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Abstract**Full Text**

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CRYSTALLOGRAPHY

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**A DIRECT METHOD FOR INVESTIGATING
THE ELECTRICAL MICRORELIEF OF CRYSTALLINE SURFACES***(Presented by Academician A. V. Shubnikov on 18 June 1965)*

The surface properties of crystalline materials determine many of their physical and chemical properties and such heterogeneous processes as adsorption, catalysis, and epitaxial growth. At the Institute of Crystallography of the Academy of Sciences of the USSR, an electron-microscopic method has been developed for studying surface inhomogeneities, based on the fact that structural defects are revealed and decorated by means of heterogeneous crystallization reactions proceeding selectively on these defects. From crystallization patterns one can judge the quantity and the topography of the distribution of various structural defects. In works ^(1,2), by means of the decorating crystallization reaction of lead sulfide, the layered distribution of impurities along single-crystal silicon ingots grown by the Czochralski method was studied with high resolution.

Impurity centers in most cases are electrically charged ⁽³⁾, which leads to the appearance of an electrical microrelief of the surface associated with them. This electrical microrelief can be revealed with the aid of a new class of decorating agents—charged microparticles—which selectively interact with charged microregions of the surface. As charged decorating substances, colloidal particles of size 50–1000 Å were used. Colloidal particles of lead selenide 300–500 Å in size proved convenient for the experiment, possessing high contrast in the electron microscope. These particles were readily adsorbed by crystalline surfaces from aqueous solutions, then separated from the surface and examined by transmission in the electron microscope.

Upon adsorption of charged colloidal particles of lead selenide by the surface of silicon specimens grown by the Czochralski method and cut parallel to the growth axis, layered decoration patterns are formed (Fig. 1), analogous to those obtained in works ^(1,2,4). This shows that both in the decorating crystallization reaction and in decoration by charged particles, the surface structural defects revealed are identical and are impurity centers, sources of local charged microinhomogeneities of the surface. In the regions between the bands containing the most active impurity centers, charged lead selenide particles are not adsorbed.

Fig. 1

Figure 1: Fig. 1

The density of charged particles on the surface of the silicon specimens studied is $(2 \div 3) \cdot 10^{10}/\text{cm}^2$, which is equal to the density of decorating lead sulfide microcrystallites formed on this surface during crystallization. This once again confirms the identical nature of the crystallization centers and the adsorption centers of charged particles.

The electrical nature of the surface forces interacting with charged colloidal particles is proved by the range of this interaction. On the surface of silicon crystals, pro-

intermediate dielectric layers (polymer films) with thicknesses from 100 to 1500 Å, and charged microparticles were adsorbed on them. It turned out that analogous layered decoration patterns are obtained both directly on the silicon surface and on the intermediate layers (Fig. 2). When shielding metallic layers are deposited on the silicon surface, the layered decoration patterns disappear completely.

Fig. 1. Layered distribution of decorating charged PbSe particles along the length of a silicon crystal grown by the Czochralski method

From the experiments performed it follows that the distribution of charged decorating particles on the surfaces under study corresponds to the electrical microrelief of these surfaces, caused by charged structural defects, in particular by impurity centers. It is very important that charged impurity centers are the most active sites of the surface, and the action of the electrical forces of these centers extends over very large distances. The latter accounts for the possibility of a long-range mechanism of many heterogeneous surface reactions ⁽⁵⁾.

Interesting patterns of electrical microrelief are revealed when decorating epitaxial silicon layers* (Figs. 3 and 4). On the surface of these layers there form packings of charged microparticles possessing both almost ideal ordering and disordering of the structure. Along with the packings there is a certain number of isolated charged particles. The formation of packings is explained by the fact that individual charged particles of lead selenide of identical size and shape, repelling one another owing to the like sign of their charges, are simultaneously attracted by oppositely charged microparticles of the surface. The sizes of the packings interacting with the electric fields of impurity centers reach several microns, and their density is $\sim 10^8/\text{cm}^2$. The presence of packings of charged micro-

* The epitaxial layers were kindly provided to us by E. I. Givargizov, to whom we express our deep gratitude.

Fig. 2. Layered distribution of decorating charged PbSe particles along the length of a silicon crystal, deposited on an intermediate layer of thickness ~ 150

Å

Fig. 3. Decoration pattern, by charged particles, of epitaxially grown silicon layers

Fig. 4. Packing of charged PbSe particles on an epitaxial silicon layer

particles indicates segregation of impurities in individual regions of the surface; moreover, there is a very strong localization of charged microregions of the surface, acting quite independently of neighboring regions. The sizes and structure of the deposits must reflect, in a complex way, the structure of the charged microareas of the surface, whose fine structure apparently determines the selectivity of action of the active regions of the surface. Each deposit decorating the local electric field of charged impurities of one sign essentially reveals a micro- $p-n$ junction. The stripwise aggregates of micro- $p-n$ junctions (Figs. 1 and 2) constitute layered $p-n$ junctions with pseudoperiods of several microns.

Thus, the new method of decoration by means of charged colloidal particles (microelectrophotography) makes it possible, with very high resolution, to establish the sizes, number, fine structure, and nature of the distribution of charged microregions of crystalline surfaces.

L. E. Guseva took part in the work.

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