temperature may help to clarify how stacking fault energy is affected by temperature.

This work received support from the National Council on Research (CNPq), and from the Ministério de Planejamento, BNDE and the Brazilian Army through IME-CPqM.

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Fracture Topography—Microstructure Correlations in the SEM

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The ability to observe simultaneously fracture features and the underlying microstructure is a tremendous asset in studies directed towards the definition of the relationship of fatigue and fracture properties to fracture topography and microstructure. The availability of the scanning electron microscope (SEM), which is capable of extending microstructural resolution far beyond that of an optical microscope and which has sufficient depth of focus to permit simultaneous viewing of fractures and underlying microstructures, has prompted development of techniques for preparing specimens suitable for such simultaneous viewing. A technique which has been used previously is one in which the fracture surface of a specimen is protected by a suitable material and the specimen subsequently sectioned, polished and etched on a plane perpendicular to the fracture path.[1] The technique suffers from two deficiencies, 1) edge rounding at the fracture-metallographic section edge may occur, obliterating some desirable features, and 2) removal of the mounting material requires considerable care in order not to damage the fracture face. Shechtman[2] has suggested a technique for preparing titanium alloy specimens which partially overcomes these drawbacks, but which relies heavily on differential polishing of the α and β phases. This communication describes a technique which is basically a refinement of Shechtman's technique and which permits considerable latitude for polishing and etching depending on the scale of the microstructure being examined and the degree of microstructural resolution desired. This technique, therefore, can be applied to literally any structural material which can be electropolished.

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Manuscript submitted July 19, 1976.

The fracture surface to be examined is cut into a convenient size for use in the SEM and the areas of the fracture surface which will be examined are protected by stop off lacquer. The exposed fracture surface is then electropolished using the method which is detailed later in the communication. One method for stopping off the fracture face is to use a circular drop of lacquer. This leads to a circular plateau of unaltered fracture surface which permits observation of the underlying microstructure in a plane parallel to, or perpendicular to, the macroscopic direction of crack propagation, or at any other location around the periphery of the plateau. A second effective method of stopping off the areas to be examined is to apply a strip of lacquer parallel to the macroscopic direction of crack propagation. This method permits examination of fatigue fractures over several orders of magnitude in growth rate. A schematic of both methods of stopping off the fracture surface is shown in Fig. 1(*a*), with details of a typical spot shown in Fig. 1(*b*).

Prior to application of the stop off lacquer (Stoner-Mudge Lacquer) the fracture surface is ultrasonically cleaned in acetone. We obtain the best lacquer adhesion if the electropolishing and etching is done within an hour following the lacquer application. To demonstrate the technique, both lacquered strips and small round patches are used. A convenient specimen size is one which has an exposed area for electropolishing of 1 cm^2 or less.

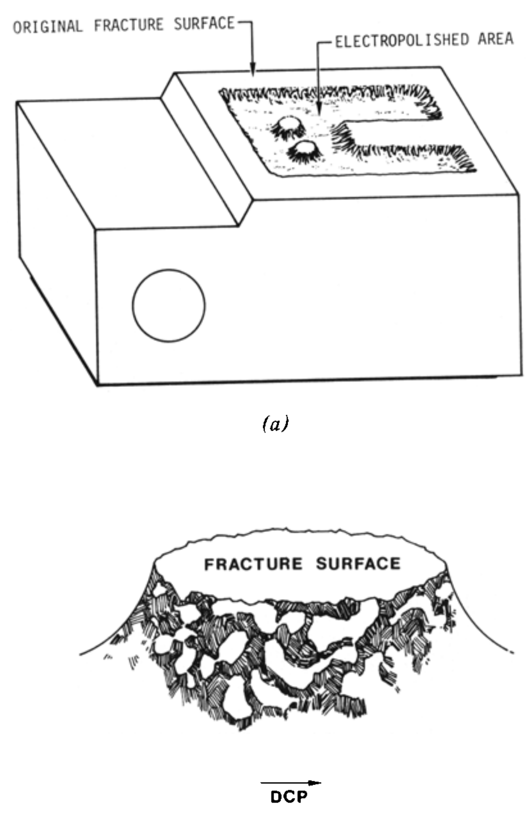

Fig. 1—Schematic of methods of stopping off areas to be examined: (*a*) overall specimen; (*b*) details of a typical spot.

