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NOTES FROM AN OPEN SESSION OF THE SUBCOMMITTEE ON MICROSTRUCTURE AND FRACTOGRAPHY

DEFENSE METALS INFORMATION CENTER
BATTELLE MEMORIAL INSTITUTE
COLUMBUS, OHIO

PREFACE

On March 10, 1964, the ASTM Subcommittee on Microstructure and Fractography of the Special ASTM Committee on Fracture Testing of High-Strength Metallic Materials held an open session at the Naval Research Laboratory in Washington, D. C. Six subcommittee members and visitors gave semiformal talks describing recently completed studies and progress on continuing studies. Because of the timeliness of the subject matter, W. R. Warke, Secretary for the Subcommittee, has prepared these notes for the Defense Metals Information Center.

Since much of the information contained in these notes is recent and unpublished, it should not be quoted or referenced without approval of the respective speakers.

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Director

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I. Stress Corrosion of Austenitic Stainless Steel

N. A. Nielsen, E. I. du Pont de Nemours & Company, Wilmington, Delaware

The problem of stress corrosion of austenitic stainless steel in the presence of stress and chloride ions has been under study for a considerable length of time, but is, as yet, not fully understood. Nielsen has studied this form of cracking at DuPont by two techniques. The early stages of cracking are observed by replicating the surface of the specimen, extracting the corrosion product in the incipient and minute microcracks. Crack propagation is studied by extraction replica electron fractography. In both of these techniques, oxide replicas were employed. The surface to be replicated was heat tinted in a sodium nitrate-potassium nitrate bath at 350-400 C and the resulting oxide layer, with corrosion product in situ, was stripped in methyl alcohol-bromine solutions.

Residual stresses produced during mill-annealing of Type 316 stainless steel were sufficient to start and propagate stress corrosion cracks in specimens placed in boiling 42% $MgCl_2$. The first of the above types of studies has shown that cracking began by the breakdown of passivity at points along slip traces and that corrosion proceeded to form tunnels filled with corrosion product along the slip planes. These tunnels then spread and joined to form microcracks. When the crack surfaces were examined fractographically, facets covered with "river markings" which looked like cleavage facets were found.

Nielsen also showed some fractographs of stress-corrosion cracks in some cold-drawn Type 304 stainless steel tubing which had cracked in boiling 42% $MgCl_2$ due to residual stresses. In this material, the fracture looked like a mixed intergranular and cleavage fracture. A mixture of smooth, clean facets and facets with river markings was seen. The exact nature of these facets, the nature of the river markings, and their relationship with the mechanism and direction of propagation are not understood at the present time; further study of this commercially important problem is needed.

II. Studies of Unidirectionally Solidified Eutectic Alloys

R. Hertzberg, Lehigh University, Bethlehem, Pennsylvania

Three systems were included in this program.

System	Volume Per Cent of Constituents	Second Phase Form	Second Phase Size,	Growth Rate, cm/hr
Cu-Cr	98Cu-2Cr	Rods	0.25-0.75	1-10
Al-Ni	$90Al-10Al_3Ni$	Rods	0.5-2	1-18
Al-Cu	$50Al-50CuAl_2$	Plates	2	2

Cu-Cr Alloys

Chromium fibers extracted from unidirectionally solidified ingots had ultimate tensile strengths in excess of 10^6 psi and elastic strains greater than 2.5 per cent. The unidirectionally solidified material did not exhibit any enhancement in properties over those obtained on randomly solidified material. The lack of increase in strength was attributed to the small percentage of the rods or fibers of chromium. Fracture of these alloys occurred along shear bands oriented at 45 degrees to the tensile axis. Fractographic examination revealed elongated dimples on the fracture surfaces, and it was shown that fracture occurred by the chromium fibers fracturing and nucleating voids in the copper which grew and coalesced by shearing.

Al-Ni Alloys

Significant increases in strength were obtained when the alloys in this system were unidirectionally solidified. Tensile strengths on the order of 43 to 44 ksi were observed. The properties of the fiber-reinforced alloys were related to the elastic properties of the fibers, and elastic and plastic properties of the matrix and the volume percentages of the two phases. Discrepancies between the observed and predicted properties were not large and were explained on the basis of premature fiber failure at sites of grown in defects. Fractographic examination of specimens of Al-Ni alloy indicated that the fracture mechanism was one of void coalescence. The fibers fractured in a brittle manner nucleating voids which grew and joined to produce both large equiaxed and elongated dimples on the fracture faces. A fractured fiber could be observed in each dimple.

Al-Cu Alloys

This alloy solidified as alternate platelets of aluminum and $CuAl_2$. Strength and ductility in bending and fracture appearance were studied as a function of platelet orientation. Outer fiber stress at fracture decreased continuously as orientation was varied from 0 to 90 degrees while ductility went through a maximum at 30 to 45 degrees and dropped off sharply above and below this range. The following crystallographic relationships were found to exist between the Al and $CuAl_2$ plates:

$$(111)_{Al} \parallel \text{interface} \parallel (211)_{CuAl_2}$$

$$[110]_{Al} \parallel [210]_{CuAl_2}$$

At orientations near 45 degrees, slip occurred without running into $CuAl_2$ platelets and ductility was therefore high. At low angles of orientation, the $CuAl_2$ platelets fracture in a brittle manner and the aluminum then either necks down or shears off. At high angles of orientation increasing amounts of delamination were observed.

III. Observations on Fatique Fractures
A. J. Brothers, General Electric Company, Schenectady, New York

Brothers is interested in fractography as a failure analysis tool and is primarily concerned with fatigue failures. His studies are, therefore, directed toward obtaining information which would be useful in examining such failures.

