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MICROINDENTATION AND MECHANICAL PROPERTIES OF SILICON

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The paper presents results of the research concerning microindentation of physical and mechanical properties of materials, which are used in modern production technology of electronic devices. On the basis of measurements, the analysis of microhardness, micro-fragility and fracture toughness of silicon, grown according to the Czochralski method (Cz-Si), was carried out, when the combined magnetic fields were applied to the melt.

Introduction. Modern economic conditions of microelectronic techniques production require reduction of material consumption and material production, and the percentage output increase of effective devices. However, the basic microelectronic materials (silicon and structure on its basis) are fragile and because of that they are prone to mechanical damages. This causes necessity of additional researches of physico-mechanical characteristics of the semiconductors, which are used in the microelectronics [1–7].

Features of microindentation of semiconductors. Researches in the field of semiconductor materials should be carried out and based on a number of features of these materials in comparison with metals.

Defect-impurity composition of covalent materials (which include semiconductors) is very mobile. Its change in semiconductors structures occurs during the growth of single crystals, and their technological processing (doping, irradiation, heat treatment) and exploitation of finished products. Formation of strong internal stress in a crystal at all stages leads to the appearance of micro-defects and as a result to brittle fracture of the material.

Basic semiconductor materials (silicon and germanium) are high purity materials, which do not have any dislocations. The predominant mechanism of deformation in semiconductor single crystals and structures (at least in initial stages) is displacement (offset) of the material by movement of the individual (interstitial) atoms, point defects.

Microindentation was selected as a method of research due to several reasons:

– Microindentation is a method of non-destructive control. This allows to use it at different stages of technological chain of semiconductor devices production;

– This method does not require complex technical devices for measurements and processing results;

– Microindentation method in conjunction with methods of mathematical statistics allows to get results with an error of no more than 5-7%;

– Microindentation allows to calculate some strength characteristics and evaluate physical and mechanical properties of material comprehensively according to one imprint;

– In this paper, by applying indentation (figure 1), three strength characteristics were calculated: microhardness (H), micro-fragility (Z) and first-order critical stress intensity factor (fracture toughness, K_{1C}).

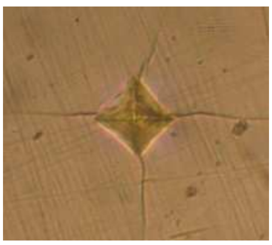


Fig. 1. Micrograph of the imprint after indentation of silicon by the Vickers pyramid

Microhardness used for Vickers pyramid indentation is calculated by the formula

$$H = \frac{1,854P}{d^2},$$

where P – the load on the indenter; d – diagonal imprint.

Measurement results were used in evaluating of silicon micro-fragility statistical methods of processing. A score of fragility was assigned to each of the applied imprints, which was determined by the arbitrary scale, which takes into account the number of cracks and chips near the imprint and the nature of their evolution [1].

During the measurements the number of imprints, which was corresponding to each score of fragility, was fixed. According to the results of measurements histograms were plotted. The total average score of fragility of the material was calculated by the formula

$$Z = \sum_{i=0}^{5} i \cdot n_i ,$$

where n_i – the relative number of imprints that have the mark of fragility i = 0, 1, 2, 3, 4, 5.

The propensity of the material to crack formation was evaluated by the value of the critical coefficient of stress intensity of the first kind (crack resistance):

$$K_{1C} = k\left(E/H\right)^{1/2} P/l^{3/2} ,$$

where k – empirical calibration coefficient; E – Young's modulus; l – length of the crack.

Experimental results and discussion. Consider the possibility of obtaining information on the strength properties of semiconductor materials concerning microindentation on the example of silicon single crystals, grown by Czochralski method (Cz-Si), when combined magnetic fields were applied to the melt. Overlay of magnetic fields on the melt allows to manage the processes of convection in the melt and to vary the defect-impurity composition of the grown single crystal [8].

Silicon plates 2 mm thick were cut from the upper and lower parts of the plate. They were subjected to the same treatment in the process of growing and passed identical chemical-mechanical polishing. Indentation was performed on a PMT-3 using the Vickers pyramid by a standard technique with loads of 50–200 g.

