A technique for selected-area observations in silicon wafers by transmission electron microscopy

During the course of an investigation to study laser-induced fracture and flaw-fracture interactions in silicon wafers and substrates, it became necessary to develop a technique for systematically isolating various and specific sections from these specimens for direct observation of crystal defects in the transmission electron microscope. While there have been a number of electropolishing techniques developed for silicon [1-4], only that developed by Lawrence and Koehler [4] was at all adaptable to this particular task. The new method is simpler than that of Lawrence and Loehler [4] and considerably more versatile.

In the present technique, selected areas on

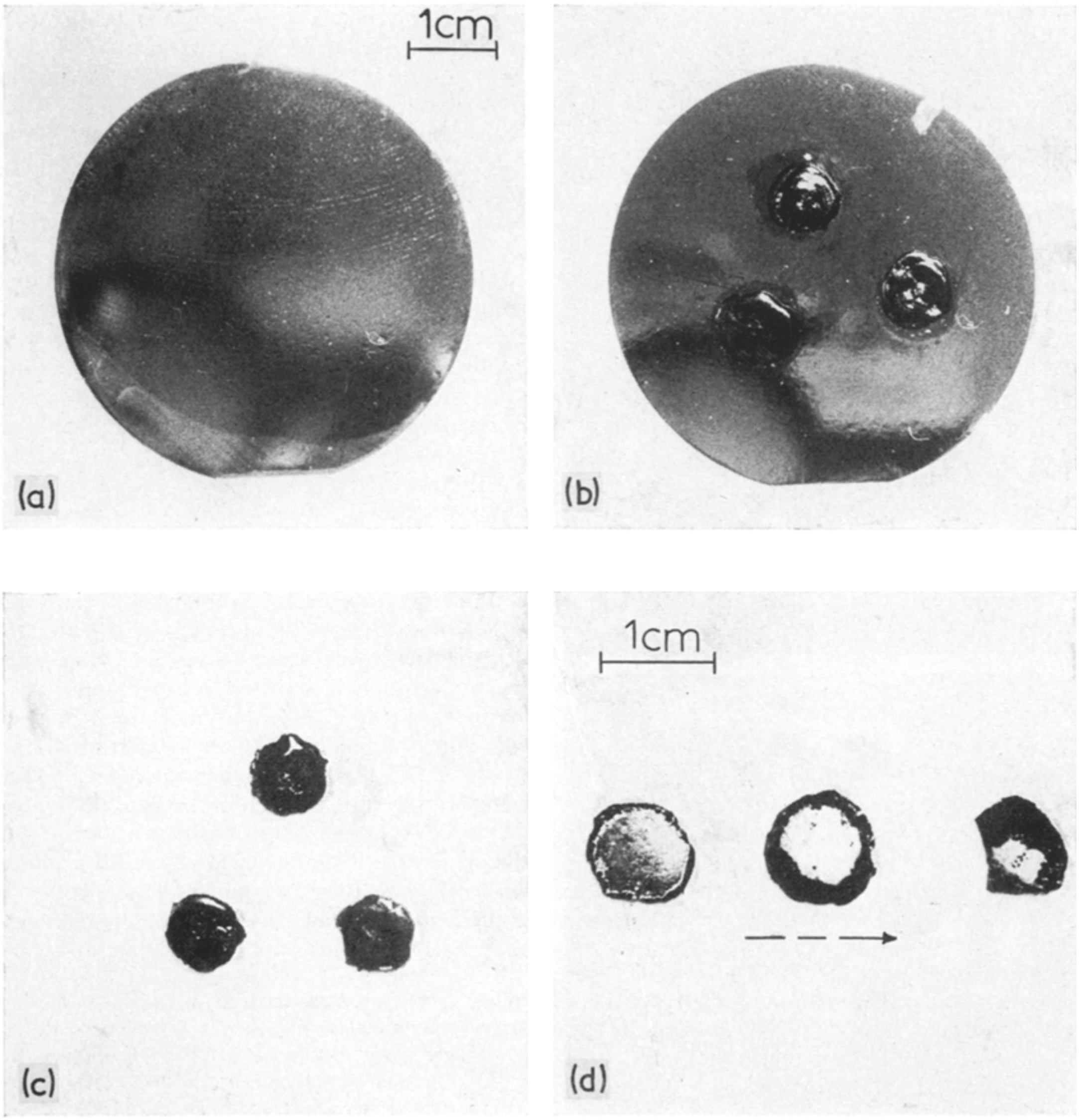

Figure 1 Preparation and isolation of selected areas on silicon wafers for transmission electron microscopy. (a) Mechanically polished Si wafer (0.040 mm thick). (b) Selected areas on surface or bulk of Si wafer to be isolated using black-wax buttons over the areas coincidentally masking both surfaces. (c) Wax buttons isolated by dissolution of Si wafer matrix. The desired area or volume is sandwiched between the buttons. (d) Sequence showing the thinning stages of isolated Si section (isolate) for electron microscopy. The wax serves as a surface preservative where desired, and a support.

