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A method of measuring the rate of crack propagation is described. The method, applicable to any specimen having a smooth surface across which brittle-type fracture will occur, is accomplished by means of a fine gridwork of conducting lines applied to the specimen by various vacuum deposition techniques. When a potential is applied to the grid, a direct reading of current (or distance) versus time is obtained with a strip chart recorder or equivalent device. The method has been applied to the study of brittle-type fracture associated with stress corrosion cracking observed in titanium alloys.

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INTRODUCTION

Stress corrosion cracking (SCC) is a complex phenomenon that requires a specific combination of stress, chemical environment, and metal composition and structure. SCC occurs at stresses considerably below the yield strength. It has been observed in specimens immersed in water and in a variety of organic liquids and is greatly accelerated by the presence of halides.

The rate at which the crack progresses is an important parameter, necessary in understanding the mechanism of SCC. The crack propagation rate is one measure of the susceptibility of a metal in a given environment. This has been demonstrated by T. R. Beck[1,2] and is illustrated in Fig. 1 for tests in which the environments were water-methanol solutions. All other factors were constant, including the stress applied to the specimens.

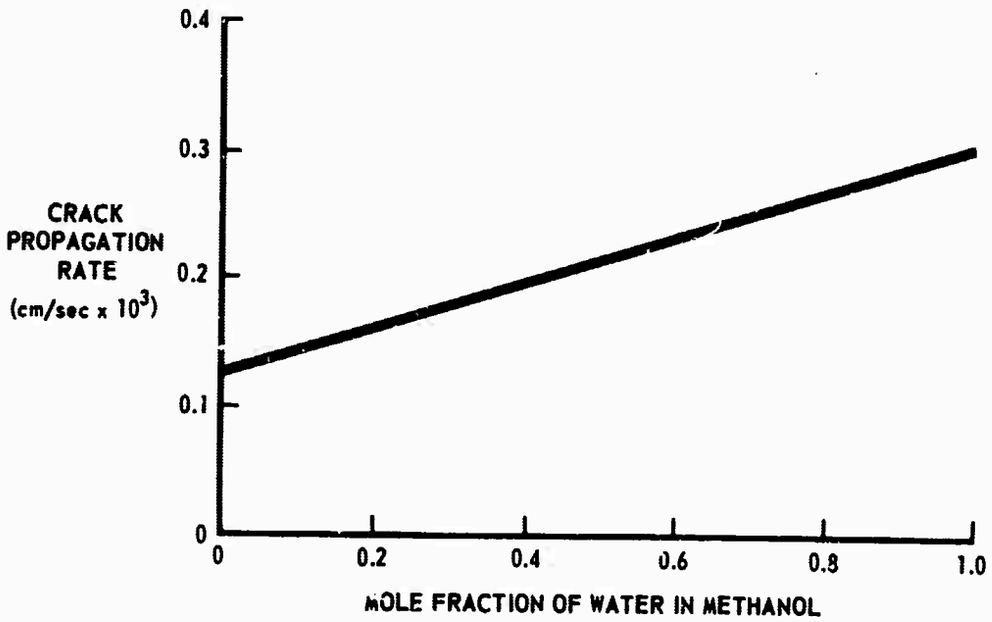


Fig. 1 Crack propagation rate as a measure of stress corrosion cracking susceptibility in a Ti-8Al-1Mo-1V alloy in water-methanol solutions. The specimens were notched and fatigue cracked, then placed in tension during the tests.

Previously in this laboratory, the crack propagation rate was measured by scribing pencil lines on the specimen and measuring the time required for the crack tip to pass between lines. This method is tedious and subject to errors of judgment by the observer.

To eliminate these errors and automate the rate measurement, a method of applying an accurate grid by vacuum deposition is being developed. Measuring the current-voltage relationship of the grid will give the rate of crack growth. Figure 2 shows a schematic of the grid system. When a constant potential is applied across the grid network, a current change will occur as each grid breaks. This current can be measured by a suitable strip chart recorder. A similar method was previously reported by Dulaney and Brace[3], who painted conducting lines on polymethyl methacrylate and used an oscilloscope to follow the crack propagation.

