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Laser Reflectogram Method for the Study of Crystal Surfaces and Epitaxial Deposits

F. H. COCKS, B. N. DAS, G. A. WOLFF
Tyco Laboratories Inc, Bear Hill, Waltham, Mass, USA

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When a collimated beam of light is reflected from an etched crystal surface or an epitaxial deposit, the pattern formed by this reflection provides information on the microscopic morphology of the reflecting surface. Using a He/Ne laser as a source of high-intensity collimated light, both SiC etched in ClF_3 and cubic CdS grown epitaxially on GaP have been examined. Certain regions of the SiC were found to give rise to diffraction effects in the resulting reflectrogram. The epitaxial layer of CdS was found to show a threefold symmetry indicative of a {111} deposit and a negligible diffraction effect.

1. Introduction

Crystallographic pits are often formed when crystal surfaces are etched or cleaved. In crystal growth, similar geometric surface features (hillocks) can also appear when one type of crystal is formed epitaxially upon another. Reflectograms or light-figures are produced when a surface exhibiting crystallographic pits or hillocks is exposed to a narrow beam of collimated light and the reflected pattern is projected on a screen placed between the crystal and the light-source (fig. 1a).

If all pits or other structural features on the surface were composed, on a macroscopic scale, of the same sets of low-angle planes, then only a series of spots, whose maximum size approximately equalled the beam diameter, would be expected. It is usually the case, however, that, instead of singularly inclined planes, entire zones of planes are exposed and, in this case, lines rather than the spots shown in fig. 1a are observed.

The light-figure technique has proved itself valuable in the investigation of a variety of crystal surfaces, including: metals [1, 2]; minerals [3]; silicon and germanium [4]; II-VI and III-V compounds [5], including GaAs [6]; and epitaxially grown layers of wurtzite-type CdTe, sphalerite-type CdS [7], and also epitaxially grown germanium [8].

All of these investigations have utilised collimated white light from an incandescent

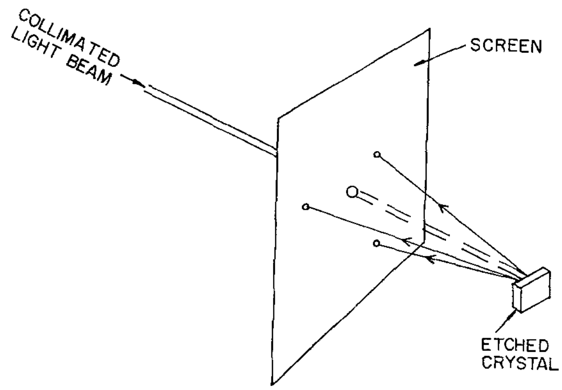


Figure 1a A schematic illustration of the light-figure back-reflection method.

source. However, with the current availability of lasers which are almost ideal sources of collimated monochromatic light, it is possible to obtain significantly greater resolution than that which can be achieved using a standard light-source and an optical collimation system on an optical bench. In addition, the much higher intensity available from a laser, as opposed to even an arc-light source, allows a smaller pencil beam to be used while retaining sufficient intensity to allow visual examination to be made. Thus, much smaller regions can be examined. Furthermore, the problem of vibration-distortion is minimised because of the shorter exposure times that can be used.

2. Apparatus

A photograph of the actual reflectogram apparatus in operation is shown in fig. 1b. (The light beam was made visible by the presence of smoke.) It consists of a He/Ne laser operating at 6328 Å, together with a shutter, Polaroid film holder, and crystal goniometer. In operation, the crystal is rotated by means of the goniometer until the principal reflection is in reverse direction parallel to the incident beam. If the screen-to-specimen distance is known, the angles for all other reflections can be calculated. By making this distance 3 or 5 cm, standard Greninger charts can be used to produce stereographic projections of all reflecting planes.