silicon wafers (pure or doped) ranging in size from 2 to 5 cm in diameter and 0.025 to 0.050 mm thick and either mechanically or chemically polished, are placed in the centre of a polyethylene mask having a 1 cm opening coincident over both surfaces. A low-melting black wax is applied to the masked areas forming 1 cm buttons centred over the section (area) of interest on both sides of the wafer surface. Depending upon the size of the wafer, any number of facing pairs of wax buttons can be placed on selected areas. The polyethylene mask is then released, leaving the hardened wax buttons. A location number is then impressed into each button and the area identified. Buttons can also be applied by tracing a 1 cm circle on opposite sides of a desired area, and with an initial wax drop in the centre, the wax is spread using a hot metal rod to allow the wax drop to melt and flow to the circle tracing. The entire wafer and located buttons are then placed in a polyethylene beaker containing a solution of 95% HNO_3 and 5% HF. A spontaneous and vigorous reaction occurs and the unmasked silicon matrix is dissolved. Normally, the silicon wafer will float on the solution surface and the reaction, being slightly enhanced at the edges of the buttons, causes the buttons to fall out of the matrix. These buttons are recovered with a polyethylene tweezer.

The wax can be removed from the isolated sections by immersion in trichloroethylene and successive rinsing in acetone, distilled water and finally in ethyl alcohol. The surface to be preserved on any of the isolated sections is then recoated with wax, and floated in a beaker of nitric and hydrofluoric acid in the proportion 3:1 respectively.

The dissolution of the isolated section can then be observed against the black wax background, and as its diameter (size) decreases, a corresponding decrease occurs in thickness. The stages in dissolution may be checked by placing the acid beaker in ice water to reduce or stop the reaction, which is sensitively exothermal. At a diameter of from 1 to 3 mm, the sections on black wax supports are usually electron transparent. At this point, the samples are rinsed in distilled water and placed in trichlorethylene to remove the wax support. The electron transparent silicon sample is then transferred to an acetone-water-alcohol rinse sequence. The alcohol is poured off in the last rinse, and the dried sample