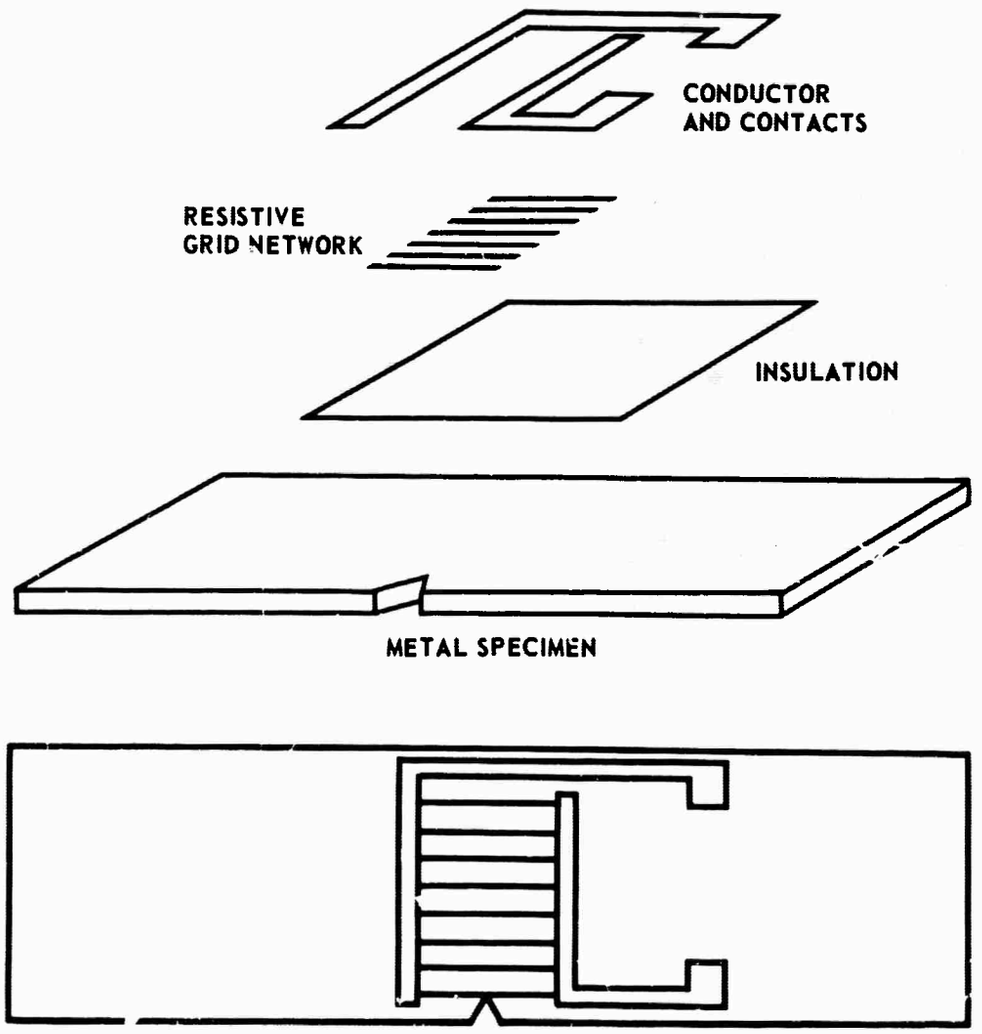


Fig. 2 Schematic of tension test specimen and grid network

DEPOSITION EQUIPMENT

The deposition apparatus used in preparing these specimens was designed at The Boeing Company for preparation of thin-film active devices. The equipment is capable of reaching 10^{-7} torr in 10 min. It holds sixteen 5- by 5-in. masks or substrates and up to 12 deposition materials. It is capable of reproducible mask registration to the substrate to within 0.5 mil. The vacuum chamber and mask indexing apparatus are shown in Fig. 3.