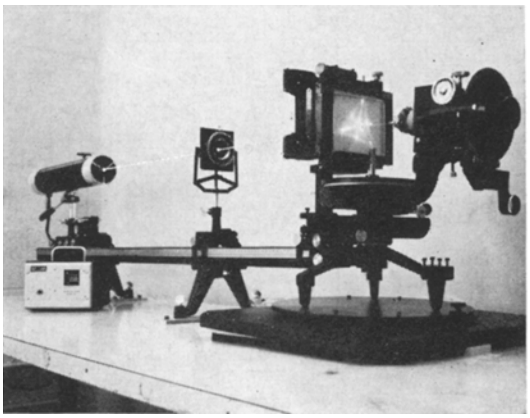
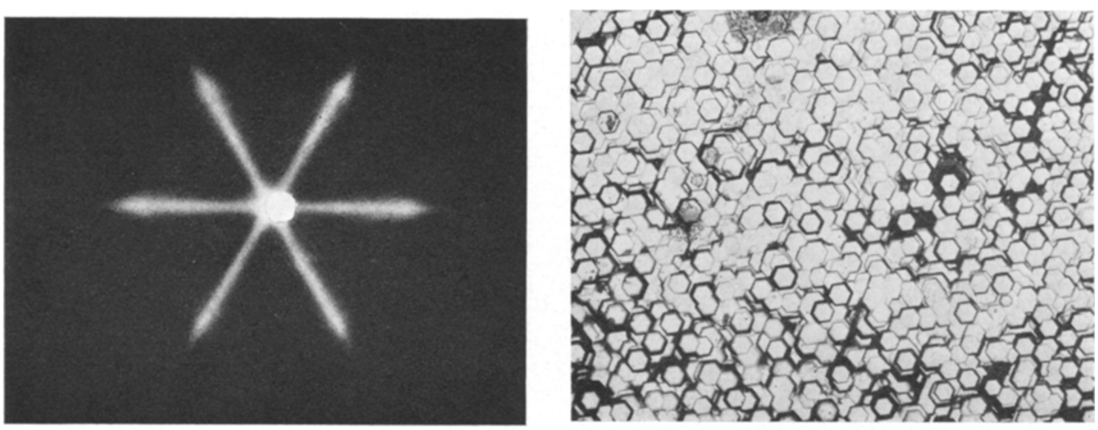


Figure 1b A photograph of the laser reflectogram apparatus in operation.

3. Results and Discussion

This system has been used to examine the surfaces of various crystals, including both etched SiC and epitaxial layers of CdS on GaP. These investigations have demonstrated the correlations that may be obtained between optical microscopy and light-figure examination. Examination of a {00.1} plane of a single crystal of SiC, which showed a marked local variation of surface structure after etching in ClF_3* at 400° C, clearly revealed an equivalent variation in its light-figure patterns. Figs. 2a and 3a show the light-figure patterns obtained from the structures shown in figs. 2b and 3b respectively. From these figures, it is seen that the microsteps of S-planes (stepped planes), which compose the pit walls as shown in fig. 3b, give rise to a diffraction grating effect along the arms of a sixfold light-figure pattern. Furthermore, the rounding off of the edges of the pits gives rise to the appearance of circular light-figure lines corresponding to an increase in polar distance (i.e. tilt with respect to the basal plane) of those planes whose vertices compose the (rounded) pit edges.

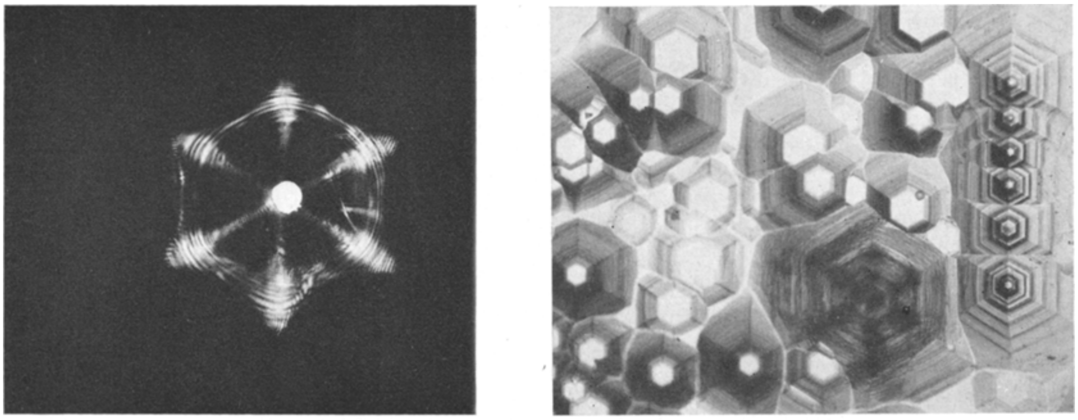
The decrease in central distance of the ends of the radial light-figure arms in going from fig. 2a to fig. 3a is a necessary concomitant of this broadening. This rounding off of the pit edges is most clearly seen for the case of the largest pit shown in fig. 3b. It is probable that, since commercial SiC was used, this variation in structure arises from impurity variations in the



