Physical Chemistry

Academician of the Academy of Sciences of the BSSR N. N. Sirota and A. A. Tonoyan

1960

SovietRxiv

Abstract

Full Text

Physical Chemistry

Academician of the Academy of Sciences of the BSSR N. N. Sirota and A. A. Tonoyan

DETECTION OF DISLOCATIONS AND SOME FORMS OF ETCH FIGURES IN SILICON SINGLE CRYSTALS

It was shown in work ([1]) that, when silicon sections are etched, the planes (111) are revealed most distinctly; on these planes defects are observed predominantly in the form of etch pits. In this case the etch pits were associated with dislocations, chiefly in connection with the manifested structure of their surface. Work ([2]) describes a method for revealing pits in germanium and silicon, but without revealing the structure.

The purpose of the present investigation is a more detailed study of etch figures caused by the presence of dislocations and other defects, and the clarification of the nature of the etch pits.

The specimens for the investigation were cut from single crystals in the form of plates along definite crystallographic directions, mainly along planes close to (111). The exact orientation was established with the aid of an optical instrument made by I. E. Voitsekhovich and described in his diploma thesis (1959). After cutting, the plates were ground with abrasive powders of various grades (Nos. 14, 10, 7). The plates were then subjected to chemical polishing and, practically simultaneously, to subsequent etching in order to reveal dislocations and defects. Chemical polishing was carried out at a temperature of 30-35° for 2-3 min in a reagent ([3]) consisting of 10 cm^3 of hydrofluoric acid (37-38%), 10 cm^3 of concentrated nitric acid, and 10 cm^3 of glacial acetic acid. Then 15 cm^3 of bidistilled water was added to this reagent, and at the same temperature etching and the revelation of defects were carried out for 1.5-2 min. In contrast to the recommendations of paper ([3]), polishing and etching were performed with heating.

Several characteristic types of etch figures can be distinguished, appearing inside pits and on the (111) plane, caused by the presence of dislocations, defects, and growth structures.

Etch figures having the form of a spiral are frequently encountered. Figure 1 shows typical spiral etch figures: *a*—a left-handed spiral, *b*—a right-handed spiral. Similar spiral-shaped etch figures in silicon single crystals were found in works ([4], [5]).

In some cases, for example Fig. 1*c*, etch figures are found in which a spiral is visible in the inner part, while in the outer part it degenerates into a terrace structure in the form of triangles of various sizes superimposed on one another. Sometimes, side by side, etch figures are found in the form of a spiral and in an intermediate terrace form (Fig. 1*g*). Of considerable interest is the presence of etch figures having the character of spirals, at the beginning and end of which there are, in turn, spirals twisted in mutually perpendicular planes (Fig. 1*d*).

For silicon single crystals in the (111) plane and in planes deviating only slightly from it, the presence is also characteristic of etch figures only in the form of nonintersecting terraces, appearing as superimposed upon one another.

Fig. 1.
a –4666×,
b –1833×,
c –1333×,
d –1333×,
e –3125×.

Fig. 2.
a –1500×,
b –170×,
c –362×.

of polygons of different sizes superimposed on one another. In Fig. 2a etch figures are shown with clearly expressed terraces, obliquely displaced in one direction.

On single crystals of low-ohmic silicon ($\rho = 0.1$ ohm · cm), etching in a boiling 20% aqueous KOH solution for 10 min reveals, at comparatively low magnification, etch figures consisting of sharply outlined closed polygons superimposed on one another, the sides of which are practically parallel to one another (Fig. 2b). In some cases, along with individual similar figures, groups of analogous figures are observed, arranged side by side and bordering one another (Fig. 2c).

In the etch figures described by us, the pitch of the screw spirals is of the order of 100-1000 Å; the distance between nonintersecting terraces is usually of the order of 1000-10000 Å. In this case, both the spirals and the nonintersecting terraces, associated with dislocations, are growth figures; they lie predominantly in the (111) planes. Measurements of microhardness carried out by us showed, in particular, that in some cases, as the etch pits are approached and at the emergence of dislocations, the microhardness H_μ decreases significantly (Table 1).

Table 1

H_μ, kg/mm^2	218.9	1430.5	1130.3	1130.3	857.4	601.8	541.7	857.4	329.61
r, mm	0.25	0.22	0.19	0.16	0.13	0.1	0.7	0.04	0.01

Subsequently, it will be of considerable interest to investigate changes in physical and mechanical properties in connection with the dislocation density and the form of the etch figures.

The new results obtained indicate that etch pits are, as a rule, associated with dislocations and disturbances caused by growth structures.

Department of Solid-State and Semiconductor Physics
Academy of Sciences of the BSSR

Received
17 VI 1960

REFERENCES

1. N. N. Sirota, A. V. Shabaeva, *Inzh.-Fiz. Zhurn.*, **1**, 2, No. 10, 57 (1959).
2. D. Trachtenberg, S. M. Feinstein, *Fizika tverdogo tela*, **1**, No. 3 (1959).
3. C. A. F. T. Spry, *Proc. Phys. Soc.*, **69**, No. 438, 689 (1956).
4. F. L. Vogel, L. C. Lovell, *J. Appl. Phys.*, **27**, 12 (1956).
5. J. Matsukura, T. Suzuki, *J. Phys. Soc. Japan*, **12**, No. 8, 976 (1957).

Note: Figure translations are in progress. See original paper for figures.