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CORROSION FATIGUE OF HIGH STRENGTH AIRCRAFT STRUCTURAL MATERIALS — CRYSTALLOGRAPHIC DEPENDENCE OF FRACTURE PATH IN AL-Zn-Mg (7075) ALLOYS

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WRIGHT-PATTERSON AIR FORCE BASE, OHIO 45433

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Low cycle corrosion fatigue tests were conducted in a variety of environments to quantitatively demonstrate the environmental effects on high strength aluminum (7075-T6) alloys. Environments used included argon, dry air, distilled water, and 3.5% sodium chloride solution. Crack growth rates were found to vary by almost two orders of magnitude, from 10^{-4} to 10^{-6} inches per cycle, with argon and dry air the lowest and sodium chloride solution the highest. Scanning electron micrographs clearly showed the transition from ductile to brittle failure going from inert to aggressive environments.

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FOREWORD

This Final Technical Report was prepared by the Fatigue Research Group of the Department of Materials Science and Engineering of the Massachusetts Institute of Technology, Cambridge, Mass., under Contract F33615-72-C-1288. The report covers work conducted from 01 July 1972 through 30 September 1974. The manuscript was submitted on 30 September 1976. The authors are R. E. Stoltz and Dr. R. M. Pelloux.

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SECTION I

INTRODUCTION

The Al-Zn-Mg alloy (7075) is a high strength age hardening aluminum alloy used predominately in aircraft applications. Extensive work has been done to study the mechanical properties of the alloy, especially its fatigue behavior and its stress corrosion susceptibility. It has been shown that a mildly corrosive environment can markedly affect its time dependent properties. More specifically, the fatigue strength in a high humidity environment, or in water with or without halides, is much lower than in a dry air environment. Figure 1 shows the extent to which the environment can affect the fatigue crack growth rates. Tests in purified argon with less than 20 ppm water vapor show the lowest crack growth rate at low ΔK levels while tests in air with 200 ppm water vapor show a somewhat higher growth rate. The crossover in the curve vs. ΔK curves is due to an uneven plane strain to plane stress transition in argon. In distilled water, the crack growth rates are higher than in argon and a marked corrosion fatigue crack acceleration is observed with a 3.5% NaCl environment. Since fracture mechanics and ideal plasticity suggests that the growth rate per cycle should be approximately half the crack tip opening displacement, the line giving the calculated crack tip opening displacement (CTOD) vs. stress intensity gives an indication of the theoretical environment free growth rate.

SECTION II

DISCUSSION

The growth rate measurements reported in Figure 1 were macroscopic rates, measured by observing a running crack in a thin plate. The microscopic growth rates are given by the striation spacings measured on the fracture surface. The striation spacing as well as the morphology of the fatigue striations are strongly dependent upon the environment. In the early 1960's, two distinct types of fatigue striations were observed and differentiated[1]: ductile striations which form in an inert or dry environment, and brittle striations which are strongly promoted by the presence water and of halide ions. Figure 2 shows typical ductile striations which are characterized by their smooth rumpled appearance, and the uniform advance of the fracture front over the width of a single grain. The fracture process resulting in ductile striations is dominated by shear flow and blunting at the fatigue crack tip.

By rapidly changing the environment one can see the strong effect this has on the fracture surface features. Figure 3 is taken from the fracture surface of a sample tested in air with a NaCl solution added without interrupting the test. The ductile striations are at the bottom of the micrograph. With the NaCl addition the striation features instantly change to a brittle morphology. The striation spacing increases approximately five-fold and river markings suggesting a cleavage fracture run perpendicular to the striation front. In Figure 4, at low magnification, the crystallographic dependence of the brittle striations can be seen. Large flat areas

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[1] C.A. Stubbington, P.J.E. Forsyth, J. of Metals, 90, (1961-62), 347.

corresponding to the existing grain structure are sharply inclined to one another. Efforts to determine the orientation of the fracture plane, however, have not proved successful. Details of the brittle striations are shown in Figure 5. Small tongue-like features extend from one striation step to the next. The crack front is highly irregular across a single grain, indicating a local cracking and "unzipping" process across the front. Also, the fracture surface between the striations is generally very flat, further evidence of a crystallographic mode of fracture.