(a) (b)

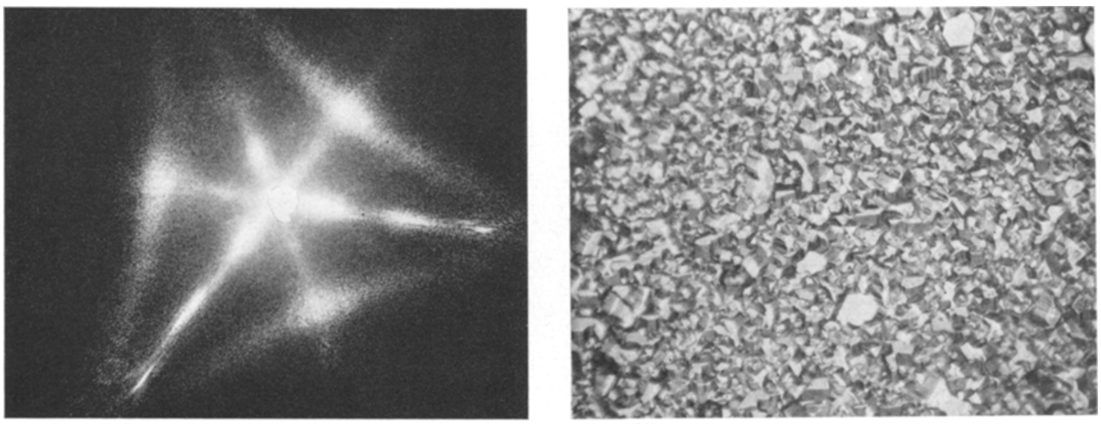
Figure 2 (a) Reflectogram obtained from the {00.1} plane of SiC etched in ClF_3 gas (film-to-specimen distance 3 cm). (b) A photomicrograph of the surface structure which gave rise to the reflectogram shown in (a) (×60).

*Work on the etching behaviour of SiC crystals in gaseous ClF_3 was performed under contract AF 19 (628)-4384.



(a) (b)

Figure 3 (a) Reflectogram obtained from a different region of the same SiC crystal used to obtain fig. 2 (film-to-specimen distance 5 cm). (b) A photomicrograph of the surface structure which gave rise to the reflectogram in (a) ($\times$60).



(a) (b)

Figure 4 (a) Reflectogram obtained from an epitaxial deposit of CdS on GaP (film-to-specimen distance 3 cm). (b) A photomicrograph of the surface structure which gave rise to the reflectogram shown in (a) ($\times$185).

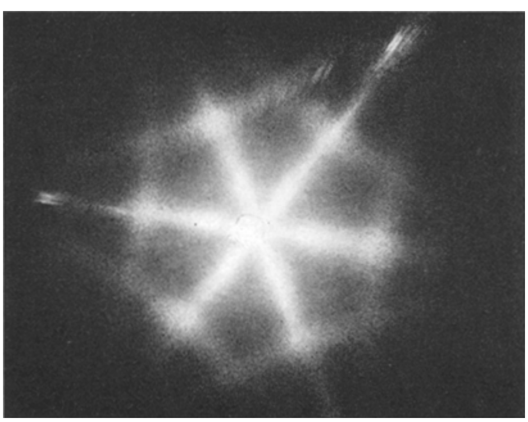


Figure 5 Reflectogram obtained from a twinned epitaxial deposit of CdS that nucleated on a twinned GaP substrate (film-to-specimen distance 3 cm).

crystals rather than from polytypism, because both structures show sixfold symmetry (although more than one polytype showing sixfold symmetry is strictly possible). Resolution of such fine effects by the light-figure technique has not been reported previously.

In the case cited above (that of etched SiC), the light-figure examination did not reveal any features which could not also be seen by optical examination. In the case of a deposit of cubic CdS on GaP [7], however, light-figure examination (fig. 4a) clearly demonstrates the threefold symmetry characteristics of a (111)-type deposit, while optical examination shows no obvious symmetry (fig. 4b). The light-figure technique also clearly shows the presence on the same crystal of two different (111)-type layers rotated by 180° (fig. 5) because of a twin boundary on the GaP substrate.

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