INTERIM REPORT

INVESTIGATION OF MOISTURE-INDUCED EMBRITTLEMENT OF IRON ALUMINIDES

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MASTER

ABSTRACT

The effect of ambient air the tensile and fatigue behavior of an Fe₃Al, Cr type intermetallic alloy is examined as a function of test temperature. Hydrogen due to moisture in the air is found to be a major cause of embrittlement. The rates and mechanisms of the observed embrittlement appear to be temperature dependent. In addition, the alloy was found to have no notch sensitivity.

INTRODUCTION

Iron aluminides are currently being studied for use in applications where low cost, relatively low weight, and excellent corrosion resistance are desired. The main disadvantage of the alloys is low room temperature ductility in air due to moisture that causes hydrogen embrittlement [1-5]. This investigation examines changes in tensile and fatigue behavior of an Fe₃Al,Cr type intermetallic compound in environments of various hydrogen contents and at various temperatures. The alloy was studied in two ordered states, the DO₃ and the B2. The DO₃ is a highly ordered superlattice, while the B2 is a superlattice of imperfect order.

EXPERIMENTAL MATERIALS AND PROCEDURE

Material

The material used in this study, designated FA129, was supplied by Vinod Sikka of Oak Ridge National Laboratory and was composed of 28.6 at.%Al, 4.8%Cr, 0.21%C, 0.5%Nb, balance Fe. Material fabrication and annealing conditions to obtain B2 and DO₃ order are described in ref. [4].

Fatigue Crack Growth

Fatigue tests in laboratory air at temperatures of 150°C, 300°C, and 450°C have been performed on DO₃-ordered FA129, and at 150°C on B2-ordered FA129. The compact tension specimens for these tests measured 25.4mm x 24.4mm x 5.1mm thick with a machined notch of 60° angle and 0.127mm radius cut by electro-discharge machining techniques. Test parameters are 20 Hz frequency, sawtooth waveform and a minimum-to-maximum stress ratio ($\sigma_{\min} / \sigma_{\max}$) of 0.2.

A load shedding routine was introduced in which the stress intensity is gradually decreased to allow measurement of crack growths rates near the threshold stress intensity. Test results with and without load shedding were compared. Results from these tests are compared with previously reported data [2]. In addition, crack opening displacement measurements were performed on one fatigue crack growth test using an extensometer attached to bolts set in the front face of the compact tension specimen. The results of these measurements will be used to determine if crack closure may be affecting the crack growth behavior.

Effect of Annealing Temperature

The effect of annealing temperature on recrystallization and fatigue crack growth in B2-ordered FA129 has been examined. Previous work had been performed on material which

received a 1 hour anneal at 700°C, resulting in a microstructure which was only 50% recrystallized. New material was prepared with a 2-stage anneal, the first at a higher temperature for recrystallization, and the second at 700°C to obtain B2 order. As the annealing temperature was increased to 900°C, the degree of recrystallization increased to approximately 90%. A specimen with the 900°C anneal has been fatigued to examine the change in behavior as compared to material with the 700°C anneal.

Microscopy

Fracture surfaces were examined under an AMR-1000 scanning electron microscope (SEM). In addition, the fatigue zones of the compact tension specimens were examined under a 300 kV Phillips transmission electron microscope (TEM).

TEM foils were prepared from the fatigued regions of the compact tension specimens parallel to and just under the fracture surface. The foils were mechanically ground to 150 μm , and electro-thinned until perforated. Electro-thinning conditions were as follows: an electrolyte of 6% perchloric acid in methanol at 100 mA and -20°C for 30 seconds.

RESULTS

Fatigue Crack Growth

Figures 1 and 2 show the da/dN curves generated for DO₃ and B2 material, respectively, in air at various temperatures. Table II lists the fracture mode for each test condition and summarizes the threshold and critical stress intensities, as well as the constant m of the Paris equation,

$$da/dN = C(\Delta K)^m.$$

The fracture surface of the B2-ordered material fatigued at 150°C displays a dimpled appearance suggesting microvoid coalescence. There also are displayed wide, irregularly spaced striations with a cleavage character as seen in Fig. 3.

Figure 1. Fatigue crack growth at elevated temperature in DO₃-ordered FA129.

Figure 2. Fatigue crack growth at elevated temperature in B2-ordered FA129.

