THE EFFECT OF TEMPERATURE ON LOW TEMPERATURE AGING EMBRITTLEMENT OF "DUPLEX" STAINLESS STEELS

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In order to establish the accuracy of current analitical models that assume that low temperature aging of duplex stainless steels is a thermally activated process characterized by a unique parameter P, and also to determine the influence of ferrite content, temperature and time on aging embrittlement mechanisms a fracture toughness characterization program was carried out.

This program involved the analysis of two duplex stainless steels with different chemical composition and ferrite content, aged at low temperatures (280-400°C) between 300 and 14800 hours. Fracture toughness, microhardness and Charpy impact tests were performed in this analysis.

The results show an important loss of toughness with aging and also that embrittlement is lower at 280°C than over 350°C when compared based on the defined aging parameter P.

INTRODUCTION

Cast duplex stainless steels composed of austenite and ferrite are used extensively in the nuclear, oil and chemical industries because of several properties such as high strength, good weldability, superior resistance to stress corrosion cracking and resistance to hot cracking, due primarily to the presence of the ferrite phase in the duplex structure. Nevertheless, those duplex stainless steels are susceptible to embrittlement processes with aging at low temperatures (280-400°C), because of certain mechanisms such as spinodal decomposition of ferrite, precipitation of G phase in ferrite and $M_{23}C_6$ precipitation carbides at ferrite-austenite boundaries [1]. It has been shown that these precipitation mechanisms increase the ferrite hardness and decrease the impact toughness of these materials.

Aging phenomena are considered [2] as activated thermal processes based on an Arrhenius-type law that produced equivalent time-temperature (t, T) pairs between 280°C and 400°C expressed by

$$t_2 = t_1 \exp [U/R (1/T_2 - 1/T_1)] \qquad (1)$$

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The process activation energy U, has been empirically correlated [3] with the chemical composition by the equation:

$$U(\text{kcal/mol}) = 43.64 + 4.76\ \text{Si} + 2.65\ \text{Cr} + 3.44\ \text{Mo} \qquad (2)$$

Introducing an arbitrary aging parameter P [2], with a reference value of 1 equivalent to 10 hours aging at 400°C into Eq. (1) gives:

$$10^{P} = t \exp\left[U/R\ (1/673.2 - 1/T)\right] \qquad (3)$$

Using Eq. (3) accelerated aging can be carried out in the laboratory, at temperatures up to 400°C, the normal service environment temperature being 280°C.

An extensive program is being performed. Firstly, in order to determine if the aging processes are the same at both the service (280°C) and accelerated (350°C and 400°C) temperatures; secondly, to study the evolution of fracture toughness with aging and thirdly, to analyse the effect of ferrite on aging. This work summarizes some of the early results [4].

MATERIAL

Table 1 shows the chemical composition and the ferrite content of the two steels analysed in this work, identified as 12F and 18F as a function of ferrite content. 12F is a commercial CF8M duplex stainless steel taken from a valve aged in service for 10 years at 280°C and 18F is a similar duplex steel obtained from an experimental cast.

TABLE 1- Chemical composition and ferrite content of the studied CF8M steels.

STEEL	C	Mn	Si	Cr	Ni	Mo	%Ferrite
12F	0.035	0.70	1.10	18.6	10.4	2.00	12.2
18F	0.076	0.83	1.25	19.4	9.6	2.29	17.8

From the chemical composition the activation energy of each material was calculated using Eq. (2). The results were 17.5 and 21.6 kcal/mol for the 12F and 18F steels, respectively. Considering these activation energies, in-service aging of the 12F steel, has a parameter P of 3.71. Overaging treatments have been performed on this steel at 280°C, 350°C and 400°C, reaching up to P=4.30 after 14800 hours at 400°C. For the 18F steel different aging treatments at 350°C and 400°C have also been performed, up to a maximum value of P=4.00 at 400°C after 10000 hours.

EXPERIMENTAL

To analyse the effect of aging on these steels a complete mechanical characterization has been performed including microhardness, Charpy impact and fracture toughness tests.

Microhardness tests

Microhardness tests were carried out on both steels at the different aging steps. These tests were done in each phase using loads of 50 g on austenite and 25 g on ferrite during 20 seconds. Microhardness tests on austenite show no variation with aging. Figures 1a and 1b plot the mean values of 25 indentations on ferrite as a function of the aging parameter P, for both materials. These figures clearly show that the microhardness of ferrite increases with aging. Service aging the 12F steel at 280℃ (P=3.71) gives a much lower hardness for the ferrite than the corresponding aging at 350 and 400℃ for the 18F steel. This suggests different microstructural processes for both groups of treatments. Furthermore after service aging the 12F steel shows different results at 280℃ compared to 350℃ and 400℃.