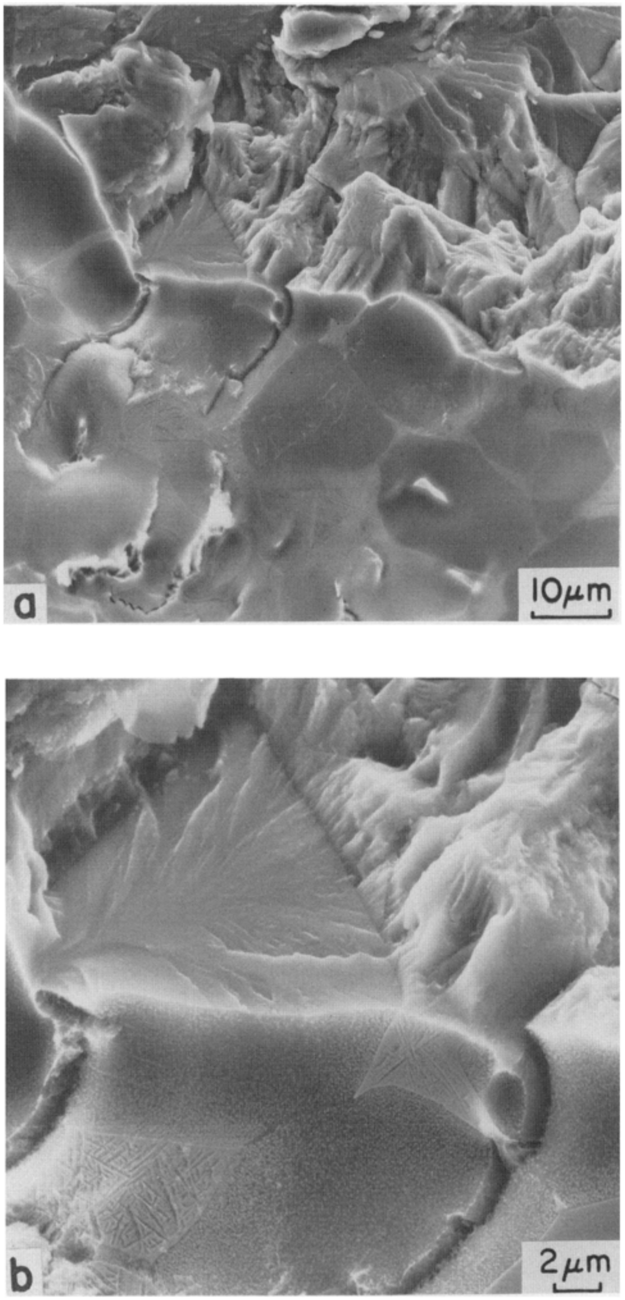

Fig. 2—Fatigue fracture of Ti-6Al-4V in a recrystallization annealed condition tested at R = 0.1 and 20 Hz in dry air, ΔK = 9.9 MN/m$^{3/2}$, da/dN = 2.5 × 10^{-6} mm/cycle.

Electropolishing of the exposed fractured surface is carried out using an electrolyte developed for Ti thin foil preparation.[3] This electrolyte consists of 5 pct H_2SO_4 by volume in methanol cooled to 238 K. The polishing operation is performed for 1 min at 14 volts DC using a Pt cathode in a slowly stirred solution. Immediately following electropolishing the specimen is washed in methanol and dried. The polished area is etched for 15 s, using equal parts of 1 pct HF and 10 pct oxalic acid solutions. The specimen is then washed with water and cleaned ultrasonically in acetone to remove the lacquer. A final rinse in fresh acetone is used to ensure a clean surface.

Examples of the technique as applied to Ti-6Al-4V (Ti-6-4) and Ti-6Al-2Sn-4Zr-6Mo (Ti-6-2-4-6) are

