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Light Figures and Surface Structures of Germanium Crystal. I

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INTRODUCTION

On determination of crystal orientation by light figure method, many reports have been made by several investigators.[1)~4)]

This method is a convenient and rapid procedure, a technique so suitable to a practical orientation determination of crystals.

It is known that the symmetrical characteristic of light figures corresponding to the symmetries of planes are invariant, while the surface treatment is different. But, the exact mechanism of pattern-shaping of light figure is not known. The present work was initiated to make a more detailed study of the relation between light figures and surface structures.

EXPERIMENTAL PROCEDURE

The arrangement for experiments is shown in Fig. 1. This consists of an optical system and a sample holder. The parallel light beams coming in through a slit from the Zircon source are projected on the surface of the sample, and the reflected light figure is developed on the screen. The sample holder is so devised as to mark the position of the projected light beam.

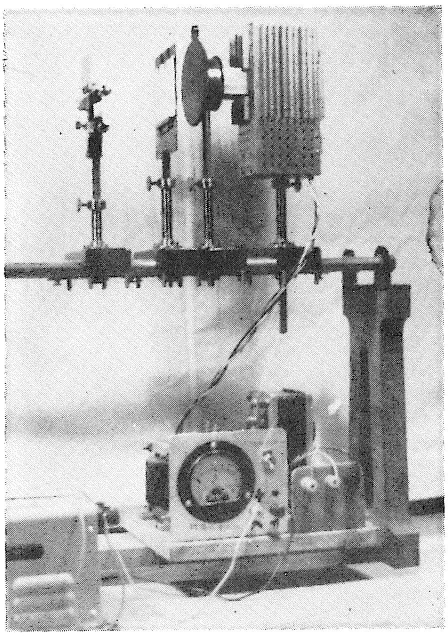

Fig. 1 Set-up of the apparatus for observing light figures.

All the treatment were performed at room temperature.

The surface structure observations were carried out with an optical microscope. The observations have been made on single crystals of 30 ohm-cm n-type Germanium which were cut in a (111) plane. The sample surfaces are finished by grinding at random by hand with abrasive powders, and then by chemical etching. Etchants used are standard CP-4, Superoxol and CPD-2.[5)]

EXPERIMENTAL RESULTS AND DISCUSSION

1. Ge polished with SiC powder

Clear light figures were obtained with Ge single crystals polished with SiC powder. As

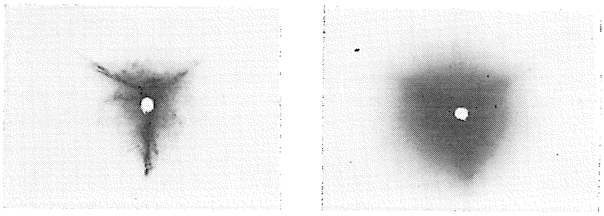

Fig. 2 (a) Fig. 2. (b)

Light figures of a (111) surface, (a) the surface polished with C-140 SiC powder, (b) the surface polished with C-300 SiC powder.

shown in Fig. 2 (a), the light figure of (111) plane lapped with C-140 SiC powder was composed of three radiated lines, the direction of which indicated (100) pole and was applicable to the determination of crystal orientation (as suggested by YAMAMOTO et. al).[6] There was a tendency for the light figures to become vague when the finer abrasive powder was used, as shown in Fig. 2 (b). Figures vanished completely when the specimen was polished carefully with Cr_2O_3 powder[7], though it was excellent for polishing to obtain mirror-like surfaces. Microstructure of (111) plane of Ge crystal polished with C-140 SiC powder appeared complex. Fig. 3 is a photographic illustration of its plane, in which the cleavage pattern is made up brightly and parallel steps are shown to run in <110> direction as YAMAMOTO et. al suggested. Lapping operation with SiC of small grit sizes will remove these structures on the surface, and the light figures disappear gradually.

Fig. 3 Microstructure of a (111) surface polished with C-140 SiC powder. (×320)

2. Chemically etched Ge

The surface of the sample was ground and polished successively with abrasive compounds such as Carborundum, Alundum and Chrominium oxide of various grit sizes. After these treatments, the specimen was immersed in CP-4 solution for 30 sec~5 min at room temperature. The surface produced by the CP-4 etchant is usually bright, smooth, and glossy (Fig. 4). No characteristic shapes of light figures were observed to appear from such surfaces, except unspecific but suggestive figure from the specimen etched in CP- 4 for 30 sec after polishing it with large grit powder (Fig. 5 (a)). Figure 5 (b) shows the surface structure of this specimen.

