MECHANICAL EVALUATION OF A REVERSION HEAT TREATMENT FOR A DUPLEX STAINLESS STEEL THERMALLY EMBRITTLED

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Duplex stainless steels possess an excellent combination of mechanical properties. However, if they are exposed to temperatures in the range 250-500°C significant embrittlement is observed. The aim of this research has been to evaluate a heat treatment which allows to recover the values of mechanical properties as existing before ageing degradation. Hardness measurements were used as indicative basis in order to select the conditions corresponding to an optimum reversion treatment for a duplex AISI 329, after different ageings grades. Tensile and fracture toughness testing have been performed to evaluate the real effectiveness of the reversion process.

INTRODUCTION

Duplex stainless steels (DSS) are extensively used for components in the oil, chemical and power industries. This is due to their excellent combination of mechanical and corrosion-related properties (Solomon and Devine (1)). However, DSS are susceptible to severe ageing embrittlement when exposed to the intermediate temperature range (250°C-500°C). This phenomenon, usually referred to as "475°C embrittlement", is attributed to microstructural changes within the ferritic phase, being the most important the spinodal decomposition. The most dramatic manifestation of this embrittlement is the drop in toughness (*e.g.* Iturgoyen (2)). For DSS this is a very important problem because limits their industrial applicability (Chung (3)).

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Studies on ferritic (Konosu (4)) and austenitic-ferritic stainless steels (3) have shown that the microstructural changes associated with "475°C embrittlement" can be reverted by a short-term reversion treatment at temperatures within the range of 500°C-600°C. However, it is not completely clear how these changes translate on the mechanical properties of the material, and vice versa. The aim of this research has been to evaluate, from a mechanical viewpoint, a chosen reversion treatment for a DSS with different ageings grades.

EXPERIMENTAL PROCEDURE

The material used in this investigation was a first generation DSS, corresponding to the standard UNS S32900 (or AISI 329). Specimens were machined from material corresponding to two heats which exhibited noticeable differences in their nitrogen content. Their chemical composition, as specified by the manufacturer, is given in Table 1.

TABLE 1 - Chemical composition of the two heats of AISI 329 steel studied.

AISI 329	**C**	**N**	**Cr**	**Ni**	**Mo**	**Mn**	**Si**	**P**	**S**
Heat A	0.044	0.027	25.0	5.3	1.4	1.75	0.77	0.031	0.026
Heat B	0.036	0.072	24.6	5.4	1.4	1.73	0.34	0.024	0.029

The as-received material was subjected to a solution treatment at 1050°C for one hour, followed by water quenching (hereinafter called annealed material). The resultant microstructure consisted of a ferritic matrix with about 21% austenitic phase for the heat A, and 38% austenitic phase for the heat B. Two sets of samples were then subjected to ageing at 475°C for holding times of 25 and 200 hours. Some aged specimens were then subjected to a restorative treatment.

Vickers hardness (HV) was measured in the bulk material by using a microhardness tester, with an applied load of 1 kg. Tensile testing was conducted in an electromechanical machine, using clip-on extensometers to measure strain. Toughness parameters were determined, based on standard J integral and K_{IC} tests, using WOL (wedge opening load) specimens. A servohydraulic machine with computer control and COD (crack opening displacement) to determine crack length was used. Examination of fracture surfaces from tensile and fracture toughness testing was conducted in a JEOL JMS-6400 scanning microscope.

RESULTS

A reversion treatment of 550°C/1 hour was chosen as the most practical reversion condition. This selection was based on previous studies of the evolution of HV for the DSS studied here after ageing at 475°C during different holding times (Mateo *et al.* (5)). Table 2 displays the average HV values obtained after such a restorative treatment. From these results, a complete recovery seems to be induced. In order to verify the restoration degree found, tensile tests were also carried out. Yield stress (σ_{ys}), ultimate tensile strength (σ_{uts}) and elongation to fracture (ε_f) were measured. Table 2 shows the obtained results, as well as a parameter defined for evaluating the restoration phenomenon. This parameter, referred to as "restoration effectiveness", is given by:

$$\%\mathbf{R.E.} = \frac{\sigma_{ys,\,\mathrm{Aged}} - \sigma_{ys,\,\mathrm{Restored}}}{\sigma_{ys,\,\mathrm{Aged}} - \sigma_{ys,\,\mathrm{Annealed}}} \qquad (1)$$

for yield stress, and similar relationships are defined for the other measured mechanical parameters.

A more complete evaluation of the reversion treatment was conducted through fracture toughness testing. This time the condition of short ageing was excluded because the previous data had evidenced a quite similar tendency for 25- and 200-hour ageings. The obtained results for fracture toughness are given in Table 3. Where needed, values of K_J calculated from the expression $K_J = (E.J_{0.2/BL})^{1/2}$, were determined for comparative purposes.

Examination by SEM of the fracture surfaces of tensile specimens evidenced an absolutely different aspect of aged and restored samples. Fig. 1 allows to compare the brittle surface, with very little reduction of area, of an aged specimen, with the typical cup-cone aspect of a restored one. SEM studies on surfaces from fracture toughness testing lead to the same results observed for tensile specimens, *i.e.* brittle fracture of aged samples, associated with cleavage of ferrite grains, and ductile fractographic features in restored materials.

TABLE 2 - Hardness values, tensile tests results and effectiveness of the reversion heat treatment for material from heat A.

