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An attempt to correlate the change of the swept area ($\Delta A/b^2$) by mobile dislocations with the mechanism of the fatigue crack propagation rate (da/dN) in environmental hydrogen: Application in Inconel 690

Xinghong Luo[a,*], Mahmoud Habashi[b]

[a] CAS Key Laboratory of Nuclear Materials and Safety Assessment, Institute of Metal Research, Chinese Academy of Sciences, Shenyang, China
[b] C.N.R.S., France

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ABSTRACT

The thermally activated dislocation motion due to plastic deformation in Inconel 690 has been carried out by performing tensile tests in air and in environmental hydrogen using the strain relaxation technique. The results show that the variation of - $\delta(\Delta A/b^2)$ or $\ln(V_H/V_{Air})$ with ε_p depends on the nickel content in the alloy and the microstructure, and generally increases with increasing Ni% for low value of ε_p (tensile test). The maximum increase in the rate change was observed for the austenized and aged microstructure. On the other hand, fatigue crack propagation rate (da/dN) has been measured in the same environments. An attempt has been carried out to correlate the rate change - $\delta(\Delta A/b^2)$ or $\ln(V_H/V_{Air})$ with the variation of $(da/dN)_H$ against $(da/dN)_{Air}$ (the variation of $\boldsymbol{\Omega}$) and has shown that for the austenized and aged microstructure, $\boldsymbol{\Omega}$ is higher than those for stress relieved or only aged at 700 °C. These results are similar to those obtained by the thermal activation parameter – δ ($\Delta A/b^2$) or the ratio $\ln(V_H/V_{Air})$. The maximum values of – δ ($\Delta A/b^2$) and $\boldsymbol{\Omega}$ are obtained when the density of precipitates on the grain boundaries is higher than that in the matrix. In this case, intergranular fractures with fin slip lines features on the grain surfaces were observed. Finally, in the presence of environmental hydrogen, about the fatigue crack propagation rate, two relationships were established and expressed as: 1) in stages I and II: $(\Delta\varepsilon_p/2) = \Theta\,\Delta K + \Gamma$ and 2) only in stage II: $(da/dN)_{\mathrm{II}} = c_{\mathrm{II}}\,(\Delta\varepsilon_p\,/\,2)^{\lambda}$, where $\boldsymbol{\lambda} \approx m = 2$, the power factor in Paris' law.

1. Introduction

Nickel based Inconel 600 alloy has been widely used as the steam generator tubing material for pressurized water nuclear reactor units operating at about 300 °C. The resistance of this alloy to intergranular attack (IGA) and stress corrosion cracking (SCC) has been studied under various simulated environmental conditions using different techniques [1–3]. Many results showed that the chromium depletion due to grain boundary carbide formation during heat treatment was responsible for the degradation of this alloy. To minimize the chromium depletion associated with grain boundary carbide precipitation, Inconel 690 was developed with higher chromium content (up to 30%wt.) to substitute for Inconel 600 used in nuclear power plants. In the past years, many papers have been published about the resistance to IGA and SCC of Inconel 690 [4–6], which indicated that Inconel 690 had much better resistance than Inconel 600 alloy. However few studies have been reported on the hydrogen embrittlement (HE) resistance of Inconel 690 [7].

The objective of this work is to understand how environmental hydrogen influences the mechanical behavior of Inconel 690 alloy. For this purpose, a series of environment simulated tests, such as tensile test, strain relaxation test and fatigue crack propagation test, have been conducted on Inconel 690 in different heat treatment conditions. The aim is to have a comprehensive picture of the effect of environmental hydrogen and microstructure on the reduction of the swept area by mobile dislocations ($\Delta A/b^2$) (nano-scale), and the role of the environmental hydrogen induced at crack tip in the fatigue crack growth rate (da/dN) (macroscopic scale), of this alloy. Many other results are also supplied, especially in view of the change of fracture mode with different microstructure (microscopic scale).

An original attempt is made to correlate the change of swept area -$\delta(\Delta A/b^2)$ as a function of true strain ε_p, due to the interaction mobile of dislocations-hydrogen atoms, with the fatigue crack propagation rate $(da/dN)_H$ as a function of ΔK from a propagated fatigue crack tip.

* Corresponding author.
E-mail addresses: xhluo@imr.ac.cn, xinghongluo@hotmail.com (X. Luo).

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Table 1
Chemical compositions of Inconel 690 (%wt.).

Element	C	Mn	Si	Cr	Ni	Fe	Cu	Al	S
Content	0.026	0.10	0.16	30.25	59.31	9.54	0.04	0.33	< 0.001
Element	P	Co	Ti	N	Mo	Nb	Mg	B	–
Content	0.008	0.022	0.28	0.05	0.03	0.02	0.01	0.004	–

2. Experimental procedures

2.1. Material

The chemical compositions of the as received material used for all tests are listed in Table 1. Three heat treatment conditions were applied to investigate the responses to hydrogen of different microstructure:

1. as received (forged) in case of tensile specimens **(A).**
2. as received + heated at 500 °C for 15 h/water quenched (W.Q.) in case of fatigue specimens **(A′)**: "stress released heat treatment".
3. as received + aging at 700 °C for 15 h/W.Q (**B**) for tensile and fatigue specimens.
4. as received + annealing at 1050 °C for 1 h/ (W.Q) + aging at 700 °C for 15 h/W.Q. (**C**) for tensile and fatigue specimens.

To compare the effects of environmental hydrogen on mechanical properties, cathodic charging technique was employed by applying a constant cathodic current density $i_C = 0.1\ \mathrm{A\ m^{-2}}$ in an aerated solution: **1 N H_2SO_4 + 0.1 g/l As_2O_3**.

