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To cite this version:
J. Armstrong, S. Carpenter. HYDROGEN-INDUCED CRACKING IN PURE IRON. Journal de Physique Colloques, 1985, 46 (C10), pp.C10-139-C10-142. <10.1051/jphyscol:19851032>. <jpa-00225416>

HAL Id: jpa-00225416
https://hal.archives-ouvertes.fr/jpa-00225416
Submitted on 1 Jan 1985

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HYDROGEN-INDUCED CRACKING IN PURE IRON

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Abstract - The modulus and internal friction of Armco iron were continuously measured during cathodic charging with hydrogen to investigate crack initiation and growth. The observed modulus decrease was attributed to crack initiation and growth. The internal friction increase during cathodic charging was attributed to plastic deformation accompanying the crack formation. Both the modulus and internal friction behavior were found to be a sum of two parallel exponential processes. The two exponential processes were consistent with different sources of carbon for the crack-producing hydrogen bubble nucleation.

I - INTRODUCTION

One of the most prominent effects of hydrogen in pure iron is the cracking observed near the outer layer of cathodically charged samples. The primary purpose of this study was to determine if continuous, in situ, modulus measurements could be used to investigate the initiation and growth of cracks in pure iron. In addition, continuous internal friction measurements, which were the by-product of the modulus measurements, were used to investigate the mechanisms involved in the hydrogen-induced cracking.

Typically, the modulus of a material should decrease with the initiation and growth of cracks or voids in the material. A theoretical treatment by Bristow (1) of the effective modulus of a material containing 'n' penny-shaped cracks per unit volume of average radius 'a' indicated such a decrease in modulus. Hydrogen-induced cracking in iron is localized to the surface. Thus the effective modulus of the total sample could reflect a sample which consists of a cracked outer layer with a solid inner core. A cylindrical sample of diameter 'd' with a cracked layer of depth 'Δd' has an effective modulus 'M' written as

\[ M = M_0 \left(1 - K_1 \frac{naΔd}{d}\right) \]  

where \( M_0 \) is the modulus of the uncracked core. The value of \( K_1 \) depends upon Poisson's ratio for the uncracked material and the mode of vibration. As the change in modulus is proportional to the change in resonant frequency, equation (1) may be rewritten as

\[ \frac{Δf}{f} = -K_2 \frac{naΔd}{d} \]  

(2)

Article published online by EDP Sciences and available at http://dx.doi.org/10.1051/jphyscol:19851032
where \( f \) is the initial resonant frequency, \( \Delta f \) is the change in frequency and the constant \( K_0 \) depends upon the material and the mode of deformation. Thus measured resonant frequency change should be directly related to measurable metallographic quantities.

II - EQUIPMENT

The diagram of the apparatus used to measure the resonant frequency and internal friction has been described elsewhere (2). The sample was driven in a standing wave at resonant frequency (\( \approx 40 \, \text{kHz} \)) by a Marx type (3) composite oscillator of 1/2 wavelength in a torsional mode of oscillation. The internal friction and vibratory strain amplitude were measured using techniques found in the literature (4,5). The center of the quartz bars and the sample were displacement nodes, thereby allowing electrical and support connections to be made without affecting the standing wavetrain. The torsional mode of oscillation was chosen as it was less susceptible to disturbance of the wavetrain while the sample was in the charging solution. The samples were simultaneously cathodically charged at current densities ranging from 10 to 30 \( \text{mA/cm}^2 \) in a 1N \( \text{H}_2\text{SO}_4 \) solution with trace amounts of \( \text{Al}_2\text{O}_3 \) and \( \text{CS}_2 \) added to enhance hydrogen uptake. A 3/2 wavelength \( \text{Al}_2\text{O}_3 \) buffer rod was placed between the oscillator and the sample. The solution was circulated around the sample and the temperature of the solution was maintained by a heat exchanger placed in a reservoir. Samples were machined to one-half wavelength from Armco iron. In all cases, the samples were annealed in a vacuum better than 10 torr for 6 hours at 500°C after machining and before cathodic charging. The samples were electropolished in \( \text{H}_3\text{PO}_4 \) at a current density of 60 \( \text{mA/cm}^2 \) prior to cathodic charging to provide uniform surface conditions.