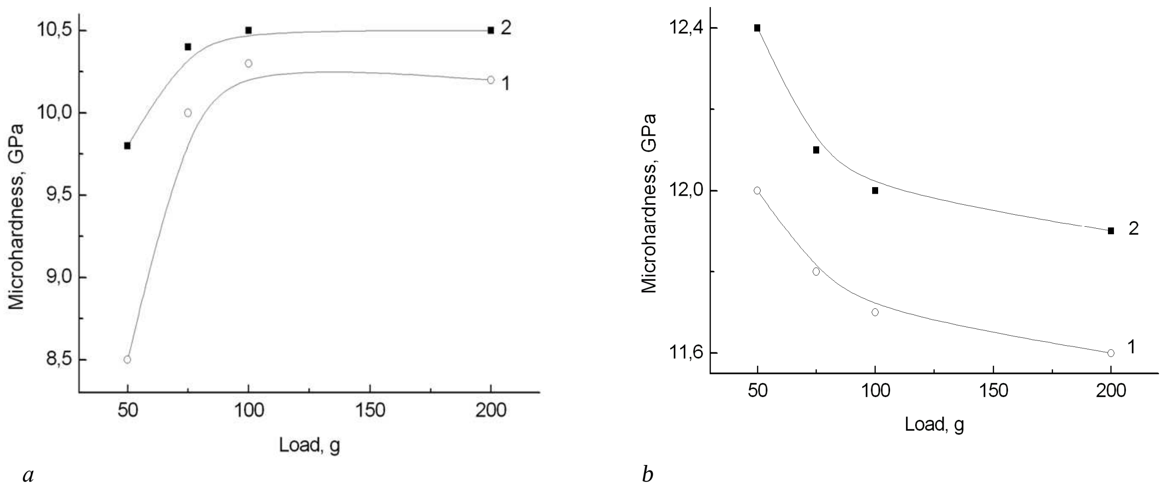


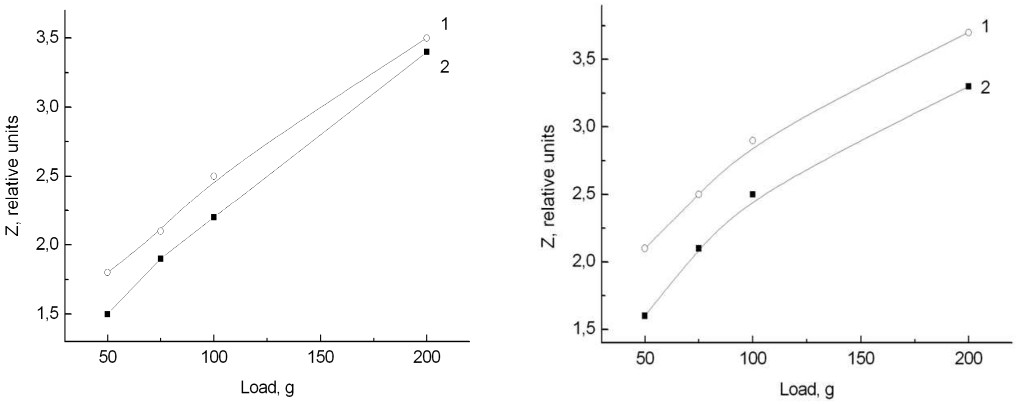
Fig. 2. Dependence of the microhardness on the load for plates cut from the upper (1) and lower (2) parts of the ingot to the orientation of *a* – <100> and *b* – <111>

Dependence of the silicon microhardness on the load **on the indenter** is presented in figure 2. Analysis of these dependencies leads to the following conclusions:

– A strong dependence of the microhardness of single crystal orientation was detected. The microhardness of plates cut from single crystals with the orientation <100> was lower by 10–15% than the plates with the orientation <111>.

– It was found that the microhardness of samples which were cut from the lower part of the ingot, was slightly higher than H samples which were cut from the upper part the same ingot.

