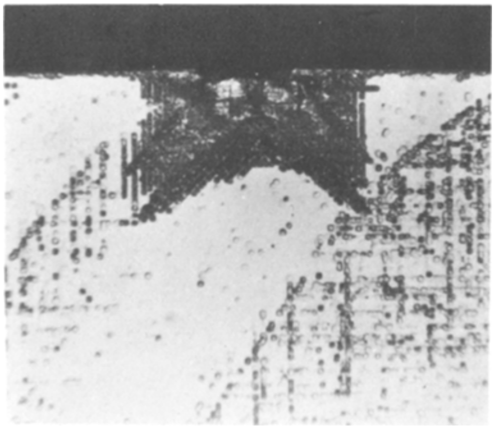

Figure 2 Section through a 60° cone indentation showing the dislocation pattern produced beneath the indentation.

The polishing and etching procedure was repeated in order to reveal the dislocation patterns produced at depths of approximately 40 μm (Fig. 1b) 80 μm (Fig. 1c) and 120 μm (Fig. 1d) below the original indented surface. Note that on the indented surface, a typical dislocation rosette is produced around the indentations due to the movement of dislocations on all of the six operative {1 1 0} slip planes. Also, it can be seen that at a depth of approximately 40 μm (Fig. 1b), the radial cracks on the {1 1 0} planes normal to the indented (0 0 1) surface, and presumably due to the Keh mechanism [2], have disappeared. It should be mentioned that the chemical polishing sequence was deliberately curtailed for this micrograph in order to demonstrate the nature of the pits remaining after the hydrochloric acid agitation treatment. At a depth of 80 μm (Fig. 1c), the dislocations are still present on all six slip planes. However, at this depth a dislocation-free region is developed at the centre of the dislocation pattern. Towards the bottom of the dislocated zone, at approximately 120 μm depth (Fig. 1d), dislocations are limited to the four {1 1 0} slip planes which are inclined at 45° to the indented (0 0 1) surface.

For comparison purposes, the dislocation pattern produced on a cross-sectional plane through a 60° cone indentation, and obtained by cleaving the crystal on a (1 0 0) plane normal to the indented (0 0 1) surface, is shown in Fig. 2.

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The decoration of surface flaws in glass

The uncertainty which still surrounds the nature and distribution of Griffith flaws in glass is a central problem in the characterization of glass surfaces; this is of technological importance in relation to the failure of optical components under arduous service conditions.

Attempts to observe Griffith flaws directly have been unsuccessful; this supports the view that if flaws exist in pristine glass surfaces they can only be described in terms of localized, atomic scale disruptions in surface structure. The exact nature of these perturbations in structure is yet unknown although it has been suggested that they may be identified with regions of weak bonding arising from an uneven temperature distribution during cooling. Alternatively they may result from local stresses associated with the presence of microstructures. Any such approach, however, must allow sufficient stress concentration and subsequent flaw growth to account for the rapid strength degradation which is observed when a pristine surface is subject to normal handling.