Since the two types of striations are so distinct they can be used to compare the effects of various environments on the microprocesses of corrosion fatigue. The effects of a rapid change from air to chloride solution on the surface features has been shown in Figure 3. Early workers[2,3] also found that in a chloride solution the striation morphology is strongly affected by the magnitude and the direction of an applied current. In order to exploit this behavior the following test was performed: while undergoing cyclic stresses in a 3.5% NaCl solution, a plate of 7075 was alternately polarized anodically and cathodically against a platinum counter electrode. The stress cycling rate was adjusted so as to impress a 0.5 ma anodic current for 20 stress cycles and a reverse 0.5 ma cathodic current for 10 stress cycles. The current was applied to an exposed sample area of 0.125 square inches. The rest potential of the sample was -0.820 vs. SCE. The applied current polarized the sample to a cathodic potential of -1.400V and an anodic value of -0.700V.

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[2] P.J.E. Forsyth, E.G.F. Sampson, RAE Tech. Report 65158, (August, 1965).

[3] R.M. Pelloux, "Fracture 1969", Second International Conference on Fracture, Brighton, England, April, 1969, 731.

The fracture surface was analyzed by scanning electron microscopy and the effects of the alternating current were striking. In Figure 6, each light and dark band represents the anodic and cathodic stress cycling period. The relative contrast of each band is related to its orientation with respect to the SEM detector. The overall features depend on the grain orientation in that some grains do not contain any light and dark bands, and in some the band contrast is reversed. At higher magnifications, Figure 7, the nature of the light and dark regions becomes clear. At each current reversal the orientation of the fracture surface changes, giving a "stair-step" fracture surface. In some grains the detail of the plateaus can be seen (Figure 8). Twenty ductile striations and ten brittle striations can be counted, indicating that a small shift in corrosion potential anodically, from -0.820 to -0.700 can markedly change the fracture mode. It was surprising to observe that the ductile striations occur under a cathodic potential of -1.400V. It was expected that a cathodic potential would minimize the corrosion fatigue affect, but the reverse is true in this alloy. Additional features can be seen at still higher magnification (Figures 9 and 10). The transition from ductile to brittle is very smooth while that from brittle to ductile is very irregular, confirming our earlier observation on the nature of the crack front advance in both cases. With proper orientation of the specimen in the SEM (Figure 11), the true striation spacing can be measured. The ductile striations are approximately half as large as the brittle, giving a two-fold reduction in the local growth rate under anodic conditions. Tilting experiments indicate that the angle between brittle and ductile regions is approximately 160^{o} for the grain shown in Figure 8. It appears that the brittle striations follow a crystallographic fracture plane, possibly a (100) or (110), while the ductile striations form on a general plane normal to the tensile axis. The "stair-step" behavior is best seen in grains with the proper crystallographic orientation. Further experiments with large grain material are planned in order to determine the precise orientation of the fracture plane.

Additional tests have been performed in order to understand the electrochemical processes occurring under anodic polarization. Addition of a sodium nitrate inhibitor to a sodium chloride solution causes the striation morphology to revert from brittle to ductile (essentially the opposite effect of Figure 3). Testing in an anodizing solution of sodium carbonate plus sodium chromate also results in ductile striations, demonstrating that a mildly oxidizing environment at the crack tip is beneficial for corrosion fatigue.

SECTION III
MECHANISM

The mechanism of corrosion fatigue in this alloy can only be inferred. Since brittle striations tend to form in distilled water, chlorine ions are not critical, though they do accelerate the process. One possible explanation is that an anodizing environment causes a uniform oxide to form which allows the uniform dispersal of shear deformation around the crack tip and a smooth advance of the fracture front. The halide or hydroxyl ions in the NaCl solution promote a local attack along the fracture front, leading to the "unzipping" process mentioned above. Alternatively, a hydrogen mechanism could be operating, whereby hydrogen liberated under mildly cathodic conditions can diffuse into the material and, in turn, affect the fracture mode.

SECTION IV
CONCLUSION

This research technique which combines the detailed study of the fracture features following the rapid variation of macroscopic corrosion parameters appears to be extremely useful in corrosion research. Micro-features on the fracture surface provide a permanent recording of the dynamic fracture processes that occur at the crack tip. Changes in load, stressing frequency, and environment which result in changes in fracture surface features can be coupled with the observations by SEM and replica microscopy in order to isolate the exact mechanisms involved at the crack tip.