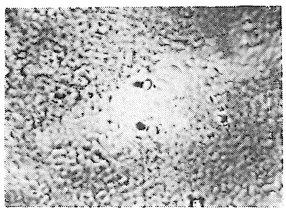

Fig. 4 Microstructure of a (111) surface etched with CP-4 for 5min. (×80)

Distinct light figures were obtained by a short-time etching with Superoxol (Fig. 6 (a)). Etching for 3 min at room temperature was satisfactory. This treatment left the Ge surface rough and etch-pitted, which was most suitable for producing the character of crystal structures. The surface structure and the typical growth type of etch pits produced by the Superoxol solution were shown in Fig. 7. The (111) face

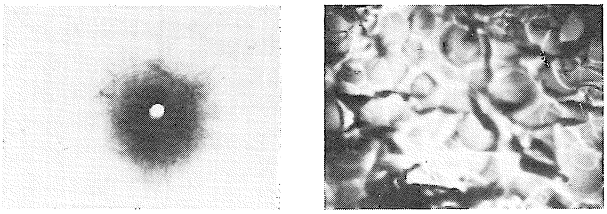

Fig. 5 (a) Fig. 5 (b)

Light figure and microstructure of a (111) surface etched with CP-4 for 30 sec after being polished in C-140 SiC powder.

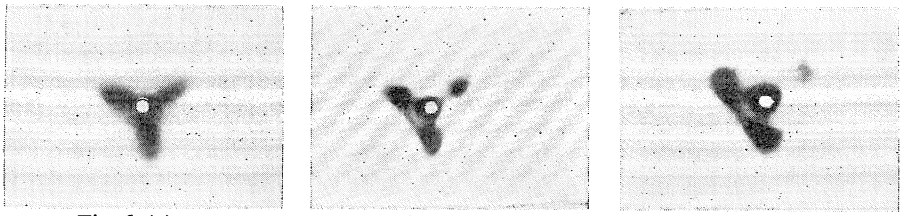

Fig. 6 (a) Fig. 6 (b) Fig. 6 (c)
Light figures of a (111) surface etched with Superoxol, (a) for 1 min, (b) for 3 min, (c) for 5 min.

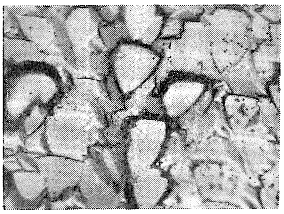

Fig. 7 Microstructure of a (111) surface etched with Superoxol for 3 min. (×600)

had triangular, flat-bottomed pits. The light figures corresponding to a series of surface structures, etched progressively deeper and larger with the prolongation of treatment periods, shifted from three arrow-shaped figure (Fig. 6 (a)) to a triangle with round vertex (Fig. 6 (b)). On the other hand, it is shown in Fig. 6 (c) that successive etching results in giving a triangle with one corner somewhat obliterated. These photograhps indicate the deviation from the original symmetrical intensity, which is presumably attributable to the preferential etching on special plane.

In order to obtain the clean surface without damaging the material, chemical polishing should take the place of mechanical lapping. Etching in CP-4 and CPD-2 etchant is better than polishing-on-a-lap method for obtaining mirror-finished surface of crystal, after these chemical operations etching in Superoxol was tried. The combination of Superoxol and CP-4, gave figure similar to the one obtained by Superoxol only, and the combination of Superoxol and CPD-2, gave no figure whatever.

Also, it can be shown by the following facts that the construction of light figure corresponds to the surface structure of crystal. The fine structure of light figure observed with the naked eye, is illustrated in Fig. 8. It shows the general structure of well defined light figures. The structure can be interpreted as superimposition of a three-slender-armed figure and a configuration of three spots. The magnitude of the spots was proportioned to the distance between the

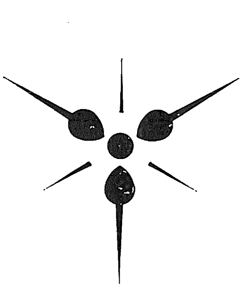

Fig. 8 Schematic diagram of light figure.

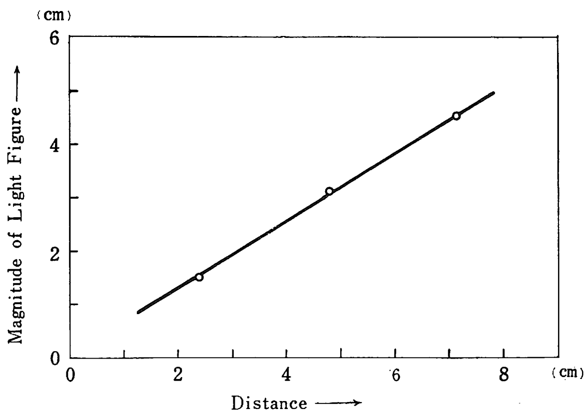

Fig. 9 Relation between shot-distance and magnitude of light figures.

screen and surface of a sample as shown in Fig. 9, and also to the dimension of the light beam projected on the specimen. It may be interpreted that the above facts show that the light figure considerably depend on the reflection from the characteristic surface etched with Superoxol. The three radiating arms of the light figure are produced by the sharp edge of the cleavage in the crystal, therefore they will vanish with proceeding of etching. In some cases certain figures, which may be termed as sub-figures, appear, some of them, representing the direction of the crystal cleavage.

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