Heat Treatment	**HV**	**%RE**	**σ_{ys} (MPa)**	**%RE**	**σ_{uts} (MPa)**	**%RE**	**$\%\varepsilon_f$**	**%RE**
Annealed	238±11	-	328	-	541	-	27	-
Aged 25 h	327±8	-	842	-	1026	-	24	-
Aged 200 h	374±7	-	837	-	1161	-	12	-
Aged 25h+ Reversion	222±7	≅100	498	66.9	693	68.7	27	100
Aged 200h+Reversion	232±9	≅100	469	72.3	684	77.0	25	87

TABLE 3 - Fracture Toughness results and calculated effectiveness of the reversion heat treatment for material from heat B.

Heat Treatment	**$J_{0.2/BL}$ (KJ/m^2)**	**K_J (MPa.m$^{1/2}$)**	**K_{IC} (MPa.m$^{1/2}$)**	**% RE**
Annealed	197	193	-	-
Aged 200 h	-	-	43	-
Aged 200h + Reversion	114	147	-	69

DISCUSSION

The evolution of mechanical parameters with ageing confirms the well known fact of the susceptibility of DSS to "475°C embrittlement". The measured changes in room temperature hardness, tensile properties and fracture toughness are clear evidences of this substantial embrittlement. Examination by SEM reveals that the observed changes are correlated with promotion of brittle fracture by cleavage of ferrite with ageing. Substructural examination points out spinodal decomposition, as responsible for the observed transition from ductile to brittle behaviour (5).

The results found indicate that a heat treatment at 550°C for 1 hour allows to restore hardness to values very close to those obtained before ageing. On the other hand, if the reversion treatment is evaluated taken into account results from tensile and fracture toughness testing, restoration effectiveness is not completely satisfactory. This aspect should be related to a much stronger sensitivity of tensile and fracture toughness testing to the embrittlement phenomenon than that of hardness testing. At this point it must be reminded that hardness values are only empirical and qualitatively comparative measures of the resistance of a material to experience plastic deformation. Therefore, the physical meaning of hardness is much more restricted than that associated with other more fundamental parameters, *i.e.* yield stress, ultimate tensile stress and elongation to fracture. From a phenomenological viewpoint, the restoration process should be associated with the demodulation of the spinodal microstructure produced by the reversion treatment; although this is not totally clear from a preliminary TEM examination (5). It may be supposed that, after reversion at 550°C during 1 hour, the state of the samples is similar to that existing after the initial minutes of ageing at 475°C, stage where ageing-related changes are noticed markedly through fracture toughness evaluation exclusively. According to Golovin *et al.* (6), what happens in these initial stages of embrittlement is the formation of Cr-C-N clusters, which have a pinning action on the dislocation movement. This may be a very important aspect under conditions where bulk-like mechanical parameters are measured. However, this phenomenon is not clear and further research, mainly in terms of substructural changes, is needed in order to understand the correlation between changes measured in given mechanical parameters and the intrinsic restoration process.

CONCLUSIONS

Mechanical tests and microstructure analyses were conducted in order to investigate the possibility of recovering the degradation of mechanical properties produced by 475°C embrittlement of a DSS. It is concluded that a reversion treatment at 550°C for one hour leads to a virtual restoration of hardness to values of unaged material. However, tensile properties and fracture toughness are only recovered up to effectiveness values of around 70%. Such a difference may be related to the sensitivity of tensile and toughness testing to substructural features remaining from spinodal decomposition, and points out the use of these two methods for a more reliable evaluation of restorative treatments for aged DSS.

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REFERENCES

(1) Solomon H.D. and Devine T.M., Proc. Duplex Stainless Steels'82, ASM, 1982, pp. 693-756.
(2) Iturgoyen, L., Ph.D. Thesis, Univ. Politècnica Catalunya, 1994.
(3) Chung, H.M., *Int. J. Pres. Ves. & Piping,* Vol.50, 1992, pp. 179-213.
(4) Konosu S., *Scripta Metall. Mater.,* Vol.26, 1992, pp. 1631-1636.
(5) Mateo, A., Salán, N., Llanes, L. and Anglada, M., to be published.
(6) Golovin, I.S., Sarrak, V.I. and Suvorova, S.O., *Metall. Trans. A*, Vol.23A, 1992, pp. 2567-2579.

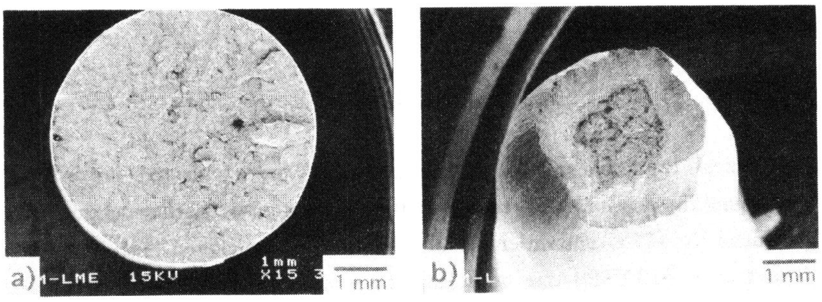

Figure 1. Fracture surfaces of tensile specimens.
a) Aged at 475°C/200 h, b) Aged at 475°C/200 h and restored at 550°C/1 h