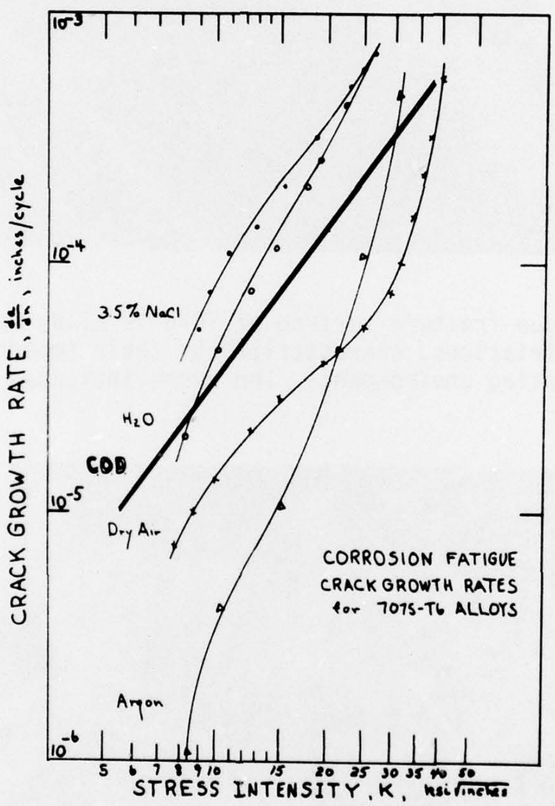

Figure 1. Corrosion fatigue crack growth rates, da/dn, vs. stress intensity factor, ΔK for a 7075-T6 alloy. Testing environments were inert argon (20 ppm water vapor), dry air (200 ppm water vapor), distilled water and 3.5% NaCl solution.

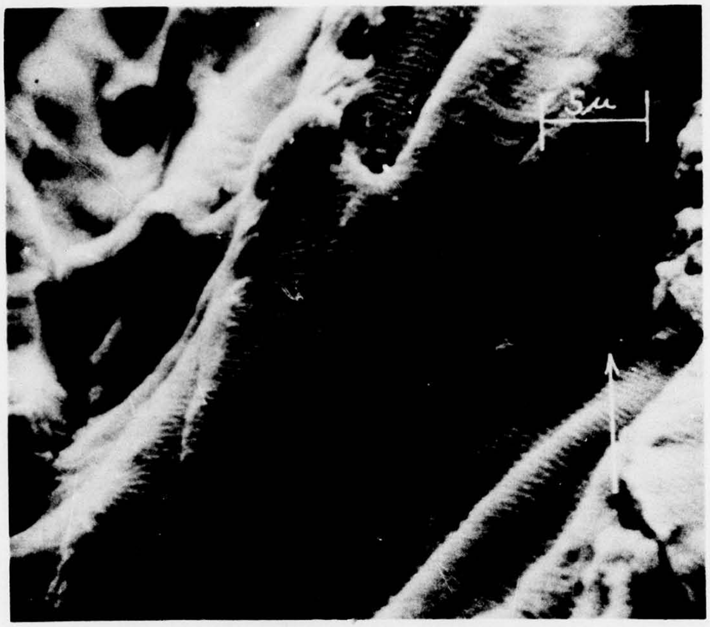

Figure 2. Fatigue fracture surface of 7075-T6 alloy tested in laboratory air. Ductile striations, characterized by their smooth fracture front, form in this testing environment. The arrow indicates the crack propagation direction.

Figure 3. Abrupt transition from ductile to brittle striation morphology in 7075-T6. Fatigue testing was begun in air with a 3.5% NaCl solution added during the test. The dotted lines demarcate the brittle striation width, approximately five times larger than the ductile striation spacing.

