Effect of oxygen on the crack growth behavior of V–4Cr–4Ti at 600°C

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Abstract

Exploratory experiments were performed to evaluate the effect of oxygen on the crack growth response of V–4Cr–4Ti at 600°C under constant load. Tests were run in gettered argon, argon containing 2000 ppm oxygen, and laboratory air using fatigue pre-cracked compact-tension (CT) specimens. Crack growth was measured primarily by post-test fracture surface examination, but also by in-test compliance measurements. Crack growth rates measured in air and gettered argon were about $2–3 \times 10^{-3}$ mm/h at a stress intensity factor of about 40 MPa $\sqrt{m}$. The crack growth rate in argon with 2000 ppm oxygen was about 7×10^{-2} mm/h at the same stress intensity level. The crack growth rates were very sensitive to the stress intensity factor. Over a limited range of stress intensity values the crack growth rate in argon plus 2000 ppm oxygen appears to be power-law dependent on stress intensity with an exponent of about 8.9. The fracture mode in air and gettered argon was transgranular cleavage with 20–30% intergranular fracture. In the oxygenated argon environment crack growth occurred predominantly by transgranular cleavage. © 2000 Elsevier Science B.V. All rights reserved.

1. Introduction

The mechanical properties of vanadium alloys being developed for fusion power system structures are sensitive to interstitial impurities such as oxygen, nitrogen and carbon. Previous studies [1–7] have shown the profound effect these impurities have on the tensile and fracture behavior of these materials. Effects include loss of ductility, increases in the ductile-to-brittle transition temperature, and a propensity for mechanical failure by low-energy modes such as cleavage or grain boundary fracture. The severity of the effect largely depends on the concentration and distribution of interstitials in the microstructure, but the details of how properties are degraded are not well understood. Relatively few studies have been performed to evaluate the effect of environment on the sub-critical crack growth and fracture behavior of candidate structural materials [8]. The objective of the present investigation is to examine the effect of oxygen on the propensity for stress-assisted dynamic embrittlement in V–4Cr–4Ti.

Dynamic embrittlement is a type of brittle fracture that occurs by quasi-static decohesion, usually along grain boundaries, caused by the stress-driven diffusion of a surface-adsorbed embrittling element. This type of embrittlement has been observed in a number of alloy systems. Oxygen is found to embrittle nickel-base superalloys and intermetallics [9–11], as well as in a precipitation-strengthened Cu–Be alloy [12]. Tin will embrittle bronze [13] and in steels sulfur is the deleterious element [14]. Crack growth rates are between 0.1 and 100 mm/h which is many orders of magnitude slower than conventional brittle fracture. The growth rate is determined by the diffusivity of the embrittling species, the stress profile ahead of the crack tip (assumed to be time-dependent), and the concentration of the embrittling element needed to cause local fracture. Excellent review papers have appeared in recent years describing dynamic embrittlement and quantitative models based on a modification of Fick's second law of diffusion [15,16]. In the present paper, the results of exploratory experiments to study the effect of oxygen

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partial pressure and stress intensity level on the crack growth behavior of V–4Cr–4Ti at 600°C are presented.

2. Experimental procedure

Compact-tension (CT) specimens 30.5 mm wide by 3.8 mm thick were machined from warm-rolled V–4Cr–4Ti alloy (Heat No. 832665) plate in the T-L orientation. The CT specimens were cut according to the dimensions specified by the ASTM standard test method for plane-strain fracture toughness of metallic materials [17]. After machining, specimens were annealed at 1000°C for 1 h in a vacuum furnace at a pressure $\leqslant 10^{-7}$ Torr. This heat treatment produced a recrystallized microstructure with an average grain size of 25 µm. Specimens were fatigue pre-cracked in room temperature air and then tested at 600°C in an appropriate environment.

Three environments were utilized: (1) flowing (0.4–0.5 l/min) purified argon, (2) flowing argon doped with 2000 ppm oxygen, or (3) laboratory air. Table 1 summarizes the test conditions for the experiments performed to date. An oxygen gettering furnace was used to purify the argon gas before passing it once through the test chamber. The oxygen concentration of the purified argon leaving the gettering furnace was much less than ~1 ppm oxygen as measured by a zirconia-cell oxygen meter. To create an environment consisting of argon containing 2000 ppm oxygen a gas mixing apparatus was used. Details of how the gas mixing system operates have been described previously [18].