Some of the work Brothers had done was on tempered martensitic materials such as Type 412 stainless steel and Ni-Mo-V rotor forging steel. In these materials, which were at relatively low hardness levels, striations were evident and easily observable. The density of the striations (lines per inch) as a function of stress level has been determined for a number of these materials. It is hoped from these data to be able to determine the approximate stress level of service failures.

Austenitic materials fatigued at elevated temperature have also been examined using electron microscopic fractography and two observations were made. First, the striations in these materials tend to be straighter than those in the ferritic steels. Secondly, flat facets are occasionally observed in these materials which have the appearance of cleavage facets. Brothers pointed out that this observation of "cleavage" facets, together with that reported earlier by Nielsen, demonstrate the need for clarification of terminology which exists in the field of fractography.

IV. Replication Techniques and Recent Studies
W. R. Warke, Battelle Memorial Institute, Columbus, Ohio

Some work was done in an effort to develop a high-fidelity nondestructive replication technique for use in fractographic studies. These experiments included attempts to use cadmium and B_2O_3 as a sublimable and a water-soluble parting layer, respectively. Because of limited success and since, with sufficient experience, existing techniques proved adequate, these experiments were suspended.

A number of fractographic studies were described which included:

(1) A comparison of brittle fractures in coarse-and fine-grained molybdenum, in which intergranular cracking was found to act as Griffith-Orowan cracks in nucleating catastrophic failure of the specimens.

(2) An unusual fracture in a center-cracked sheet tensile specimen which fractured by cleavage from one end of the center crack and dimpled rupture from the opposite end. The material was electron-beam-melted columbium.

(3) Fracture studies of a series of five cast irons.

(4) An observation of cleavage in a face-centered cubic nickel-base alloy (IN-100).

(5) A study of the effect of three nondestructive testing penetrants on fracture appearance in steel and aluminum in which no etching, pitting, or other evidence of corrosion was seen.

(6) Fractographic studies of impact fractures in chromium over a wide range of temperature and of tensile fractures in chrome-rhenium alloys.

V. Fatigue Striation Formation and Fracture of Epoxy Resins

C. Laird, Ford Scientific Laboratory, Dearborn, Michigan

Laird described some specimen sectioning experiments intended to demonstrate the mechanism of formation of ripples or striations on the fracture surface of fatigue cracks in ductile materials. As tensile stresses are applied across the crack tip, the material slips on planes at 45 degrees to the crack plane and the crack tip blunts. Upon reversal of the stress, the material slips in the reverse direction to bring the newly created surfaces together. As the result of the geometry of blunting and flow, the crack length is increased by an amount related to the radius of curvature of the blunted crack, and a trough or groove is left on each surface at the crack tip.

Studies were made of the proportion of the cycles of load applied which were spent in propagating the crack by the above mechanism. It was found that at high stress or strain (i.e., few cycles to failure), the number of striations could be as many as 3/4 of the number of cycles applied, and that striations existed right up to the origin. At low stresses (many cycles to failure), a much smaller proportion of the total life was spent in propagation by striation formation; the ratio of the number of striations to the number of cycles to failure was less than 0.1. Under these conditions, a large portion of the lifetime was spent in slip nucleation.

Some experiments on fatigue in vacuo were reported. No difference in fracture surface morphology was seen between specimens fatigued in air and in vacuum. If the lifetime in air was less than 1000 cycles, no change in lifetime was observed. However, at longer lifetimes, fatigue in vacuum gave longer lifetimes than fatigue in air.

Laird also reported some work by C. E. Feltner on the fractography of epoxy resins. Bright circular facets associated with inclusions were found to initiate fracture in these materials. The fracture stress was found to vary inversely with the area of the shiny facet to the one-fourth power; these facets were acting as Griffith cracks. Fatigue can also grow an origin for catastrophic failure in epoxy resin and failure will again occur in accordance with an $A^{-1/4}$ rule.

VI. Fractographic Studies at Frankford Arsenal

C. M. Carman, Frankford Arsenal, Philadelphia, Pennsylvania

Carman described a study concerned with a comparison of the fracture toughness of commercial and high-purity 7075-T6 aluminum alloy. The fracture toughness, K_{Ic}, of the commercial purity material was 34,700 psi $\sqrt{in.}$, while the high-purity material, which had low iron and silicon contents, had a fracture toughness of 50,800 psi $\sqrt{in.}$ Krafft has recently proposed the following relationship:

$$K_{Ic} = E\sqrt{2\pi d_T}\, n$$

where E is Young's modulus, d_T is called the process zone size, and n is the strain-hardening exponent. The strain-hardening exponent was measured, and d_T was calculated for the two materials:

Material	n	d_T
Commercial	0.0788	2.85×10^{-4}
High purity	0.0832	5.40×10^{-4}

Since d_T is the distance from the crack tip at which tensile instability occurs, it was expected that the size of the dimples on the fracture surface would more or less correspond to d_T. Dimple sizes were measured on electron microfractographs and values of 0.2×10^{-4} inch and 0.4×10^{-4} inch were obtained for the commercial and high-purity materials, respectively. The values are in about the right ratio but differ from d_T by an order of magnitude.

Some results on steel slow-notched bend specimens were reported in which a material with a K_{Ic} of about 30,000 psi $\sqrt{in.}$ was found to have fractured intergranularly. The steel had been quenched and tempered at 475 F, and it was brought out that this temperature was in the 500 F embrittlement range which is known to be associated with intergranular fracture.