III - EXPERIMENTAL RESULTS

Typical internal friction and sample resonant frequency behavior during cathodic charging of pure Armco iron at a current density of 30 \( \text{mA/cm}^2 \) at 6°C are shown in figure 1. In all cases, the resonant frequency decreased as a function of time. The decay was rapid during the first 20 minutes of charging, followed by a slow decrease for up to 900 minutes of charging. The internal friction increased significantly during the first 20 minutes, but tends to decrease for longer charging time. A micrograph of a sample cathodically charged a current density of 30 \( \text{mA/cm}^2 \) for 60 minutes at a temperature of 6°C is shown in figure 2. Note that the cracking was essentially intergranular and was localized to a layer near the surface.

The careful removal of the cracked outer layer of two samples cathodically charged for 5 and 480 minutes respectively resulted in the recovery of the resonant frequency loss observed during cathodic charging as shown in figure 3. The slight decrease in resonant frequency below its original value is most likely due to uncertainty caused by the necessary removal, machining and regluing of the sample.

Samples were charged for a variety of current densities, vibratory strain amplitudes, charging solution temperatures and charging times. All samples were subjected to metallographic analysis to determine the average crack radius, depth of cracking and number density of cracks. A plot the quantity \( \pi \Delta f / d \) (equation 2) versus the corresponding change in resonant frequency is given in figure 4. The data yield a fair linear fit, with a slope of \(-1.33 \times 10^3\) and a correlation coefficient of 0.822. The line was constrained to go through the origin as no frequency loss was observed without cracking. An increase in the charging current density, which increases the effective hydrogen pressure, affects the magnitude and kinetic behavior of the frequency decay (2).

IV - DISCUSSION

The resonant frequency behavior during cathodic charging appears to be an exponential decay. However, a careful analysis shows it is not a simple exponential decay. Analyzing the frequency behavior with a process known as 'subtraction of
tails' used in determining decay constants for mixed radioactive sources, the frequency was found to be parallel sum of a rapid and a slow exponential decay process. Regardless of charging time, current density, vibratory strain amplitude or charging solution temperature, a sum of two parallel processes was obtained. A typical frequency process is shown in figure 5. Likewise, the internal friction was found to be a sum of two parallel exponential processes as shown in figure 6.

Both processes have been shown (2) to be the result of the initiation and growth of cracks. Thus, a plausible explanation for the existence of a two part parallel process may be that the hydrogen bubbles responsible for the initiation and growth of cracks nucleate at different rates. Carpenter and Fawks (6) showed that annealed samples exhibited a more rapid, greater frequency decay than samples that were unannealed. As both samples were carefully machined from an ingot of iron not subjected to severe deformation, one can assume that the only difference was the source of grain boundary carbon on which the hydrogen bubbles would nucleate. The unannealed sample would show a majority of the carbon in the form of carbides while the annealed sample would exhibit more free carbon. A similar behavior was noted at elevated temperatures in steels by Westphal and Worzala (7).

V - CONCLUSIONS

There are a number of conclusions which may be made from this investigation. First, the method of continuous monitoring of the resonant frequency and internal friction of a material can be useful in detecting the presence of cracks in a material. Second, there is a definite relationship between the metallographic data and the modulus loss. In all cases, the resonant frequency and internal friction behavior were found to be parallel exponential in nature. The behavior was consistent with the nucleation of hydrogen bubbles at different rates on either grain boundary carbon or carbides.

VI - ACKNOWLEDGEMENTS

This research was funded by the U.S. Department of Energy, Basic Sciences Division.

VII - REFERENCES

Figure 1 - Typical resonant frequency and internal friction.

Figure 2 - Micrograph of cathodically charged Armco iron.

Figure 3 - Frequency recovery by removal of the outer cracked layer.

Figure 4 - Plot of $\frac{\Delta a}{d} \Delta d$ versus resonant frequency loss.

Figure 5 - Analysis of the two part parallel exponential frequency decay.

Figure 6 - Analysis of the two part parallel exponential damping behavior.