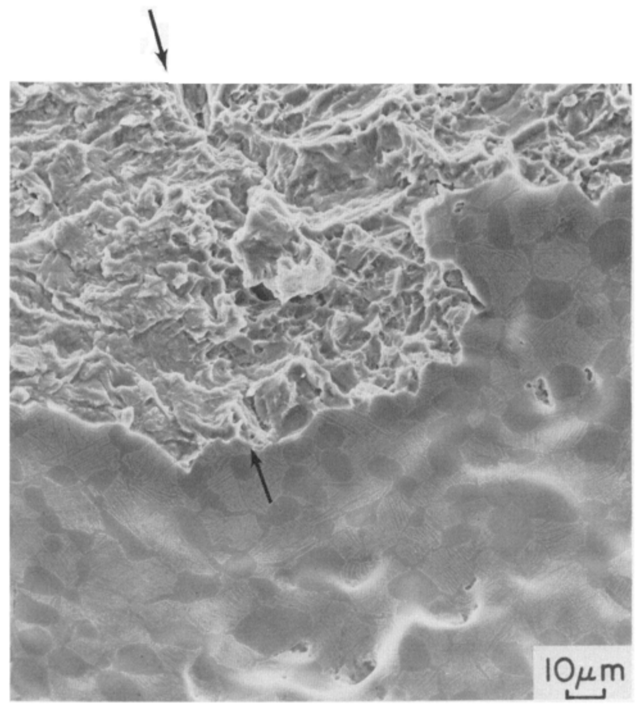

Fig. 3—Fracture surface of a fracture toughness specimen of Ti-6Al-4V in a STOA condition. Note the transition region (arrows) between the fatigue precrack and the tensile fast-fracture regions.

shown in the following figures. Fig. 2 shows a portion of a fatigue fracture from a Ti-6-4 specimen in a recrystallization anneal condition.* This figure shows

*Details of processing and resultant properties for all microstructures discussed are found in Ref. 4.

crack propagation by cyclic cleavage of primary α (darker grey equiaxed grains) and tearing of the transformed β. The transition from cyclic cleavage to tearing can clearly be seen in Fig. 2(b). Fig. 3 was obtained from a plane-strain fracture-toughness specimen (K_{Ic}) of Ti-6-4 in an STOA condition and shows the region (arrows) of the precrack to fast fracture transition; little change of surface topography is noted. Fig. 4 shows the difference in fracture topography in α/β *vs* β processed Ti-6-2-4-6. In the α/β processed material (Fig. 4(a)), the fatigue crack propagates primarily by tearing and appears to do so without much effect from the underlying microstructure. On the other hand, in the β processed material (Fig. 4(b)), the effect of microstructure can clearly be seen in the form of considerable secondary cracking along Widmanstätten α/β interfaces. In Fig. 5 the details of this secondary cracking can be seen, especially in the polished portion of the specimen shown in Fig. 5(b).

Two further comments seem appropriate. The use of nonlinear (gamma) signal amplification as suggested by Shechtman does indeed reduce the bright line at the edge joining the fracture face and the microstructural portion of the image, but it may also reduce significantly the resolution of lightly etched microstructures. For a majority of the micrographs in this paper, linear amplification was used. The second point is that with the SEM operating in the normal contrast mode, the contrast of the polished and etched micro-