– For samples from the lower part of the ingot the effect of the surface hardening was observed, comprising in the microhardness decrease by 10–20% as the load increases from 50 to 100 g. This effect is typical for the traditional (Cz-Si). However, the microhardness for samples cut from the upper parts of the ingot, measured under a load of 50 g, was close or lower than the microhardness that measured with a load of 100 g.



a *b*

Fig. 3. Dependence of micro-fragility of the load for plates cut from the upper (1) and lower (2) parts of the ingot to the orientation of *a* – <100> and *b* – <111>.

Analysis of micro-fragility dependence on the load (fig. 3) shows:

– Micro-fragility of single crystals increases with increasing of the load.

– Values of micro-fragility of investigated single crystals are close to the values which are typical for traditional Cz-Si [9-11].

– Values of micro-fragility of the plates cut from the lower part of ingot were slightly lower than of the plates cut from the upper part of the same ingot regardless of the ingot orientation.

– When applying magnetic fields to the melt, the amount of oxygen-containing growth defects increases. The increase of micro-fragility of the silicon single crystals in comparison with the traditional Cz-Si was observed [9–11].

Studies of tendency of silicon single crystals to crack formation (figure 4) showed the following results:

– Crack resistance was decreasing with the load increasing (for all samples).

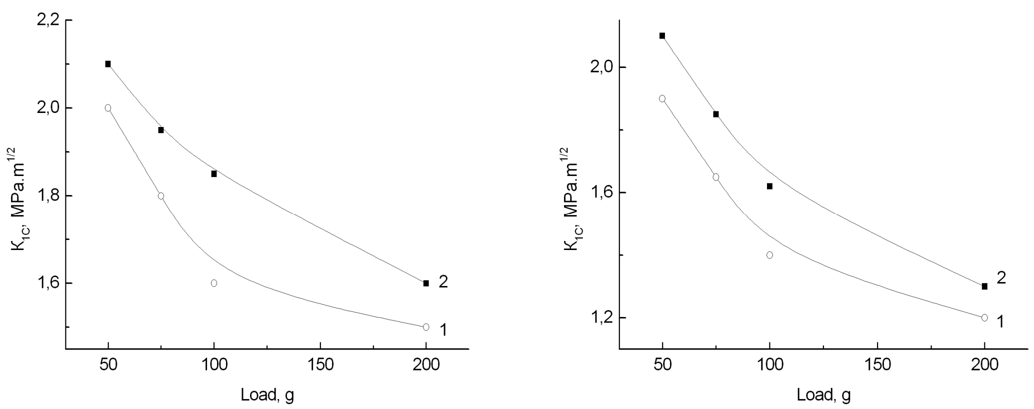
– Values of K_{1C} of investigated single crystals were close to the values that are typical for traditional Cz-Si [9–11].

– The critical coefficient of stress intensity of the first kind K_{1C} was slightly higher in ingots with orientation <100>, especially under heavy loads of 100–200 g.

– K_{1C} from the ingot to ingot increases with decreasing the load.

– K_{1C} is lightly higher in plates, cut from the lower parts of ingots, for both investigated orientations of ingots.

– The presence of oxygen-containing growth defects formed during the growth of a single crystal in complex magnetic fields led to some reduction in silicon crack resistance compared with traditional Cz-Si [9–11].



a *b*

Fig. 4. The dependence of the critical stress intensity factor K_{1C} from the load for plates cut from the upper (1) and lower (2) parts of the ingot to the orientation *a* – <100> and *b* – <111>

Increasing of the microhardness and fracture toughness K_{1C} of plates, cut from the lower parts of the ingots, due to the fact that in the lower parts the ingots technological impurities concentration is substantially higher, due to the effect of impurities pushing into the melt during the growth process.

Conclusion. Using the example of silicon which was grown overlaid with magnetic fields on the melt, it was found that all the strength characteristics of silicon substantially depend on growing conditions. Variation of crystal growing conditions allows us to manage physical and mechanical properties of the material. Microindentation is a method that allows us to assess the range of the strength characteristics of the semiconductor material. This method also allows us to choose material with the most optimal physical and mechanical properties for the production of microelectronics and electrical devices.

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