TABLE II - Fatigue Crack Growth Data for FA129 in Air

<u>Condition</u>	<u>Temperature</u> <u>(°C)</u>	<u>Fracture Surface</u>	<u>m</u>	ΔK_{TH} <u>(MPa√m)</u>	ΔK_C <u>(MPa√m)</u>
B2	25	TG, many striation	9.4	30	44
B2	150	Dimpled, few striations	5.3	32	65
DO ₃	25	Mixed, few striations	22.8	19	24
DO ₃	150	-	0.8	26	52
DO ₃	300	-	3.1	25	49
DO ₃	450	-	8.7	21	48

Figure 3. Fracture surface of B2-ordered FA129 fatigued in air at 150°C.

The change in crack growth curves measured by employing a load shedding procedure was found to be slight. Figure 4 shows a comparison of curves for B2-ordered material tested with and without load shedding. Crack opening displacement (COD) measurements yield a linear COD versus load curve, indicating no crack closure, as seen in Fig. 5.

Annealing Effects

Optical micrographs of the grain structure of FA129 with the 700°C anneal, and the 900°C + 700°C anneal are shown in Figs. 6 and 7, respectively, where the increase in degree of recrystallization and grain size can be clearly seen. The fatigue crack growth curve in air at room temperature in each of these conditions is shown in Fig. 8.

Figure 4. Effect of load shedding on fatigue crack growth behavior of B2-ordered FA129

Figure 5. Crack opening displacement at various crack length, a , for B2-ordered FA129 in 25°C air

Transmission Electron Microscopy

In fatigued DO₃-ordered FA129 much higher dislocation densities were observed at elevated temperatures than at room temperature, and large arrays or networks of dislocations were common. Figure 9 shows a typical low dislocation density region, with the dislocations paired, in a DO₃-ordered fatigue specimen tested in air at 25°C. Figure 10 shows a network of dislocations in a DO₃-ordered fatigue specimen tested in air at 450°C. More detailed microscopy is underway on both B2 and DO₃-ordered FA129.

Figure 6. Grain structure of B2-ordered FA129 with 700°C anneal.

Figure 7. Grain structure of B2-ordered FA129 with 900°C anneal.

Figure 8. The effect of annealing temperature on fatigue crack growth in FA129 in 25°C air.

DISCUSSION

In the B2-ordered material fatigued in air at 150°C (as compared with 25°C), a decrease in crack growth rate, an increase in fracture toughness, and a predominantly dimpled fracture surface is observed, which are consistent with an increase in intrinsic toughness of Fe_3Al as temperature increases. A similar increase in fracture toughness is seen between DO_3 -ordered material tested in air at 25°C and 150°C. However, as the temperature was increased further there was an unexpected result: the fracture toughness decreased slightly and growth rates and stage II slopes increased dramatically.

Figure 9. Dislocation array in DO₃-ordered FA129 fatigued in air at 25°C.

Figure 10. Dislocation array in DO₃-ordered FA129 fatigued in Air at 450°C.

The increase in toughness at 150°C may be due in part to a decrease in the embrittling effect of hydrogen at high temperatures, as well as the increased intrinsic toughness. At 150°C, increased oxidation rates or a change in the oxidizing species (H₂O to O₂) may provide a more protective oxide layer at the surface. Another effect of temperature is that hydrogen diffusion in the matrix becomes a significant mode of transport, and build-up of hydrogen at grain boundaries or slip planes no longer occurs, resulting in a more uniform hydrogen distribution and no excessively weakened regions.

The increased crack growth with further increases in temperature in DO₃-ordered material may be explained by lower yield strengths and elastic modulus, or oxygen-induced embrittlement. More detailed examination of fracture surfaces and dislocation structures must be performed in order to explain this observation.

There was a moderate effect of annealing temperature on crack growth rates which cannot be explained at this time.

FUTURE WORK

Work planned for the next year includes:

- Perform high temperature tensile tests to determine yield strength behavior as a function of temperature.
- Continue high temperature fatigue crack growth tests for FA129 in vacuum at elevated temperature.
- Confirm notch-sensitivity tests which were previously performed, utilizing thicker, electropolished specimens with a higher stress concentration factor.
- Continue examination of fracture surfaces of high temperature fatigue crack growth specimens via scanning and transmission electron microscopy.
- Extend crack growth tests at room temperature, in air and in oxygen, to low Al contents (around 16 at.% Al).

CONCLUSIONS

Fe₃Al,Cr undergoes an increase in crack propagation rate and a loss of fracture toughness in moisture-bearing environments. Crack closure effects do not appear to explain any of the test results. TEM and SEM analyses of microstructure and fracture surfaces were consistent with the change in fracture toughness with order and environment. Elevated temperature affects the degree of embrittlement in a complex manner, possibly changing the rates of several of the processes involved.

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