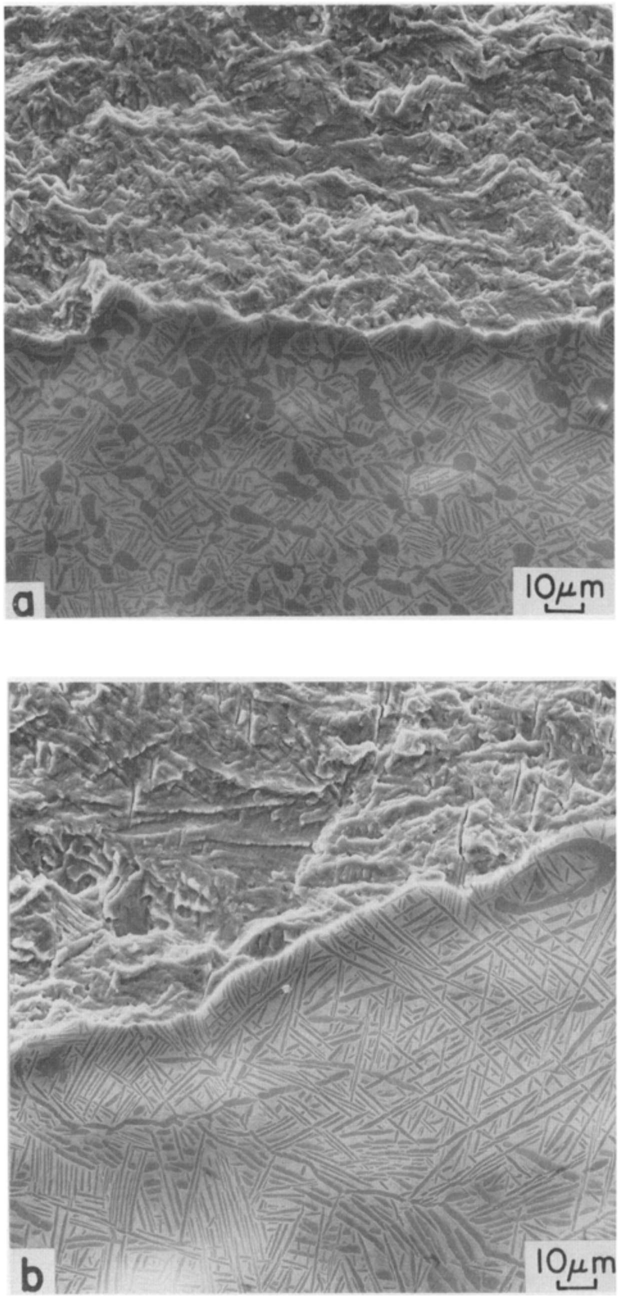

Fig. 4—Fatigue fracture of Ti-6Al-2Sn-4Zr-6Mo tested at R = 0.1 and 20 Hz in dry air: (*a*) α/β processed material, ΔK = 4.5 MN/m$^{3/2}$, da/dN = 2.5 × 10^{-6} mm/cycle; (*b*) β processed material, ΔK = 5.0 MN/m$^{3/2}$, da/dN = 2.5 × 10^{-4} mm/cycle.

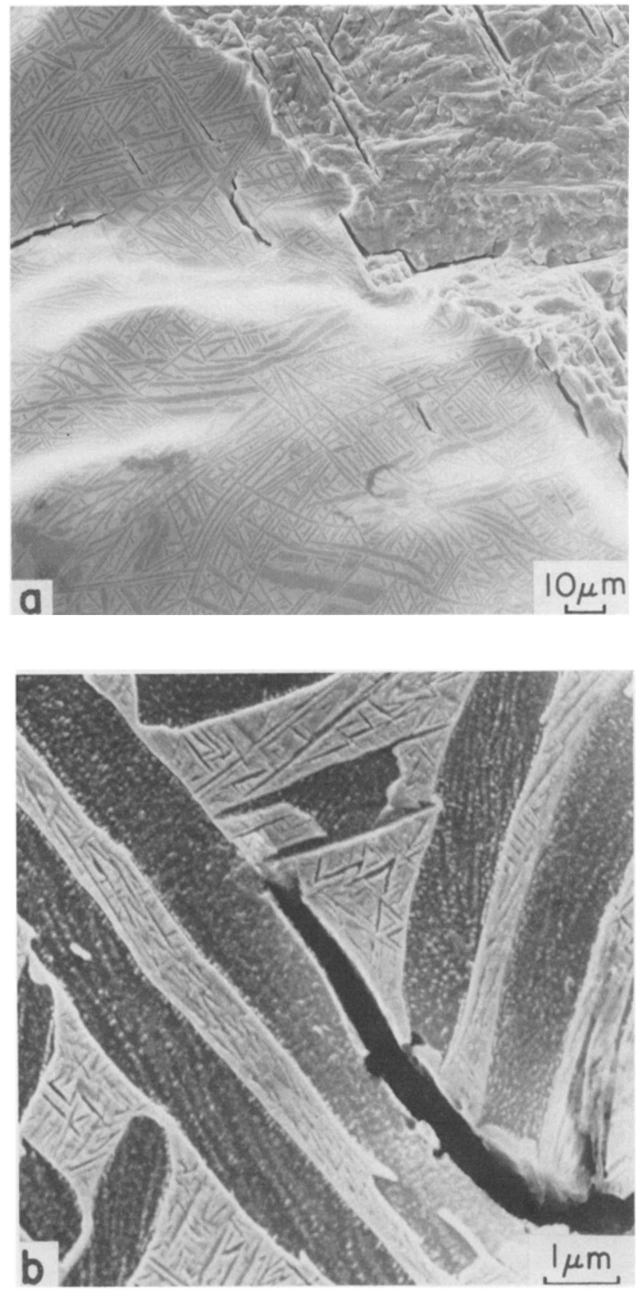

Fig. 5—Fatigue fracture of Ti-6Al-2Sn-4Zr-6Mo tested at R = 0.3 and 20 Hz in dry air, ΔK = 3.5 MN/m$^{3/2}$, da/dN = 2.5 × 10^{-4} mm/cycle.

structure is reversed from that of incident light micrographs in that α phase appears a darker grey than β phase.

In conclusion, we have shown by example the powerful nature of this technique for two $\alpha + \beta$ titanium alloys. These examples comprise only a small fraction of the titanium alloys we have examined using this technique. The technique has proven to be generally applicable to any material which can be electropolished and promises to be very helpful in elucidating the relationship between fracture topography and microstructure.

This technique was developed under Air Force Contract No. F33615-74-C-5067.

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