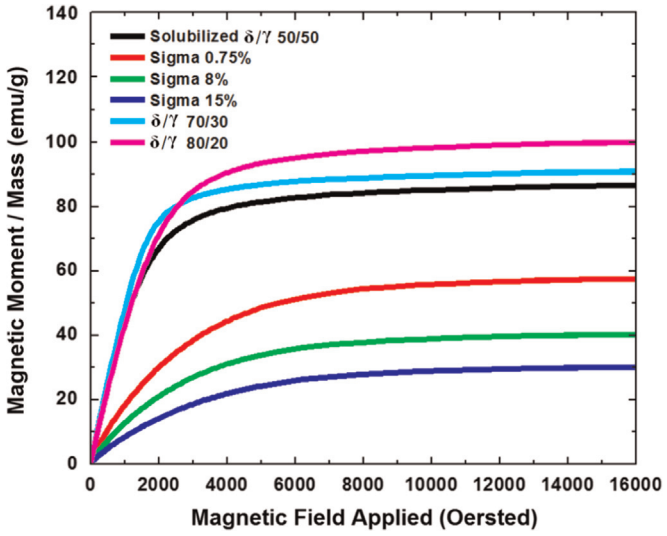

Fig. 1. M–H Curves from VSM measurements [13].

results achieved with VSM was confirmed that the σ phase precipitation is followed by a correspondingly decrease on the δ ferrite volumetric fraction changing the balance between δ and γ, consequently the magnetic properties of the steel. Knowing that σ phase is non-magnetic, an increase on its volumetric fraction causes *a* decrease of the ferromagnetic behavior of the material. Therefore, the decrease in the magnetic saturation level presented in Fig. 1 can be explained by two factors: precipitation of the non-magnetic σ phase and decrease of the ferromagnetic δ content.

Some previous publications discussed the advantages of using electromagnetic techniques for material characterization [12–17], however none of them presented the potentiality for low amount of σ phase characterization in SDSS, and neither have correlated the electromagnetic results to the δ content. In addition, only few samples were tested, leaving some uncertainty about the reliability of the techniques. The present work consist in the using of electromagnetic techniques, ECT and SLOFEC, testing an extensive number of SDSS samples representing the worst scenario for detection, low amount of σ phase, or samples with no σ phase but different balance of δ and γ. Finally, a correlation between the δ content and the electromagnetic results was performed.

3. Materials and methodology

SDSS samples, following the UNS S32750 specification, with dimensions $70 \times 40 \times 6$ mm^3, and the chemical composition presented in Table 1 are used.

Twenty four samples were submitted to a preliminary solution heat treatment in order to obtain a balance of approximately 50% of δ and γ phases. The solution heat treatment was conducted at 1220 °C during one hour, followed by water quenching. Fourteen samples received additional aging heat treatments which introduced different amounts of σ phase volumetric fractions. The aging heat treatment was conducted at 1000 °C for different time periods, followed by water quenching. Three samples did not receive additional aging heat treatment and remained in the solubilized condition. Other three samples were analyzed as received, i.e., without any heat treatment. Finally, seven samples were prepared in order to have no σ phase but higher amounts of δ phase. These were heat treated at 1320 °C and at 1350 °C during different time periods, obtaining balances δ/γ around 70/30, without any content of σ phase. It is worth mentioning that despite the δ/γ range established by NORSOK standard [8], not well controlled welding or manufacturing operations can modify the microstructure balance, surpassing the standard limits, because of that test samples out of the standard range is also important.

X-Ray Diffraction (XRD) was performed in a D8 Discover (Bruker AXS) using cobalt Co K_α radiation ($\lambda = 1{,}789$ Å), equipped with *a* Lynx Eye PS Detector. The equipment operated at constant values of tension (35 KV) and current (40 mA), respectively.

The primary optics was mounted using a Co Göbel Mirror followed by two slits of 1 mm and 6 mm and a soller slit with an aperture of 2 cm × 1 cm. The secondary optics consisted of a Fe-K_β filter followed by an 8 mm slit and an axial Soller slit with maximum divergence of 2.5°. The scanning data was obtained in the 2θ range of 45° to 105°. The step-size applied was 0.001° and the scanning velocity was 0.5 s step–1. Rietveld analysis [18] of each scan was carried out using Diffrac PlusTOPAS (ver 4.2) software, based general non-linear least squares system driven by a scripting language, which focus is in crystallography, solid state chemistry and optimization and, as consequence, has also been applied for phase quantification.

Phase volumetric fractions were measured in 9 different areas of each sample. Results of these calculated average values related to the heat treatment suffered by each sample are presented in

Table 2
Correlation between samples, heat treatment temperature and volumetric phase content.

Samples	Temperature (°C)	Time (min)	γ phase (%)	δ phase (%)	σ phase (%)
01	1000	60	64.0 ± 2.3	32.5 ± 2.7	3.4 ± 1.0
02	1000	45	49.3 ± 3.0	47.5 ± 3.5	3.1 ± 0.9
03	1000	22	64.3 ± 3.9	32.6 ± 4.0	3.0 ± 0.6
04	1000	45	62.4 ± 4.3	34.8 ± 4.1	2.7 ± 0.7
05	1000	25	52.2 ± 12.1	45.1 ± 11.5	2.6 ± 1.2
06	1000	25	65.1 ± 9.8	31.7 ± 7.8	2.4 ± 1.1
07	1000	5	68.1 ± 7.9	29.6 ± 8.2	2.2 ± 0.7
08	1000	60	61.1 ± 5.0	36.6 ± 4.9	2.1 ± 1.9
09	1000	20	64.4 ± 4.5	33.4 ± 4.5	2.1 ± 0.2
10	1000	20	56.7 ± 6.5	41.2 ± 6.9	2.0 ± 0.7
11	1000	1	57.9 ± 5.5	40.4 ± 5.3	1.6 ± 0.6
12	1000	1	59.3 ± 7.1	39.0 ± 7.1	1.6 ± 0.2
13	1000	6	68.5 ± 3.6	29.9 ± 3.6	1.5 ± 0.4
14	1000	10	61.6 ± 5.4	37.0 ± 5.3	1.2 ± 0.4
15	As received		47.7 ± 2.0	52.2 ± 2.0	0.0
16	As received		44.2 ± 4.9	55.7 ± 4.9	0.0
17	As received		47.1 ± 1.6	52.8 ± 1.6	0.0
18	1220	60	50.2 ± 7.8	49.7 ± 7.7	0.0
19	1220	60	56.8 ± 5.1	43.1 ± 5.1	0.0
20	1220	60	54.3 ± 5.7	45.7 ± 5.7	0.0
21	1320	60	38.8 ± 3.3	61.1 ± 2.9	0.0
22	1320	60	28.3 ± 5.1	71.6 ± 5.0	0.0
23	1320	120	44.8 ± 3.0	55.1 ± 3.0	0.0
24	1320	60	36.2 ± 7.4	63.7 ± 7.4	0.0
25	1320	240	41.7 ± 6.9	58.2 ± 6.9	0.0
26	1350	60	34.4 ± 6.4	65.7 ± 6.3	0.0
27	1350	60	40.5 ± 8.7	59.4 ± 8.6	0.0

Table 1
SDSS Chemical composition.

Element	C	Si	Mn	Ni	Mo	Cr	N
UNS S32750 (Weight %)	0.022 ± 0.003	0.25 ± 0.01	0.79 ± 0.01	7.2 ± 0.2	3.85 ± 0.04	24.80 ± 0.2	0.32 ± 0.005