All experiments were performed in a controlled atmosphere furnace, with quartz windows on two opposite sides, attached to a rigid, electromechanically controlled, load frame. The CT specimens were loaded via Inconel 625 pin and clevis grips. These grips extended out of the furnace hot zone and were, in turn, connected to water-cooled, stainless steel loading rods inside the controlled atmosphere chamber. The loading rods passed through stainless steel bellows and were connected to the load frame.

The samples were loaded to a nominal constant load by cycling the load 1 N above and below a desired value at a crosshead speed of 0.05 mm/min. The load-line displacement of the specimen notch was measured with a
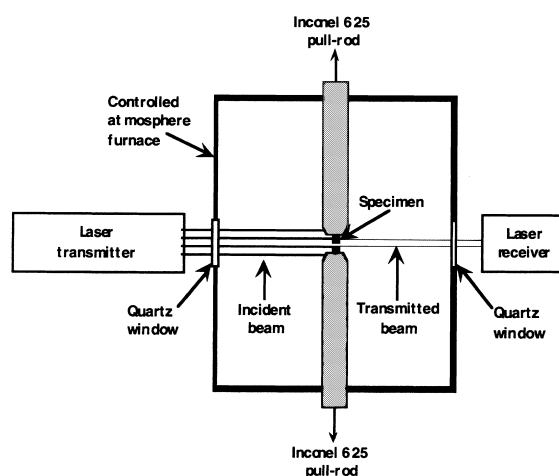

Fig. 1. Schematic of experimental test set-up for constant load crack growth tests.

laser extensometer, Fig. 1. The extensometer transmitter scanned a laser beam vertically through one of the quartz windows on the furnace. The extensometer receiver collected the resultant signal after it had passed through the furnace chamber. The sample was arranged so that the laser beam could only be transmitted through the notch of the sample. The resolution of the extensometer is 0.5 µm.

The load-line displacement and load were recorded once a minute by a computer data acquisition system. Periodically the specimen compliance was measured by unloading to 80% of the current load level. During unloads the data acquisition rate was increased to once a second. The specimen compliance was used as an indication of crack growth at a particular load level. At the end of a test the extent of environmentally induced crack growth was marked by causing additional fatigue crack growth in room temperature air. Specimens were then broken open and the fracture surfaces examined both optically to measure crack extension, and in a scanning electron microscope (SEM) to characterize fracture mode.

3. Results

Fig. 2 presents a representative plot of the load and load-line displacement as a function of time for specimen CT44-08 tested in argon plus 2000 ppm oxygen. The load-line displacement measurement includes displacements from several sources. Increasing displacement may be attributed to either crack growth or to creep of the material in the vicinity of the highly stressed crack tip. A decrease in load-line displacement may be caused by growth of an oxide film on the surfaces of the

Table 1
Test conditions employed

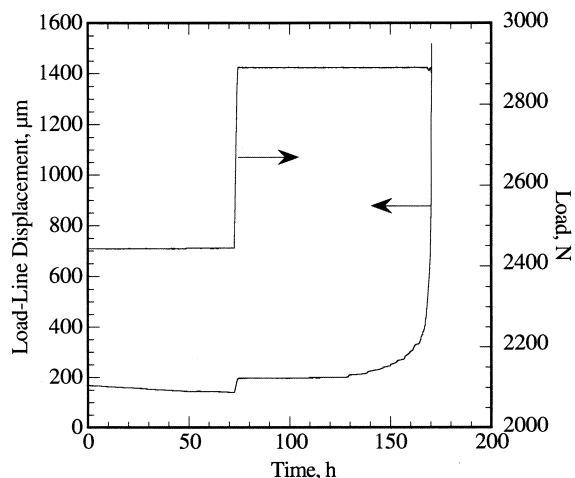| Specimen ID	Total test time (h)	Test environment
CT44-01	407	Gettered Ar
CT44-02	529	Air
CT44-05	241	Air
CT44-08	171	Ar + 2000 ppm O_2
CT44-09	212	Gettered Ar

Fig. 2. Time dependence of the applied load and load-line displacement for specimen CT44-08 tested in 2000 ppm oxygen at 600°C.

