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Divergent-beam X-ray structural studies of a disturbed surface layer in silicon plates

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Abstract. A Kossel chamber for reflected-beam X-ray studying of single crystal surfaces has been developed on the basis of a BS-340 scanning electron microscope. We have examined the structure of a disturbed layer of silicon plates after chemico-mechanical polishing. The intensity of X-ray reflection from the lattice planes intersecting a polished surface of a plate characterizes the perfection degree of the disturbed layer, is of a periodic nature and exhibits a tendency to damp deep within the plate.

Keywords: Kossel diffraction, X-ray, structure, silicon.

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Kossel's method (divergent-beam X-ray diffraction) is used for precision studies of the lattice structure perfetion of single crystals. A number of publications on the subject are reviewed in [1]. K. Lonsdale was the first to apply the Kossel method to study the perfection of the diamond lattice structure [2]. Her paper gives not only the values of interplanar spacings in natural diamonds, but analyzes the Kossel's line profile in the kinematic approximation. Refs [3-5] report the use of Kossel's method for measurements of interplanar spacings, analysis of stressed state, studying the effect of system growth on the Kossel's line intensity profile of diamonds of various origin as well as for determination of interplanar spacing anisotropy of diamond single crystals in equivalent crystallographic directions.

The experimental results showing the possibility to use Kossel's method for studying the layer disturbed due to chemico-mechanical polishing of silicon plates are discussed in the present paper.

A Kossel chamber for reflected-beam X-ray studying of the surface structure of single crystals has been made as an attachment to a BS-340 scanning electron microscope. A schematic diagram of taking Kossel's diffraction patterns is given in Fig. 1. As an anode, to obtain the initial X-ray beam, a thin (~ 5 to 10 μm) foil of any material can be used. In this work, we used X-ray characteristic CuK$\alpha_{1,2}$ radiation from a 5-μm thick foil, accelerating voltage (30 kV) of the BS-340 microscope electron beam and a FT101 highly contrasting film taking Kossel's diffraction patterns is given in Fig. 1.

It should be noted that with this geometry, the thickness of a layer from which diffraction patterns can be taken is about 30 μm. As opposed to the divergent-beam X-ray photographing with the use of extinction lines (the case of Laue [1 – 5]) where the information is gained from the entire crystal, X-ray diffraction patterns in a reflected light allow one to examine surface layers of the crystal, which is of great importance when studying the imperfect structures of semiconducting plates for microelectronics products.

Monocrystalline silicon plates for microelectronics after chemico-mechanical polishing parallel to the (111) plane have been the subjects of our investigation. Fig. 2 shows a typical Kossel diffraction pattern from a silicon single crystal in the CuK$\alpha_{1,2}$ radiation. The symmetry of diffraction reflections from the {444} and {622} planes about the axis of the initial X-ray beam indicates that the crystal has been well oriented during its chemico-mechanical treatment.

When studying the surface layer, particular attention should be given to the Kossel lines produced due to the the scattering of rays by the {442} planes of the lattice that intersect the polished surface of the plate. It follows from Fig. 3a (indicated fragment) that due to radiation scattering by imperfect surface layers of the crystal, the Kossel lines broaden.

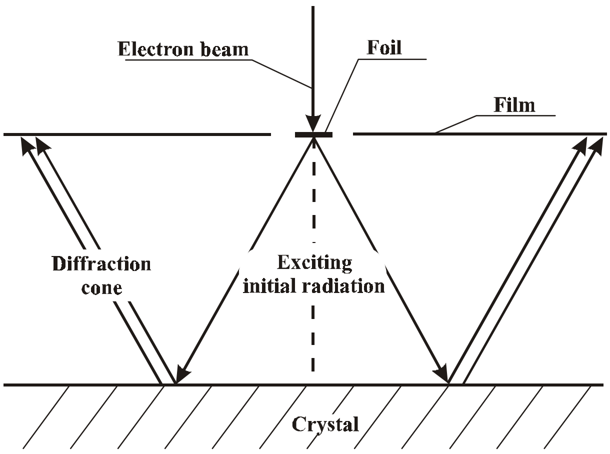


Fig. 1. Schematic diagram of taking the Kossel diffraction pattern in a reflected X-ray.