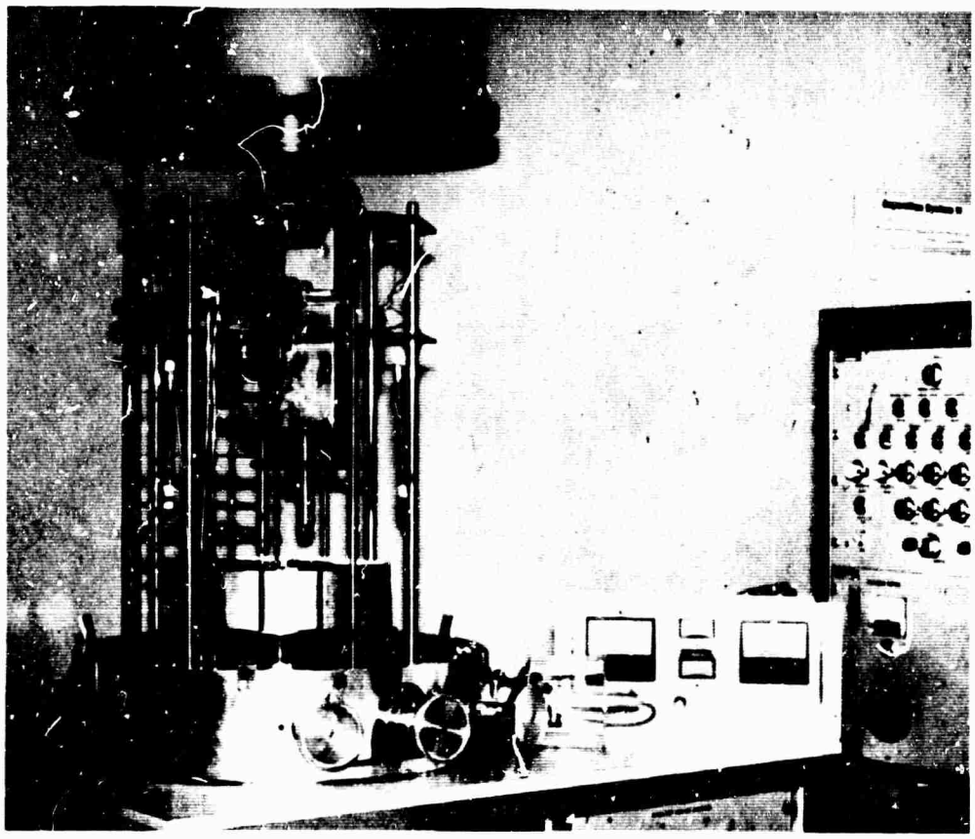


Fig. 3 Vacuum chamber and mask indexing apparatus.

GRID SYSTEM REQUIREMENTS

The large variety of environments used in the study of SCC places severe requirements on the materials used in the preparation of the grid system. The major requirements are that the grid be electrically isolated from the metal specimen and the solution and that the insulation material be insoluble in the test environment. Also, the grid system must adhere to the specimen and have similar physical properties

METHOD DETAILS

The usual material used for insulation in thin-film circuits, SiO, is not suitable here for several reasons. Its elongation and adhesion are low, causing the grid to break and separate from the specimen where metal yielding has taken place. Figure 4 is a photomicrograph of a polished metal specimen on which SiO was used as an insulator. SiO is also prone to pinhole formation, which may result in short circuits between the grid and metal or solution. Other ceramic materials have similar physical properties and therefore also would not be satisfactory insulators. Organic insulators capable of being vacuum deposited will be considered in the continuing program.

35X

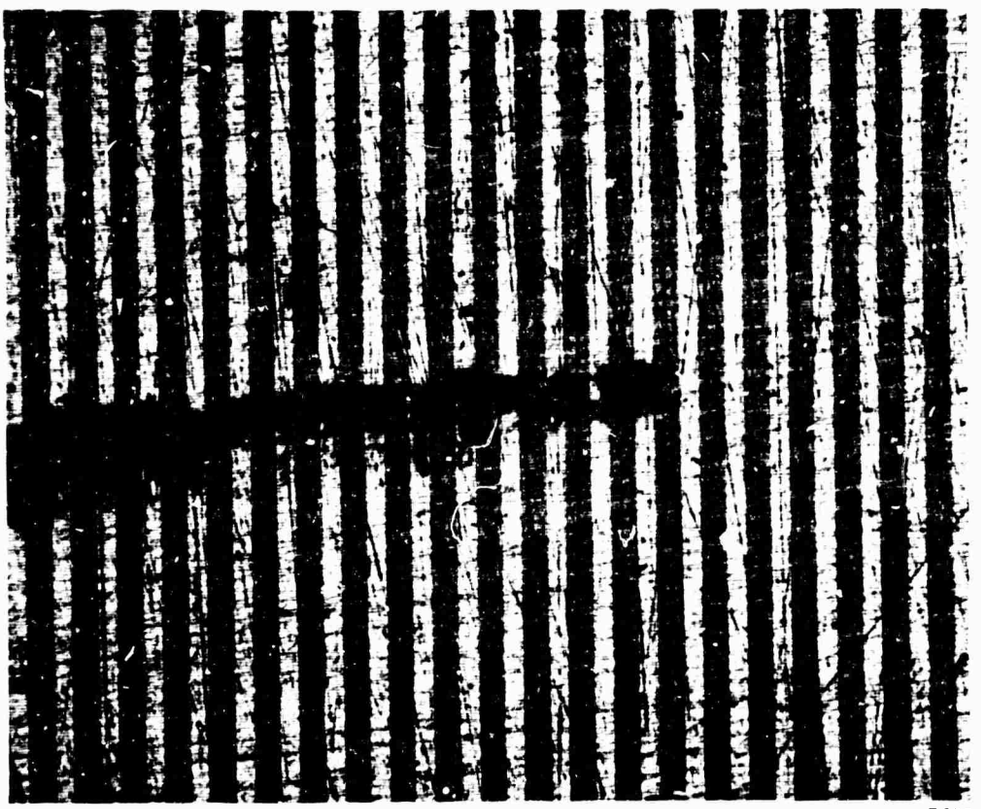
Fig. 4 Photomicrograph of area around the tip of the crack, showing poor SiO adhesion. The specimen was highly polished.