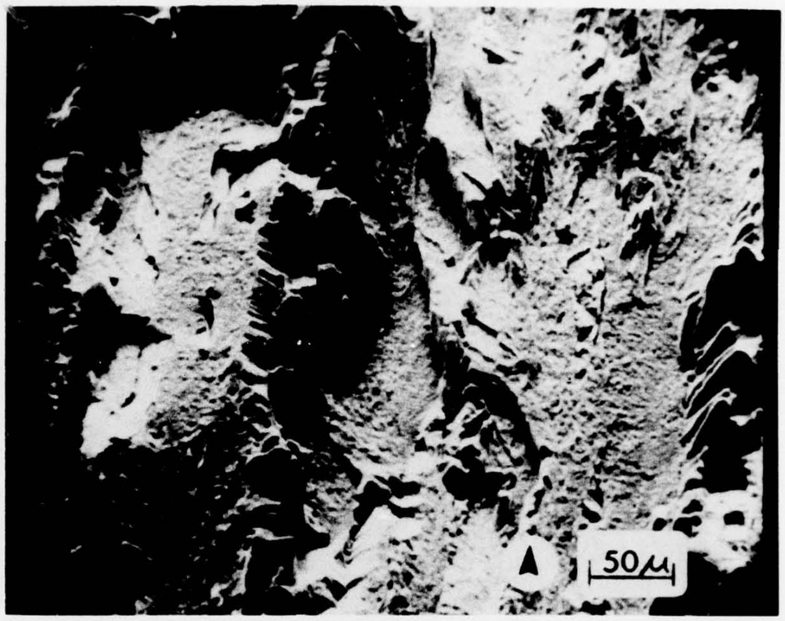

Figure 4. Low magnification micrograph of 7075-T6 tested in 3.5% NaCl solution. Fracture surface orientation changes from grain to grain, indicating a strongly crystallographic fracture mode.

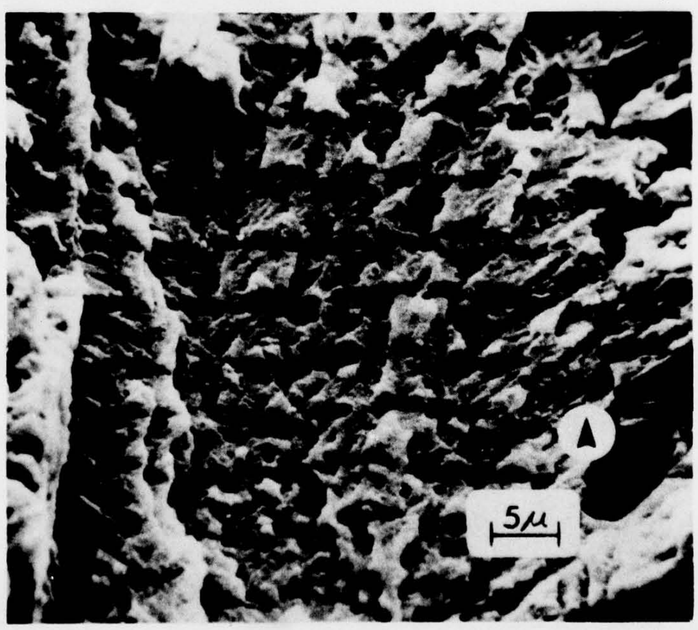

Figure 5. Brittle striations formed in 7075-T6 in a 3.5% NaCl solution. The fracture front is highly irregular, with small features extending across each striation step and river markings in the direction of crack propagation.