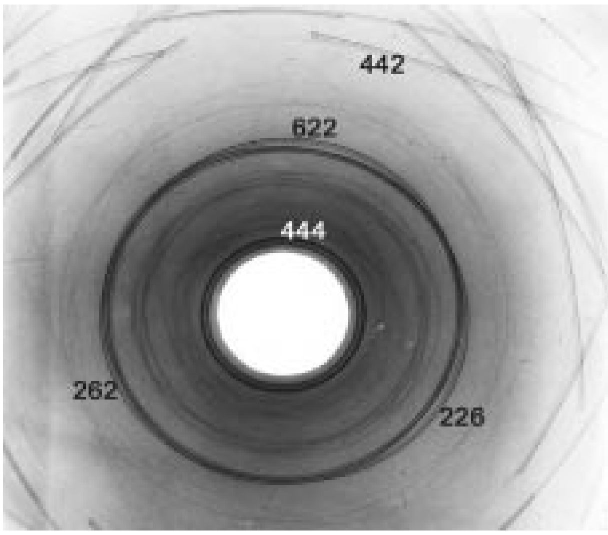


Fig. 2. Fragment of the Kossel diffraction pattern from a plate of polished silicon. Cu$K\alpha_{1,2}$ radiation, the [111] direction.

Microdensitometric measurements of the line structure have shown that the intensity of the X-ray reflection is of a periodic nature and tends to damp deep within the plate. Taking into account the dimensions of diffraction nonuniformity of the line in the (111) plane as well as the angle between the (442) and the (111) crystallographic planes, we have calculated the total thickness of the disturbed layer and the thickness of dislocation sublayers that constitute the former (see Fig. 3b).

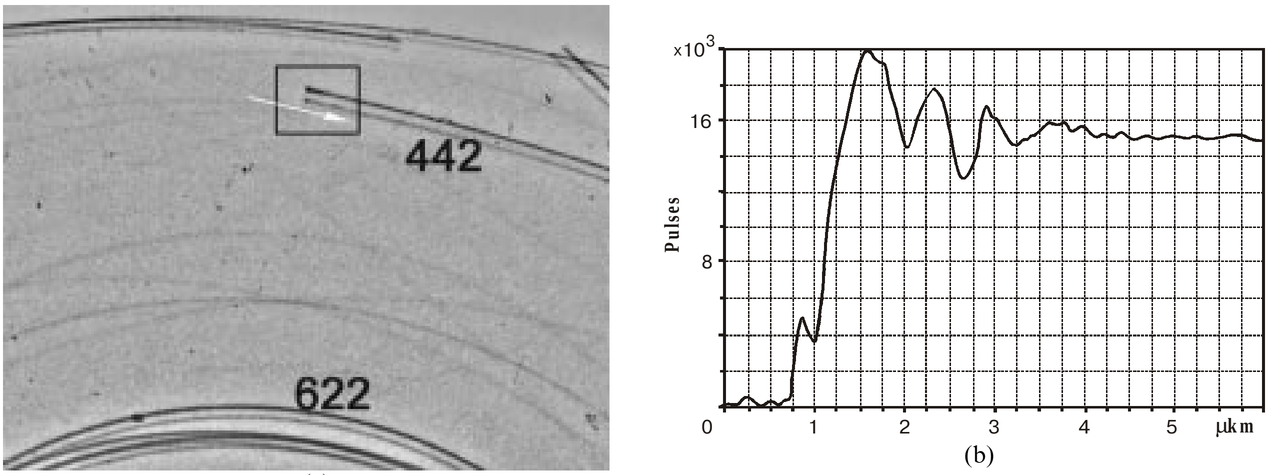
Our findings agree well with the data obtained by a destructive check of the disturbed layer from the electron scattering [6] (see Figs 4a and b).

It follows from Fig. 4b that the average defect content of the disturbed layer decreases according to exponential curve marked –1.5X. Thus, the data obtained by both the method of scattered electrons and Kossel's method point to the dislocation mechanism of wear in machining the material surface.

Fragments of Kossel diffraction patterns from silicon plates after the chemico-mechanical polishing and subsequent treatment with an abrasive powder are given in Figs 5a and b. In Fig. 5b regions (some of them are indicated by arrows) typical of lines due to non-centrosymmetrical X-ray reflection by the lattice planes are observed in diffraction lines. It follows that under the mechanical action of abrasive grits, around a scratch dislocation distortions of the lattice (in sizes above 30 μm) appear, in which recording Kossel's lines is difficult.