The metal grid may be isolated from the metal specimen by means of a thick anodic coating. The anodic coating may also improve grid adhesion. The specimens used in this study, however, were Ti alloy, were difficult to anodize uniformly, and thus would require additional insulation.

It is also possible to apply a nonconducting film by spraying or painting. Such films are acrylic spray, epoxy, "Teflon," etc. The chemical inertness and ease of application of epoxy dictated its selection as first candidate over several other possibilities.

Surface topography is also an important factor. As indicated before, polished surfaces may decrease adhesion, whereas rough surfaces can protrude through a thin insulator, causing short circuits. In the case of vacuum-deposited insulators, a rough surface may cause discontinuities in the resistors.

There are two methods of applying the grid conductors: masked evaporation and selective etching. Vacuum deposition through masks can provide closely spaced conductors as small as 5 mils wide. However, selective etching can produce lines as small as 0.5 mil wide.



50X

Fig. 5 Photomicrograph of a grid network prepared by selective etching, showing the area around the tip of the crack.

Selective etching is accomplished by first applying a blanket coating of the resistor material, applying a coating of photoresist (a commercially available compound such as Kodak KTFR), and then exposing the photoresist through a master with a carbon arc to define the grid. The unexposed photoresist is soluble in xylene and the unprotected conductor can be dissolved with a suitable acid. The remaining (exposed) photoresist can be removed with acetone and the specimen placed back in the vacuum chamber for the final deposition.

Figure 5 is a photomicrograph of a grid prepared by the selective etching technique, showing the fine resolution possible. Each conductor is 2 mils wide with a 2-mil separation. This specimen was Ti-8Al-1Mo-1V and was first anodized, then coated with SiO_2. The grid resistors were vacuum deposited Ti and were etched with a mixture of nitric and hydrofluoric acids.

Figure 6 is an example of the data taken directly from the recorder trace. The Ti-8Al-1Mo-1V specimen was stressed in a standard four-point loading test in 3.5 percent NaCl solution.

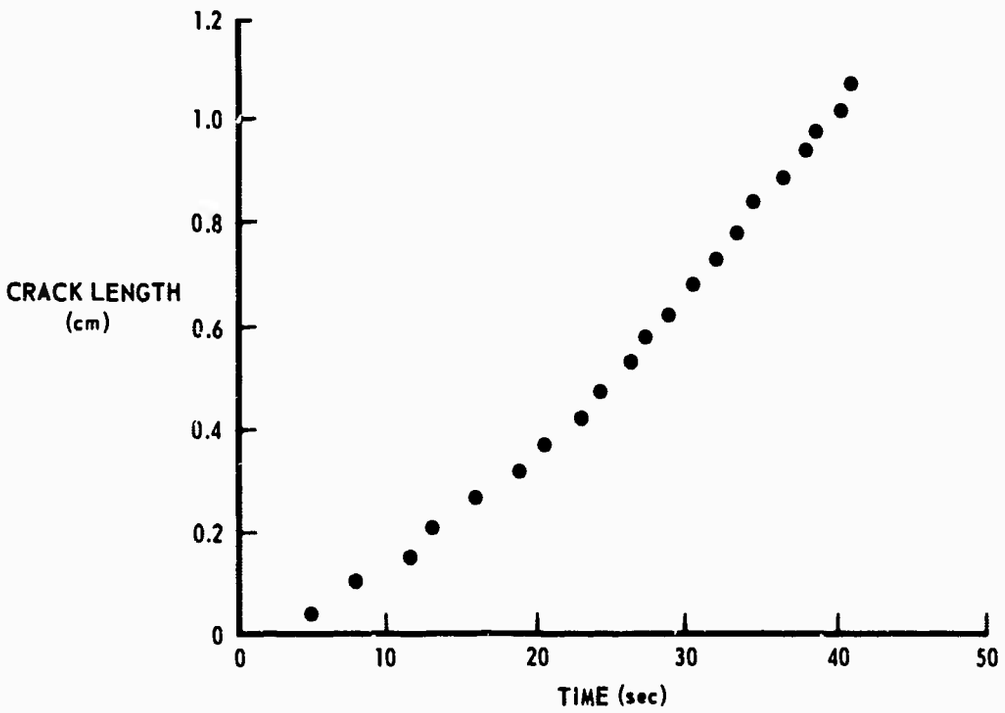


Fig. 6 Crack length versus time for 0.5-in.-thick Ti-8Al-1Mo-1V in 3.5 percent NaCl solution.

SUMMARY

It is believed that an accurate network of resistors can be prepared with a suitable combination of materials that will provide a reproducible method of measuring the propagation rate of brittle-type fractures in small specimens.

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3. E. N. Dulaney and W. F. Brace, Velocity Behavior of a Growing Crack, Journal of Applied Physics, 31, No. 12, 2233-2236 (1960) December.

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