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SCANNING ELECTRON MICROSCOPE FRACTOGRAPHY OF A
URANIUM-7.5 WEIGHT PERCENT NIOBIUM-2.5
WEIGHT PERCENT ZIRCONIUM ALLOY

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BY

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INTRODUCTION

A fractographic study of the uranium-7.5 weight percent niobium-2.5 weight percent zirconium alloy was initiated to gain a better understanding of this relatively new material. Brittleness and cracking were encountered in some worked material, and an anomalous behavior was observed in a series of double cantilever beam specimens.

The original fractographic studies of this alloy were performed using standard replica techniques in a transmission electron microscope. However, shortly after acquisition of a scanning electron microscope it was found that this instrument was faster, more direct, and supplied more information concerning the overall fracture surfaces of the alloy than the transmission electron microscope. Thus, the entire fractographic study of this alloy was transferred to the scanning electron microscope.

STUDY OF THE ALLOY SPECIMENS

DUCTILE MATERIAL SPECIMENS

Tensile specimens of alloy usually fracture predominantly in a dimple rupture mode, as shown in the upper left fractograph in the first slide (Slide 1). Second-phase precipitates resembling those found in some maraging steels are frequently observed in the pits and sides of the dimples. Examples of these particles are shown in the upper right fractograph (of the same slide). A view of these particles at higher magnification appears in the fractograph at the bottom left of the slide. Fractographic studies indicate that the particles are not well dispersed in the alloy and tend to segregate. Considerable difficulty has been experienced in identifying these inclusions. Electron microprobe studies indicate they are zirconium rich, possibly zirconium nitride.

DOUBLE CANTILEVER BEAM SPECIMENS

As part of a program to provide fracture-toughness data on the alloy in various environments, special double cantilever beam specimens were machined and stressed. The sketches on the next slide describe the specimen shape and how it is placed in tension (Slide 2). As shown on the left side of the slide, grooves are machined into a seven-inch rectangular bar, a two-inch slot is cut by electrodischarge techniques along the groove at one end, and holes are drilled above and below the slot. The sketch at the right shows diagrammatically how the specimen is stressed in the tensile machine.

-A-

-B-

-C-

DIMPLE RUPTURE FRACTURE IN DUCTILE URANIUM -
7.5% Nb - 2.5% Zr ALLOY (HEAT TREATED AT 800° C,
WATER QUENCHED, AND AGED FOR ONE HOUR AT
150° C)

- A - PREDOMINANTLY DIMPLE RUPTURE
- B - AREA WITH HIGH CONCENTRATION
OF INCLUSIONS
- C - DETAIL OF INCLUSIONS

SLIDE 1

A

B

C

A - SIDE VIEW
B - PERSPECTIVE
C - IN TENSION

DOUBLE CANTILEVER BEAM TENSILE SPECIMEN

SLIDE 2

A natural crack formed by a preload is used to initiate slow crack growth. For these alloy specimens, the load was applied in a 1,000-pound testing machine until the crack speed was approximately 0.002 inch per minute, then the machine was turned off while the strain was maintained. As the crack propagated, the load decreased correspondingly. Values of the fracture toughness parameter, K_{Ic} , can be measured in various atmospheres by means of this type of specimen.

As illustrated by the sketch in the upper right portion of the next slide (Slide 3), several of the test specimens broke perpendicular to the prenotched path, after fracturing only part way along the restraining groove. Visual examination of the resulting fractures revealed bright and dark areas. The bright areas corresponded to the fracture in the direction of the notch and also to the final overload areas; the dark areas were observed where the fracture appeared to change from the prenotched path to the perpendicular break.

Fractographic examination of both bright areas revealed the dimple fracture mode characteristic of the ductile alloy. The fracture mode of the dark areas is best defined as a form of quasi cleavage. Examples of this quasi-cleavage mode are shown in the next slide (Slide 4). Note the deep perpendicular crack in these fractographs, all of the same area at various magnifications.