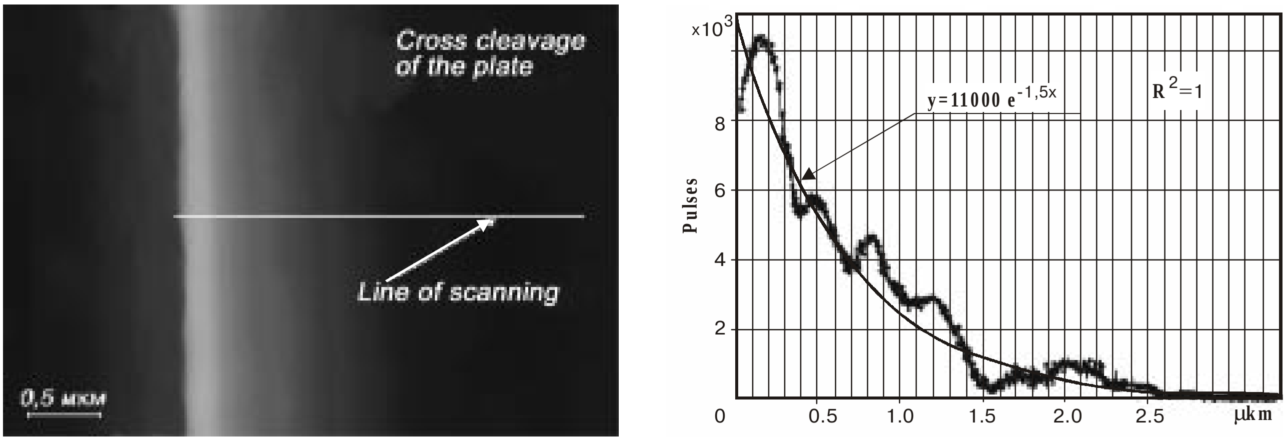
Thus, Kossel's method allows us to study the thickness and structure of a disturbed layer of silicon plates used in production of electronic microcircuits. It can be used as nondestructive method of controlling the surface quality and for studying mechanisms of material machinability.

Some papers report the study of the surface of semiconducting materials using mutual arrangement of Kossel's lines in the diffraction patterns. In Ref. [7], the Kossel's method was applied to define the difference in lattice parameters between the initial semiconducting



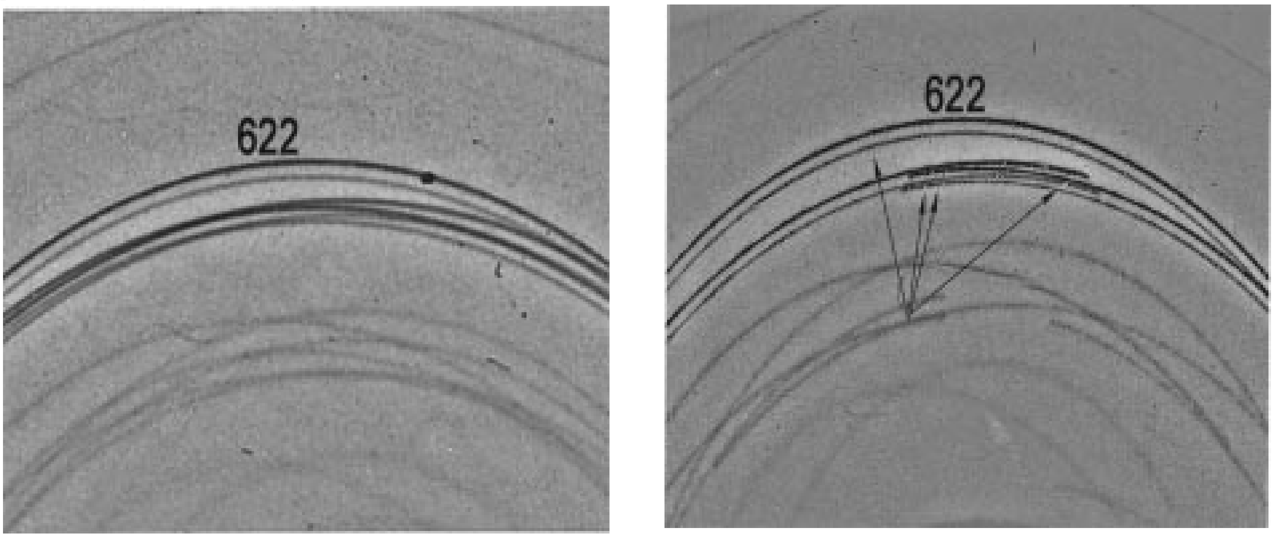
(a) (b)

Fig. 3. Fragment of the Kossel diffraction pattern from the silicon plate surface in Cu$K\alpha_{1,2}$ radiation (a) and microdensity pattern (b) from the 442 line when scanned along the indicated direction.



(a) (b)

Fig. 4. SEM images of: a cleavage of the silicon plate (magnification 40,000x (a)) and intensity of scattered electrons during the probe scanning of the cleavage (b).



(a) (b)

Fig. 5. Fragments of the Kossel diffraction pattern from the polished silicon plate (a) and from the same plate after machining with an abrasive powder (b).

crystal and epitaxial coating designed for microelectronics. The authors reported the formation of an imperfect layer at the crystal-coating interface that affects the coating structure.

Unfortunately, the number of publications on experimental studies of the surface of semiconducting materials using the intensity profile of Kossel's lines is limited. Currently nanotechnologies are being intensively developed in microelectronics, and analysis of the intensity profiles of Kossel's lines can provide an additional information for studying the materials for quantum electronics.

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