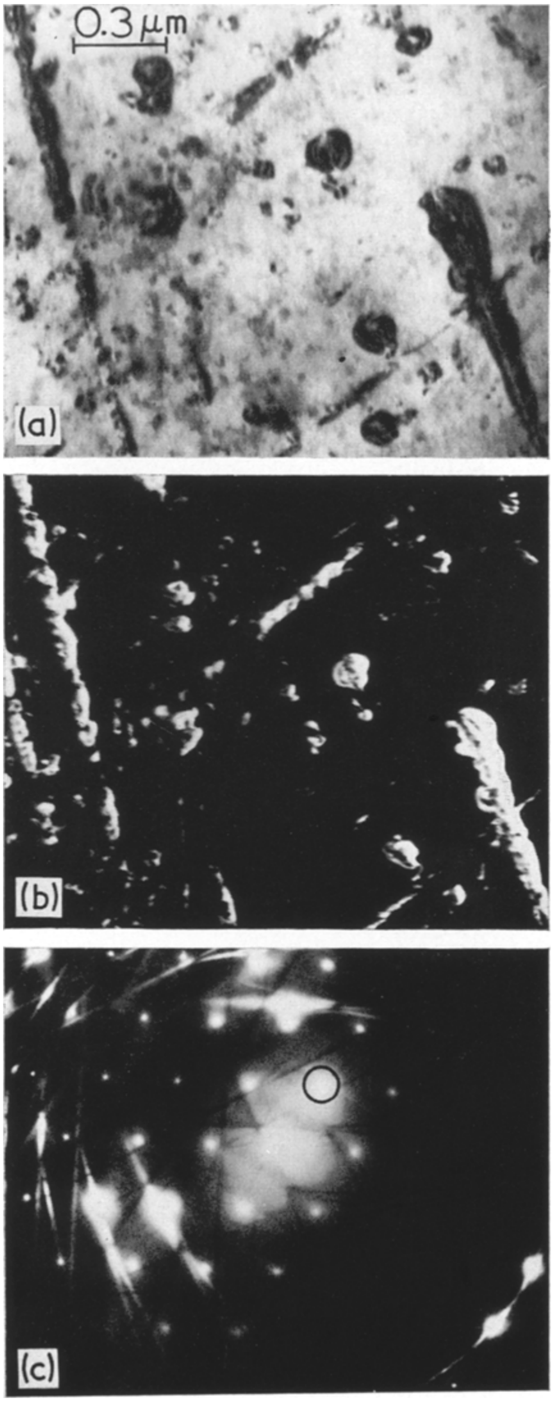

Figure 2 Transmission electron micrographs showing surface-related defects in a P-type Si wafer isolate. (a) Bright-field image, (b) dark-field image of (a) using the [02$\bar{2}$] reflection shown circled in the corresponding selected-area electron diffraction pattern of (c).

transferred to a screen grid for electron microscopy utilizing a vacuum tweezer.

Fig. 1 illustrates the stages in preparing isolated sections from a boron-doped (P-type) silicon wafer described above. The preserved surface of one of the isolates is recognized as the surface next to the wax support in the chemical thinning sequence shown in Fig. 1d. In cases where it is desired to examine a section within the wafer bulk away from both surfaces, the procedure involves thinning the isolate from one side for a short period of time, dissolving the wax from the preserved side, and reapplying wax to the polished side. The final polishing is then performed from the preserved surface side. By manipulating the rate of dissolution of the isolate, it is possible to look at the microstructure associated with either surface, or at some estimated depth beneath a surface or between the surfaces. Fig. 2 illustrates somewhat typically the observation of an isolate section in the transmission electron microscope taken from a mechanically polished boron-doped (P-type) wafer. Surface-related defects resulting from precipitation and induced stresses are shown in bright and dark-field electron transmission micrographs.

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References

1. R. P. RIESZ and C. G. BJORLING, *Rev. Sci. Instrum.* **32** (1961) 889.
2. G. R. BOOKER and R. STICKLER, *Brit. J. Appl. Phys.* **13** (1962) 446.
3. H. J. QUEISSER, A. H. FINCH and J. WASHBURN, *J. Appl. Phys.* **33** (1962) 1536.
4. J. E. LAWRENCE and H. KOEHLER, *J. Sci. Instrum.* **42** (1965) 270.

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Fractography of a styrene-acrylonitrile co-polymer—microcracks and band structure

Attention has been given since 1950 to periodic bands which may be observed on the fracture surfaces of glassy polymers [1]. Recently Hull suggested a detailed explanation of their formation in polystyrene. The central idea is that bands are formed by the crack tip jumping into crazes which are generated by its own stress field [2]. Doyle *et al* rejected this idea on the grounds that the bands are formed under conditions where the propagation of the crack tip is too rapid to allow time for extensive craze formation by the mechanism invoked. They offered another explanation which involves periodic detachment of the craze wedge generated ahead of the crack tip [3].

The purpose of the present note is to demonstrate that a banded structure observed on the fracture surface of a styrene-acrylonitrile co-polymer is due to the crack tip jumping into microcracks which are generated in crazes.

The co-polymer was reported by the manufacturer to include 30%, by weight, of acrylonitrile and to have a number average molecular weight of 6×10^4 (Tyril 867; Dow Chemical Company). A powder sample was heated to 180°C and pressed at 84 kg cm^{-2} for 10 min to provide a transparent disc (diameter, 2.7 cm; thickness 0.3 cm). A razor blade was wedged into a saw cut. This resulted in formation of a craze (Fig. 1) which was succeeded by a crack which eventually resulted in fracture of the disc. The fracture surface of the disc included bands irregularly spaced at distances in the range 200 to 800 μm (Fig. 2a). When viewed from the side (along the z-axis in Fig. 1) by reflected light it could be seen that these band spacings correspond to steps on the surface. The detail displayed at higher magnification in Fig. 2b, and designated at its extremes by marks, measures 220 μm.

The progression of the crack tip through the sample could be followed under the microscope as a series of pulses and seemed to fit the "slip-stick" description often used in accounts of the fracture of polymers [4]. At high magnification and low crack speed, of the order 10^{-2} cm sec^{-1}, this process could be followed in detail at the microstructural level. For example Fig. 3a shows the tip of the crack (A), which generated the fracture surface up to this point,