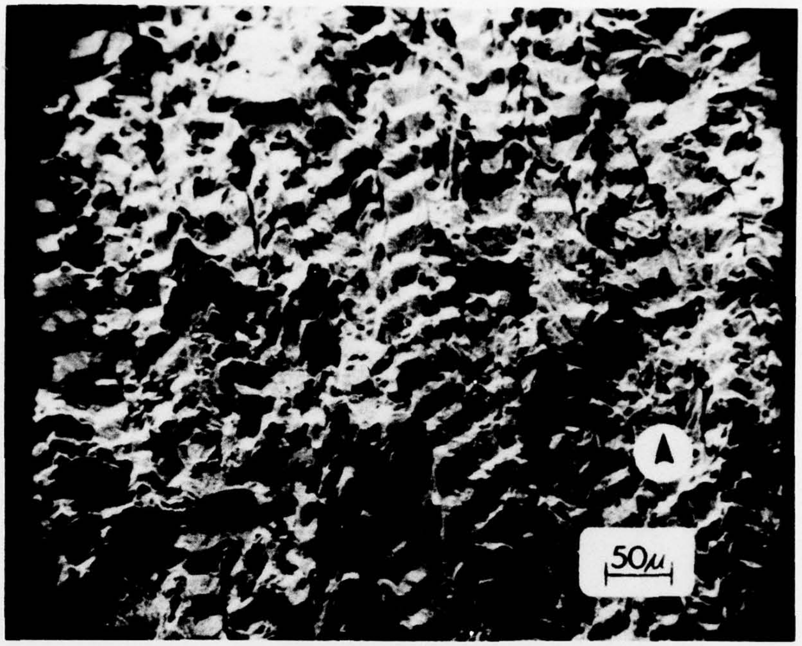

Figure 6. Fracture surface of 7075-T6 tested in a 3.5% NaCl solution with an alternating 0.5 ma anodic and cathodic current. Light and dark bands, due to fracture surface orientation differences occur in selected grains.

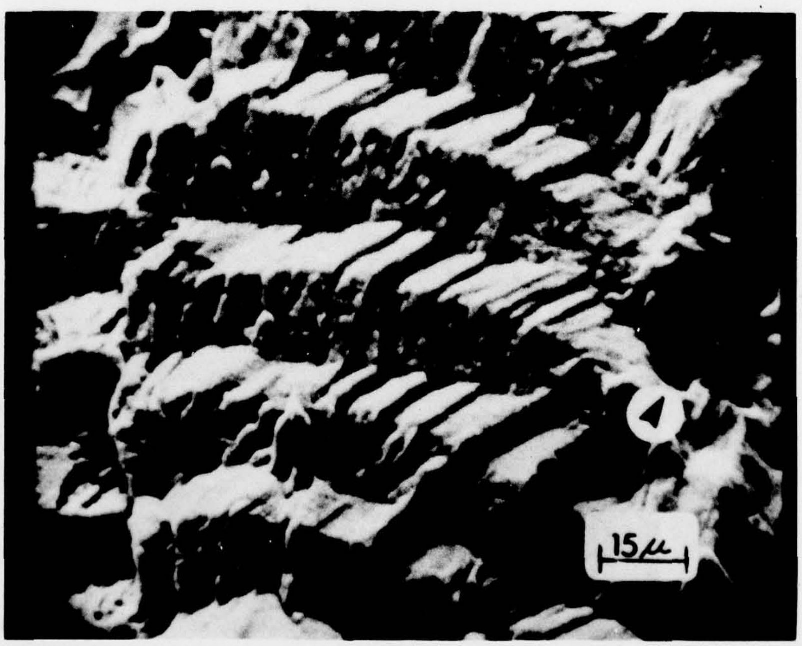

Figure 7. "Stair-step" nature of fracture surface in 7075-T6 due to anodic-cathodic current reversals.

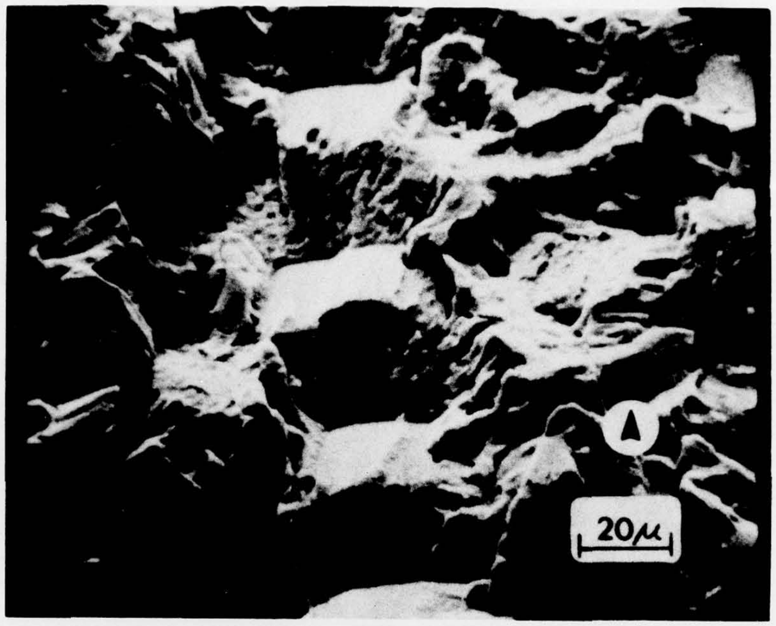

Figure 8. Details of fracture surface plateaus in 7075-T6 tested under alternating current condition. Twenty ductile striations form under an anodic potential of -0.700V vs. SCE while ten brittle striations form at -1.400V cathodic. The contrast effects indicate a sharp change in fracture surface orientation at each current reversal.