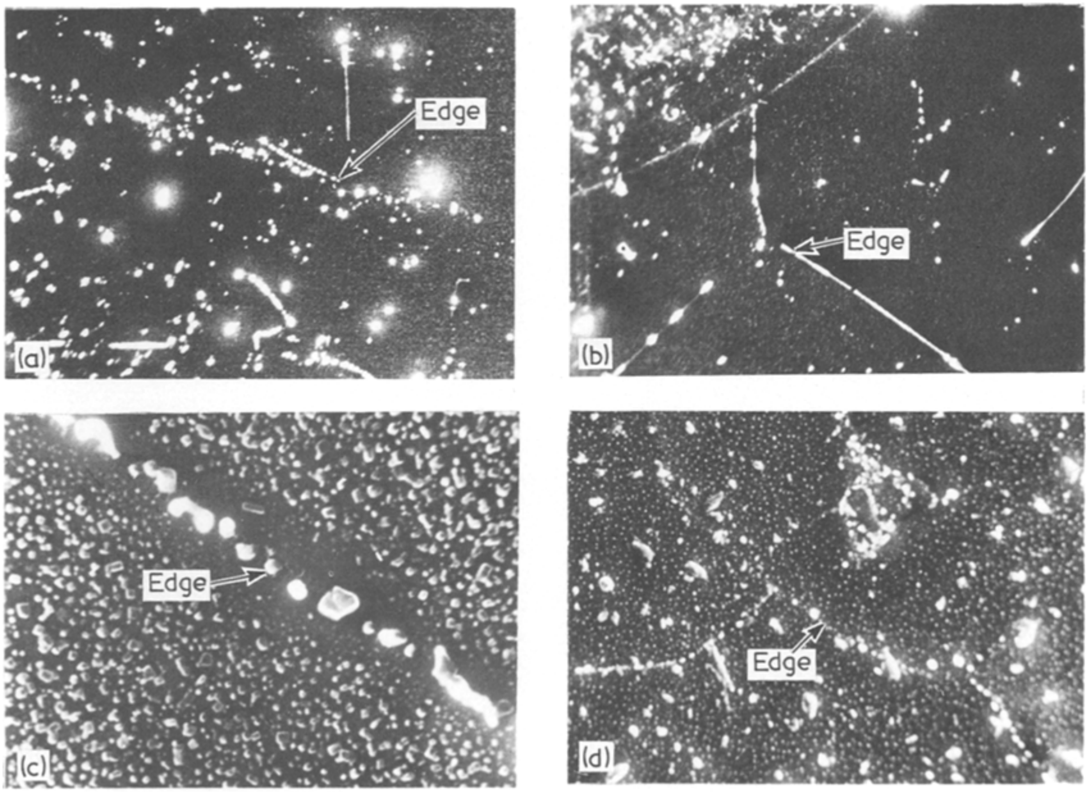

Figure 1 Decoration of float glass specimens by gold deposition and vacuum heat-treatment. Gold sputtered by argon beam bombardment at 6 kV accelerating potential for 5 min (ion beam current density, $3.0 \times 10^{-3}\,\mu A\,m^{-2}$) (a) "As received" float surface. Substrate temperature, $500 \pm 10^\circ$ C ($\times$ 130). (b) "As received" float surface showing fracture edge. Substrate temperature $500 \pm 10^\circ$ C ($\times$ 280). (c) Polished surface (6 μm diamond lapping following initial grinding using 600 grit silicon carbide). Substrate temperature $500 \pm 10^\circ$ C ($\times$ 5040). (d) "As received" float with scratch mark. Substrate temperature $400 \pm 10^\circ$ C ($\times$ 2310).

We report here the development of a technique for the "decoration" of surface flaws not normally visible using an electron microscope. The method was conceived on the basis of the early observations of Andrade and Martindale [1] who showed that thin lines appeared on glass surfaces after vacuum deposition of gold and subsequent heating. The form of these features was invariant to cleaning procedure and only after repolishing did the original lines disappear, others appearing in different positions. There is, perhaps, a relationship between these observations and the epitaxial growth of metal films on crystalline substrates [2]. This process is dependent upon the diffusion of deposited adatoms which, after reaching thermal equilibrium with the substrate surface, execute a random walk until they desorb, are trapped by preferential nucleation sites or form polyatomic aggregations by interactions with other adatoms. The topography of the resultant deposition is found to be strongly dependent upon substrate structure and particularly upon the presence of point defects, dislocations and cleavage steps [2]. The temperature at which surface mobility is initiated has been shown to be in the range, 200 to 400° C for gold and silver deposits [4].

It is not unreasonable, therefore, that glass surfaces which cannot contain structural defects in the crystalline sense, might contain preferential nucleation sites associated with the presence of surface flaws, resulting in localised film growth. This necessarily requires a substrate temperature which will provide mobile adatoms with a sufficiently long path length to seek out such nucleation sites. Further, it is assumed that at some stage in the process the mutual attraction of adatoms leading to anisotropic film growth takes precedence over nucleation at less preferential sites; this is consistent with the concept of a saturation density of nuclei. Furthermore, the use of a range of substrate temperatures should allow flaw populations associated with different variations in surface energy to be sampled.

Gold was deposited on float glass specimens using ion beam sputtering at 6 kV under vacuum conditions of approximately 10^{-6} mm Hg. Subsequent heat-treatment also under vacuum resulted in thin film segregation; specimen surfaces were then examined by scanning electron microscopy. A decorated float glass specimen is shown in Fig. 1a. This shows the presence of several lines and aggregation effects, higher magnification micrographs suggesting a general background of similar particles; amongst these there is also an indication of alignment. Fig. 1b shows a region of decorated float glass close to a fracture surface which originated from specimen cutting. In this case the marked departures from uniformity suggest the presence of more extensive flaws or faults attributable to localized stresses. In all cases high magnification examination prior to decoration showed no visible cracks.

If decoration effects are due to the presence of submicroscopic flaws, then it is reasonable to expect that macroscopic features should exhibit a similar attraction to mobile gold adatoms. A decorated float glass specimen which contained a scratch mark is shown in Fig. 1c; the substrate temperature was reduced to 400° C in order to decrease the mean path length of mobile adatoms.

Float glass specimens which had been ground and repolished were also decorated (Fig. 1d). There appears to be a considerable accumulation of gold in the vicinity of polishing pits and a general alignment throughout the field of view; the latter features do not necessarily coincide with visible cracks or rifts. In preliminary work, a marked sensitivity to substrate temperature and the amount of gold deposited was observed; calculations based on these observations suggest that mean diffusion lengths in the range, 1 to 70 μm, may be associated with substrate temperatures of between 400 to 500° C.

The development of these techniques has allowed surface features at the submicroscopic level of structure to be indirectly observed by allowing the growth of gold aggregations to dimensions compatible with SEM. It appears from our observations that decorated features in as-received float surfaces reflect the presence of developed Griffith flaws, accepting that inherent flaws in an undeveloped state are only present in pristine surfaces. In the case of repolished specimens, flaws resulting from surface treatment and not normally sensitive to conventional methods are rendered visible. Used in combination with destructive techniques such as ion-exchange [5] and indentation fracture [6], gold decoration should result in a more complete physical characterization of glass surfaces.

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