2.2. Tensile test

Sheet tensile specimens, 3 mm wide, 0.50 mm thick, and with a gauge length of 21.40 mm were machined from the as received materials, and then were heat treated in the three conditions (**A**, **B** and **C**) and mechanically polished for tensile tests. Tensile tests were performed using an Instron machine with strain rate ranging from 7.8×10^{-7} to 7.8×10^{-5}/s at room temperature. Half of the specimens were cathodic charged throughout the test. The other specimens were tested in air. During tensile tests both in air and in the presence of hydrogen, several strain relaxation tests, which lasted more than 4000 s, were performed in order to study the influence of hydrogen on the swept area by mobile dislocations ($\Delta A/b^2$, ΔA is the area swept out during thermal activation event, and b the magnitude of Berger' victor.) and the change of strain rate sensitivity parameter m'.

2.3. Fatigue crack propagation test

Fatigue crack growth test compact tension specimens, shown in Fig. 1, were machined from the as received materials (forged), and then were heat treated (**A′**, **B** and **C**) and polished for fatigue crack growth tests. The tests were performed on a hydraulic servo-valve MTS machine. One part of the specimens grooved on both sides were tested in air with frequency $f = 10$ Hz. The second part of the specimens not grooved have received cathodic charging during the test with $f = 0.1$ Hz. All the tests were conducted at room temperature with initial stress intensity factor $\Delta K \approx 15\ \mathrm{MPa}\sqrt{m}$ and load ratio $R = 0.35$. Due to the little volume of the available material, for each microstructure, one specimen was tested either in air or in the presence of cathodic polarization. Before the cathodic polarization, the fatigue crack was initiated in air with $f = 10\ Hz$ from $\Delta K = 15–16\ \mathrm{MPa}\sqrt{m}$.

2.4. Microstructure

The microstructure of Inconel 690 after the three different heat treatments are observed as follows:

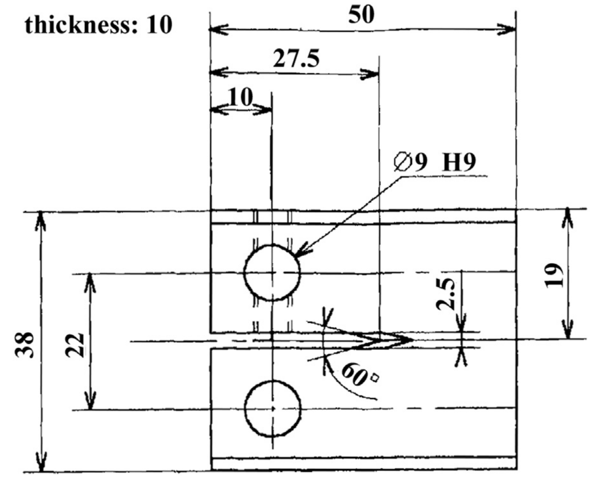

Fig. 1. Compact tension specimen used in fatigue crack propagation test. (Dimensions in mm.).

1) For the as received alloy (**A** and **A′**), high density of carbides can be observed essentially in the matrix. Some grain boundaries free of carbides were also found.
2) Compared with the as received microstructure, after aging at 700 °C for 15 h (**B**), less carbide precipitates in matrix and semi-continuous distribution of carbides on the grain boundaries can be observed.
3) After solution treatment and aging (**C**), large amounts of carbides precipitated essentially on the grain boundaries. The carbides $\mathbf{M_{23}C_6}$ [8] on the grain boundaries are distributed almost continuously.

The average grain size d is about 44 µm for heats **A** or **A′**, **B** and **C**.

3. Experimental results and discussion

3.1. Tensile test and mechanical properties

Tensile properties of the Inconel 690 alloy with three different microstructure tested in air and with cathodic polarization are listed in Table 2, the fitted mechanical properties following the relation $\sigma_{ys} = \sigma_0 + k\varepsilon^n$ are also presented.

The results obtained in air show that compared with as received condition (**A**), solution and aging heat treatment (**C**) evidently decreases σ_{ys} (yield strength) and increases ductility at failure ε_f with ***UTS*** (ultimate tensile strength) almost unchanged. While, aging at 700 °C for 15 h (**B**) slightly decreases σ_{ys} and increases ε_f, n values and ***UTS***, suggesting that some residual stresses exist in the as received alloy. On the other hand, cathodic hydrogen decreases ***UTS*** of the material, especially for heat treatments including aging. Furthermore, hydrogen significantly raises the n value with respect to that of the as received microstructure, and has little effect on those of the other two heats.

According to the collected data from the results of Abraham et al. [9] in 310 S steel and Lenartova et al. [7] in Inconel 600 and 690 under heat treatment condition **C**, a calibration curve, shown in Fig. 2, was established between σ_{ys} measured in the presence of internal hydrogen and the quantity of hydrogen Q_H introduced in the metal. It shows that for heat **C** σ_{ys} keeps almost unchanged at about 240 MPa with Q_H up to 46 ppm. Following this curve and the result from Table 2, the presumed Q_H introduced by environmental hydrogen could be about 100 ppm, which produces a hydrogen embrittlement index (loss in reduction of elongation), ***H.E.I.%***, of 85.5. However, our another work shows that for the same Inconel 690 and with heat treatment **C**, 115 ppm internal hydrogen produced 100% intergranular fracture without cracks (Fig. 3a), while the fracture type provoked by environmental hydrogen in this work was a mixture of severe intergranular cracks and embrittled matrix (Fig. 3b). Comparing the fracture features shown in Fig. 3a and