notch. All of these effects occurred during our tests. As noted in Section 2, the specimen compliance was measured in an effort to separate crack growth from specimen creep and oxide film growth. Reliable measurements of the specimen compliance were not obtained under low stress intensity and severe oxidation conditions. Build-up of oxide at the crack tip wedged the crack open preventing complete unloading. Thus, crack growth measurements were based primarily (but not entirely) on post-test visual and SEM examination of the fracture surface. These results suggest that another crack growth measurement technique, such as the DC potential drop method, may provide better crack growth monitoring capability. The dependence of the crack growth rate on stress intensity factor is shown in Fig. 3. It is evident from Fig. 3 that the crack growth rates in air and gettered argon are similar at a stress intensity of about 40 MPa $\sqrt{m}$. In contrast, the crack growth rate in argon with 2000 ppm oxygen is one to two orders of magnitude higher at similar stress intensity level and appears to be power-law dependent on the applied stress intensity. The data gathered under other conditions are too few to make conclusive statements about the dependence of the crack growth rate on stress intensity factor.

SEM fracture surface examinations were performed on all specimens. For specimens tested in air the fracture surfaces were heavily oxidized. Attempts to remove the scale by ultrasonic cleaning in an acetone bath were partially successful, but this treatment did not reveal the details of the fracture process since the surfaces were severely corroded. The oxide scale did not interfere with optical measurements of the extent of crack propagation, however, due to clear differences in surface
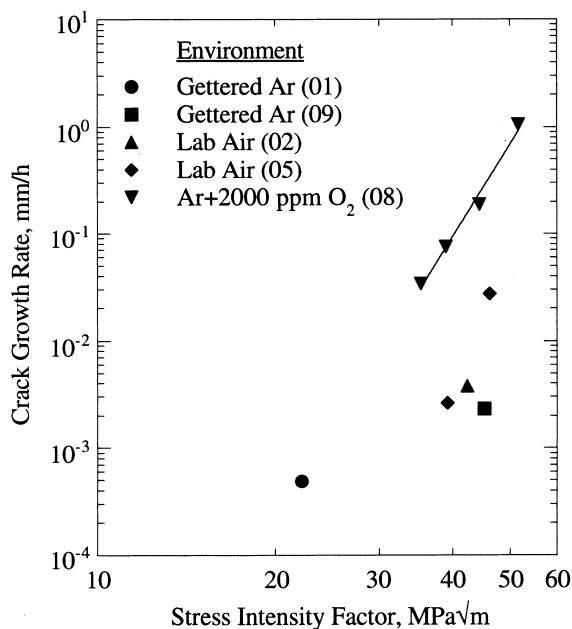

Fig. 3. Crack growth rate as a function of stress intensity factor for unirradiated V–4Cr–4Ti tested in gettered Ar, laboratory air, or Ar + 2000 ppm oxygen at 600°C.
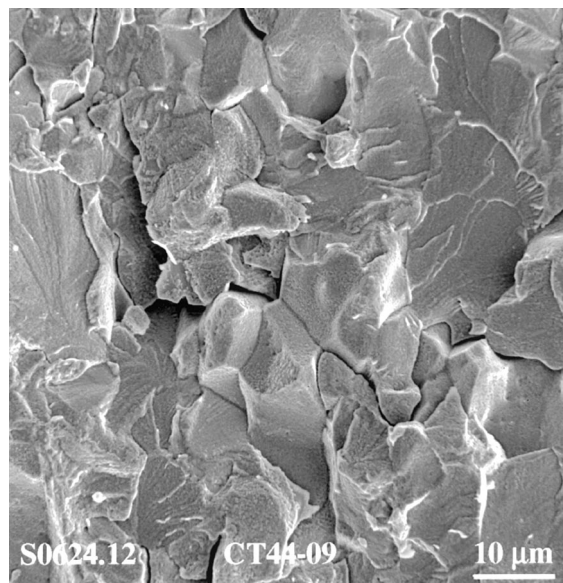

Fig. 4. SEM image of the fracture surface of specimen CT44-09 tested in gettered Ar at 600°C.