Figure 9. High magnification micrograph of Figure 8. Ten brittle striations are shown, corresponding to the ten stress cycles applied under -1.400V cathodic conditions.

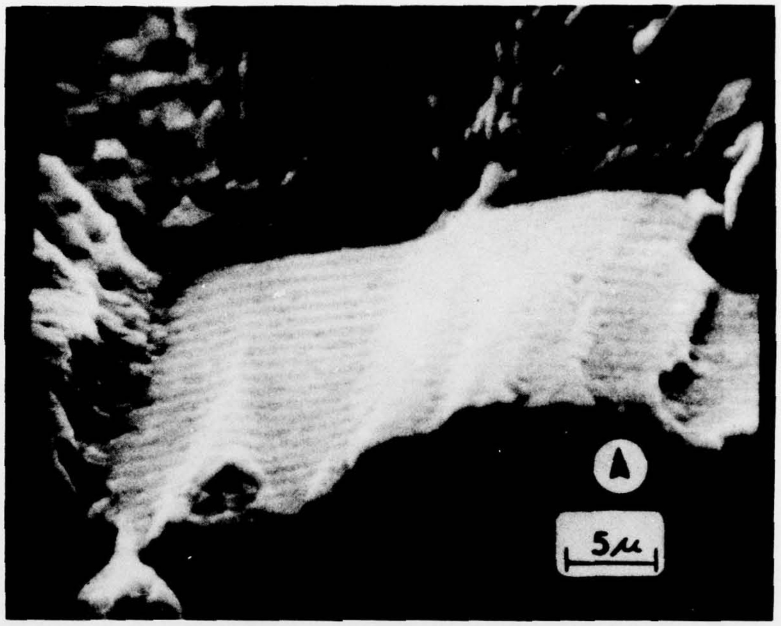

Figure 10. High magnification micrograph of Figure 8. Twenty ductile striations are shown, corresponding to the twenty stress cycles applied under -0.700V anodic conditions.

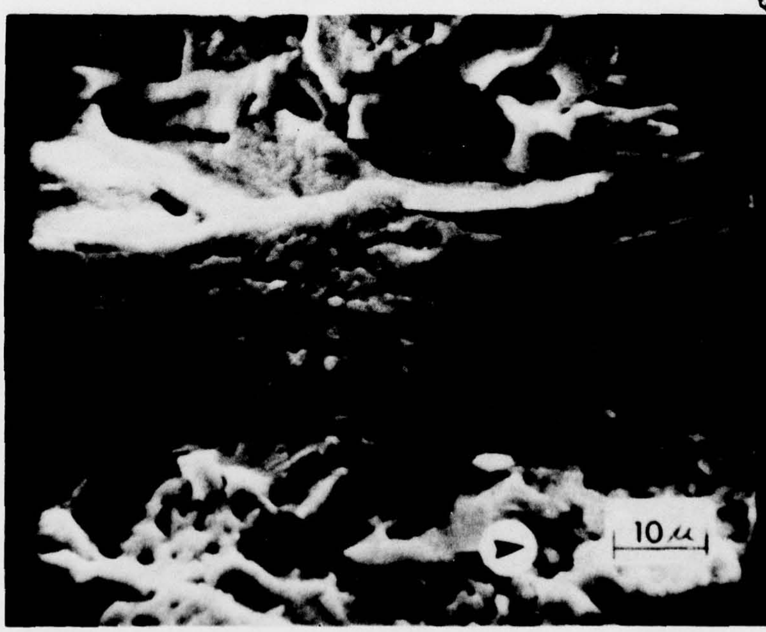

Figure 11. Micrograph of the same region as Figure 8, with fracture surface oriented at zero degree tilt with respect to the SEM collector. True striation spacing measurements indicate the spacing is twice as large under cathodic conditions (brittle) as under anodic conditions (ductile).

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