Charpy impact tests

Standardized Charpy impact tests were carried out at room temperature. Figures 2a and 2b show the values of impact resistance, for both steels as a function of the aging parameter. The obtained results show an important embrittlement with aging for the two steels. From the results obtained for the 12F steel it can be observed that the effect of aging at 280℃ is different (much lower) compared with the effect of aging at 350℃ and 400℃ for the same aging parameter values. This suggests once again that the aging processes are different at the service and accelerated aging temperatures.

Fracture toughness tests

Fracture toughness was determined by J-integral R-curve, in accordance with the European Recommendations ESIS P1-92 [5], and following the unloading compliance single specimen method. 20 mm wide CT specimens were used, with 2 mm deep sidegrooves machined after the fatigue crack propagation. Figures 3a and 3b show the evolution of the most representative J_R curves with aging and Figures 4a and 4b show the values of $J_{0.2/BL}$ against P. In general these results show an important influence of aging on toughness. If the aging time increases there is a decrease of $J_{0.2/BL}$ for any temperature between 280℃ and 400℃. The evolution of toughness as a function of the aging parameter shows two different tendencies: one of low effect for the service temperature, 280℃, and another of higher effect at 350℃ and 400℃. From these results it is possible to conclude that fracture toughness testing is more sensitive to aging than the other forms of mechanical characterization used in this investigation.

Given that the observed aging effect at 280℃ is less than at 350℃ and 400℃ it can be assumed that the 12F steel has an initial state of P=0. With this assumption the new P values can be obtained and it can be seen that the plot showing the evolution of $J_{0.2/BL}$ against P is similar to that obtained for the 18F steel (see Fig. 5a). A similar situation can be seen for impact resistance (see Fig. 5b) and ferrite microhardness when plotted against the new P values.

Complementary to the fracture toughness tests a fractographic study was made using scanning electron microscopy. The fracture type of the as-received materials was very ductile caused by microvoids coalescence in austenite as is shown in

Figure 6a. On the other hand the general fracture form of the aged steels was similar to the as-received ones but an important number of brittle fractures appeared because of cleavage associated to the ferritic phase confirmed by microanalysis (EDS). Figure 6b shows an example of these cleavages.

CONCLUSIONS

Some precipitation mechanisms exist with aging that lead to hardening of the ferrite causing a loss of toughness in the steels, observed both in the impact tests and in the fracture toughness tests.

There is some evidence that aging mechanisms are different at the service temperature, 280℃, compared with the accelerated aging used in the laboratory. Therefore, it is not correct to extrapolate to 280℃ the results obtained with accelerated aging using thermally activated models.

Although there is no effect on microhardness and impact toughness with aging at 280℃ on the 12F steel, a decrease in the values of the fracture toughness resistance was observed, but was less important than the one obtained for equivalent aging at the other temperatures.

From the fractographic analysis it was observed that the tendency of the fracture path to follow the ferrite phase increased with aging. It has been established that embrittlement of ferrite has a direct influence on the loss of toughness in these steels.

According to these results, part of the future work should be supported by high resolution transmision microscopy techniques to differentiate precipitation processes at the temperatures used and then analyze their micromechanical effects.

ACKNOWLEDGMENTS

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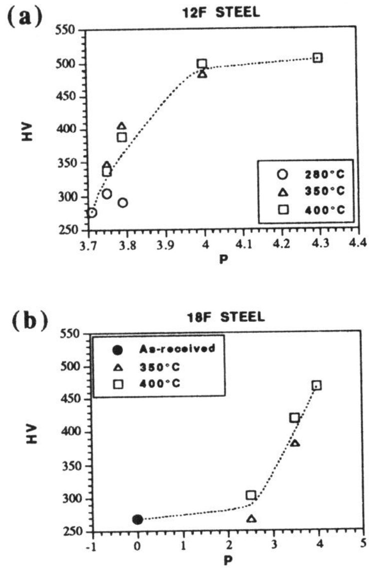

Figure 1. Evolution of ferrite microhardness with P: (a) 12F steel and (b) 18F steel.

