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#### ABSTRACT

The fracture toughness and sulphide stress corrosion behaviour of a duplex stainless steel after various sensitization and regeneration heat treatments has been studied. A marked influence of the sensitization treatments on the material toughness and S.S.C. resistance was observed being maximum in those annealed at 825°C. The use of scanning electron microscopy reveals the fracture and corrosion operating mechanisms.

#### INTRODUCTION

Duplex austenitic-ferritic stainless steels are becoming increasingly popular and are replacing fully austenitic or ferritic steels due to their good combination of properties as: higher mechanical strength than austenitic stainless steels, better stress corrosion resistance and similar price to conventional stainless steels (1).

The optimum combination of properties is achieved when nearly equal proportions of austenite and ferrite are present in the microstructure (2). Nevertheless, neither the austenite nor the ferrite are completely stable and their decomposition creates still more possible structures. The precipitation of carbides, nitrides and various intermetallic phases produced during thermomechanical treatments and its strong influence on properties have been reported (3-5).

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However no study that relates these different microstructures with the fractographic facets of fracture and sulphide stress corrosion test specimens has been published.

The aim of this paper is to investigate the effect of different heat treatments on the material microstructure, fracture toughness, sulphide stress corrosion resistance and fracture topography of a duplex stainless steel.

#### EXPERIMENTAL PROCEDURE

The material chosen for the present study was a 13,5 mm thick hot rolled plate conforming to ASTM A240 type UNS 31803 whose chemical composition is (%wt) C: 0.017, Si: 0.41, Mn: 1.48, P: 0.028, S: 0.001, Cr: 22.1, Ni: 5.6, Mo: 3.0, N: 0.13 remainder Fe. The as-received mechanical properties in the longitudinal direction were as follows: 0.2% yield strength 553 MPa, tensile strength 782 MPa, elongation 37% and CTOD in the L-T orientation 1.57 mm.

The various microstructures were obtained sensitizing the material at 675 or 825°C for different periods of time up to 24 hours and giving a regeneration treatment at 1050°C to some of these samples. Fracture toughness test were performed according to B.S. 5762 standard (6) and sulphide stress cracking following the NACE TM 0177 recommendations (7). Following failure the broken specimens were examined in a scanning electron microscope in order to analyze the fracture mechanisms.

#### RESULTS

A dramatic effect of the sensitization treatments on the fracture toughness and S.S.C. resistance was detected (Table 1); the more pronounced effect has been observed in those coupons treated at 825°C where a marked embrittlement is found after only two hours of treatments. This loss of toughness is associated with sigma phase precipitation as it was identified by EDX in a previous work (8). Scanning observations of these specimens reveal narrow, elongated, parallel fissures whose walls possess a brittle character (9). Moreover, specimens sensitized at this temperature fail during the S.S.C. tests after periods of time as short as 1.8 hours. Fracture surfaces exhibit

splits similar to those found in fracture toughness specimens pointing towards the action of the same fracture mechanism.

Due to the C shaped curve of sigma phase precipitation its rate of formation at lower temperatures is slower and no evidence of this phase was found in coupons annealed at 675°C for a short period of time (5). However, copious carbide precipitation has been observed in these samples, producing a certain embrittlement although clearly smaller than that found in the 825°C treated specimens (8). Fracture surfaces of these specimens exhibit elongated fissures but these are significantly wider than those noticed in the 825°C samples and their walls are almost featureless suggesting a decohesion of austenite-ferrite interfaces embrittled by the precipitation of carbides (9). Short time sensitized S.S.C. specimens pass the test without failure showing just a sulphide attack favoured by the chromium depletion in the neighbourhood of the grain boundaries. After 24 hours annealing copious intermetallic phases precipitation embedding the carbides is detected leading to a greater decrease in toughness and S.S.C. test failures. Fracture topography of these specimens is very similar to those found in 825°C treated ones having been associated with brittle phase precipitation.

1/2 hour annealing at 1050°C is long enough to give back their good primitive fracture toughness and S.S.C. resistance to samples sensitized for 2 hours either at 675 or 825°C. Fracture surfaces of these specimens are covered with ductile dimples pointing to the action of a mechanism of microvoid coalescence. Longer time sensitized samples also recover their toughness after similar treatments but S.S.C. testing produce a wide scatter in the results. Even if some brittle zones found in the fracture surfaces of these specimens have been identified as not fully solved sigma phase, indepth research is needed before a conclusion can be reached (10).

#### CONCLUSIONS

- a) 825°C annealing induces a marked loss of toughness and S.S.C. resistance having been attributed to sigma phase precipitation.
- b) Fracture surfaces of these specimens exhibit narrow, elongated fissures whose walls possess a brittle character.

- c) Samples treated at 675°C for short periods of time show a certain decrease in toughness associated with the precipitation of carbides. Even if elongated fissures are observed in the fracture surfaces these are significantly wider than those noticed in 825°C samples and their walls are almost featureless suggesting a decohesion along the austenite-ferrite interfaces.
- d) S.S.C. specimens pass the test without failure just showing a sulphide attack favoured by the chromium depletion near the grain boundaries.
- e) Longer exposure times produces a greater loss of toughness, S.S.C. embrittlement and fracture morphologies similar to those obtained in 825°C treated samples.
- f) Short time sensitized samples recover their good toughness and S.S.C. resistance after regeneration treatments.
- g) Samples sensitized for 24 hours at 675 or 825°C and regenerated exhibit a recovery of toughness but a strange S.S.C. behaviour probably due to the inhomogeneous distribution of the remaining unsolved brittle phases.

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TABLE 1.- EFFECT OF THE SENSITIZATION TREATMENTS ON FRACTURE TOUGHNESS AND SSC FAILURE TIMES

Temp °C	Time (h)	COD (mm)	Time to failure
825	2	0,07	326
825	8	0,064	2
825	24	0,063	1,8
675	2	0,41	>720
675	8	0,22	401
675	24	0,08	380

TABLE 2.- EFFECT OF THE REGENERATION TREATMENTS ON PREVIOUSLY SENSITIZED SAMPLES

Sensitization Temp °C/time h	Regeneration Temp °C/time h	COD mm	Time to failure
825/2	1050/0,5	1,31	>720
	1050/1	1,32	>720
	1050/2	1,35	>720
	1050/4	1,31	>720
825/24	1025/0,5	1,24	492
	1050/1,5	1,36	590
	1050/2,5	1,39	>720
	1060/6	1,34	451
675/2	1050/0,5	1,29	>720
	1050/1	1,38	>720
	1050/2	1,26	>720
	1050/4	1,38	>720
675/24	1050/0,5	1,32	>720
	1050/1	1,35	>720
	1050/1,5	1,35	697
	1050/2	1,37	489