roughness between fatigue crack growth regions and environmentally assisted crack growth. On the other hand, the fracture surfaces of specimens tested in gettered argon or oxygen doped argon were less severely oxidized, so only brief ultrasonic cleaning was needed
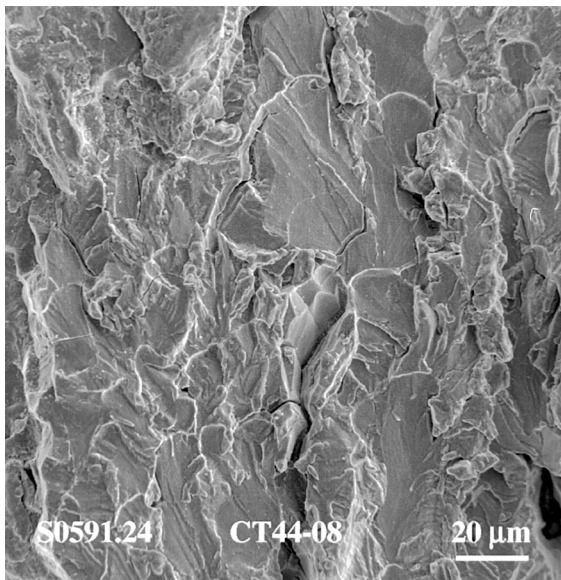
Fig. 5. SEM image of the fracture surface of specimen CT44-08 tested in Ar + 2000 ppm oxygen at 600°C.

prior to SEM examination. For these specimens it was not difficult to determine the fracture mode. Transgranular cleavage and intergranular fracture were the principal fracture features observed on specimens tested in gettered argon, Fig. 4. About 20–30% of the fracture features are considered intergranular in these specimens. For the specimen tested in argon containing 2000 ppm oxygen the fracture mode was predominantly transgranular cleavage with some evidence of tear ridges separating cleavage patches, Fig. 5.

4. Discussion

A clear difference in crack growth behavior was found for specimens tested in air or gettered argon compared to tests conducted in oxygenated argon. The diffusion coefficient of oxygen in V–4Cr–4Ti at 600°C is about 3×10^{-5} mm²/h [1]. The distance oxygen must diffuse in order to cause the observed crack growth rates may be computed from

$$x = \frac{D}{\dot{a}}, \tag{1}$$

where $\dot{a}$ is the crack growth rate in mm/h and D is the diffusion coefficient in mm²/h. For tests conducted in air or gettered argon the required oxygen diffusion distance is between 8 and 61 μm depending on the specific environment and stress intensity level. These diffusion distances are on the order of the grain size in this material. This suggests that the cracking process in air or gettered

argon is intermittent. In a low oxygen environment such as gettered argon a relatively long time is needed for oxygen to diffuse into the material and elevate the oxygen concentration to a value high enough to cause local fracture. It is interesting to note that the fracture mode observed during tensile testing of pre-charged specimens seems to differ from that found here. The primary fracture mode seen by Natesan et al. [3] and DiStefano et al. [2] following tensile testing was intergranular rather than transgranular cleavage. In contrast, Fujiwara et al. [5] observed transgranular cleavage in specimens exposed to air for 1 h at 500°C or 700°C. Air has been found to be a more aggressive oxidation environment than low partial pressure oxygen [2,3]. In air it appears that formation of a relatively thick oxide scale inhibits oxygen transport to the crack tip relative to the oxygen doped argon environment. For the argon with 2000 ppm oxygen the characteristic diffusion distance ranges from 0.03 to 0.4 μm. In other words, the depth of penetration is 10 to 1000 times smaller than the crack tip opening displacement. Thus, oxygen must be embrittling the material at or very near the surface. There is very little time for protective oxide film to form, as should be the case for a continuous cracking process. These results indicate that under certain conditions of stress intensity and oxygen partial pressure the near surface region is easily embrittled and rapid crack growth occurs.

5. Conclusions

The crack growth rate in gettered argon and laboratory air was similar at a stress intensity factor of about 40 MPa $\sqrt{m}$. The crack growth rate in argon containing 2000 ppm oxygen was more than one order of magnitude faster than for tests run in laboratory air or gettered argon at similar stress intensity factors. The crack growth rates were very sensitive to the stress intensity factor. Over a limited range of stress intensity values the crack growth rate in argon plus 2000 ppm oxygen appears to be power-law dependent on stress intensity with an exponent of about 8.9. The fracture mode in air and gettered argon was transgranular cleavage with 20–30% intergranular fracture. In the oxygenated argon environment crack growth occurred predominantly by transgranular cleavage.

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