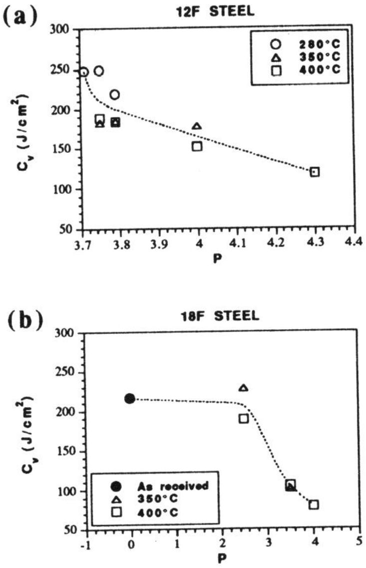

Figure 2. Evolution of impact resistance with P: (a) 12F steel and (b) 18F steel.

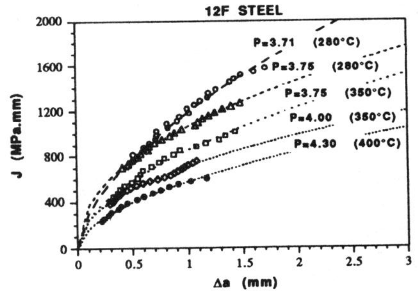

Figure 3a. Evolution with aging of J_R curves for the 12F steel.

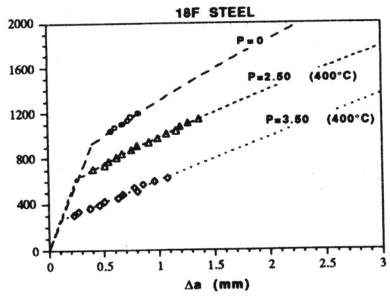

Figure 3b. Evolution with aging of J_R curves for the 18F steel.

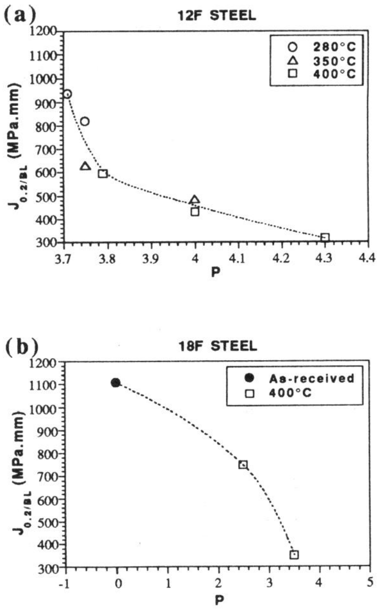

Figure 4. Evolution of $J_{0.2/BL}$ with aging: (a) 12F steel and (b) 18F steel.

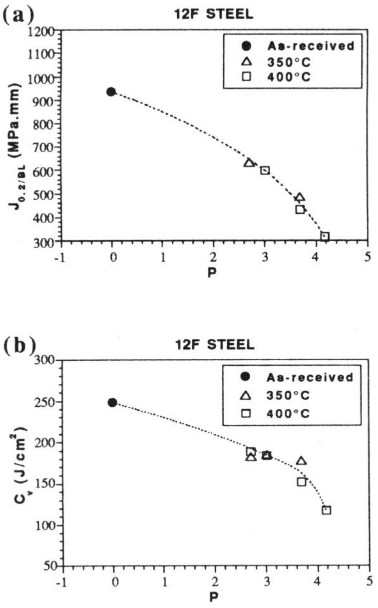

Figure 5. Evolution with the new P values of (a) $J_{0.2/BL}$ and (b) impact resistance, for the 12F steel.

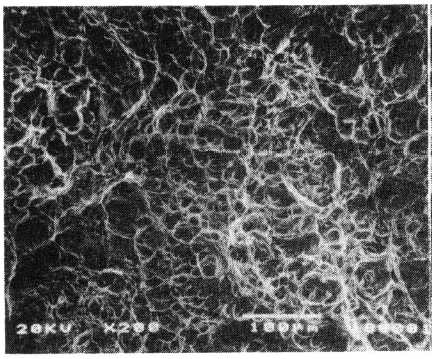

Figure 6a. Fractography of 18F steel as-received (P=0).

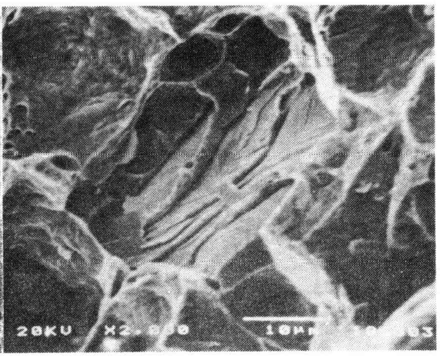

Figure 6b. Fractography of aged 18F steel (P=3.5).