In studying these transition areas with the scanning electron microscope, several rather dramatic montages were made, two of which appear in the next two slides. (Slide 5). In this area the crack was propagating from the dimple area at the top

SLIDE 3

QUASI-CLEAVAGE FRACTURE AT THE DARK AREAS
NEAR THE PERPENDICULAR BREAK IN A DOUBLE
CANTILEVER BEAM SPECIMEN

SLIDE 5

to the quasi-cleavage area at the bottom of the slide. In the next slide (Slide 6), the fracture was changing from the quasi-cleavage mode at the lower portion of the fracture to a dimple overload at the top.

STRESSED SPECIMENS

Test coupons stressed by bending in moist air and under distilled water have exhibited mixed modes of fracture, with a predominance of intergranular or grain-boundary cleavage. The failure time of coupons stressed in water or moist air is generally measured in thousands of hours; the presence of chloride ions drastically reduces failure time. In all environments, intergranular fractures are observed if water is present.

Some of the various types of grain facets observed in these intergranular fractures are shown in the next slide (Slide 7). There are relatively clean facets, as shown in the top left; there are mottled facets, as seen in top right; and there are facets with cleavage step markings, bottom left. On the next slide (Slide 8), there are other formations frequently found, including: deep cracks between grains, upper left; areas leading toward the quasi-cleavage mode, upper right; and areas showing intermittent transgranular quasi cleavage, bottom left. In almost every stress corrosion fracture examined, an overload area of dimple fracture mode was observed where the final fracture occurred.

BEND-FATIGUE SPECIMENS

One of the more interesting fractographic studies performed with the alloy involved the fracture resulting from a prenotched, low-cycle bend-fatigue specimen. The

SLIDE 6

-A-

-B-

-C-

GRAIN FACETS OBSERVED IN INTERGRANULAR FRACTURES

- A - CLEAN FACETS
- B - MOTTLED FACETS
- C - FACETS WITH CLEAVAGE MARKING

SLIDE 7

-A-

-B-

-C-

GRAIN FORMATIONS OBSERVED IN THE INTER-
GRANULAR FRACTURES

- A - DEEP CRACKS BETWEEN GRAINS
- B - AREA LEADING INTO QUASI CLEAVAGE
- C - INTERMITTENT QUASI CLEAVAGE

SLIDE 8

upper left fractograph of the next slide (Slide 9) shows characteristic fatigue striations. In the case of this bend fatigue, note that the striations are generally parallel rather than in the characteristic chevron pattern normally observed in tensile-fatigue fractures. The deep, perpendicular cracks seen in the lower fractographs were frequently observed at inclusion sites. The overload portion of this specimen was produced by impact loading and was in the characteristic dimple mode.

FAILURES

Two cases of brittle failure in worked material were investigated in considerable detail. In the first specimen, the fracture mode of all areas studied was intergranular with a predominance of extremely clean facets. In fact, as may be seen in the next slide (Slide 10), the facets are so clean we refer to this sample as our example of "classic intergranular fracture".

The other fracture, a large crack, started in a cold-worked area. Study of this fracture indicated that a crack had been initiated by stress corrosion in moist air. The fracture mode was predominantly intergranular. However, the corrosion rate appears to have been quite slow; and, as the crack propagated, some grains fractured in the quasi-cleavage mode, as may be seen in the next slide (Slide 11).

CONCLUSIONS

The use of the scanning electron microscope for this fractographic study instead of conventional replica techniques in the transmission electron microscope not only shortened the time necessary to obtain the data, but also furnished information

-A-

-B-

FATIGUE FRACTURE MODES

- A - FATIGUE STRIATIONS
- B } PERPENDICULAR CRACK AT
- C } THE INCLUSION SITES

-C-

SLIDE 9

-A-

-B-

-C-

INTERGRANULAR FRACTURE OBSERVED IN
THE CRACKED ALLOY

SLIDE 10

-A-

-B-

-C-

MIXED INTERGRANULAR AND QUASI-CLEAVAGE
FRACTURE OBSERVED IN THE CRACKED ALLOY

SLIDE 11

concerning the deep cracks which might have been difficult to obtain or even lost in the replica technique.

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