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Study of the mechanical strength of thin silicon wafers in the dependance on their surface treatment during thinning

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Received May 4, 2022
Revised July 7, 2022
Accepted July 18, 2022

The mechanical strength of silicon wafers of 100 μm thickness was studied. Loading of the wafers was carried out by the „ring-on ring" method, stress and deflection under the small ring were determined by finite element modeling. The validity of the calculation model was checked by comparing the dependences of the deflection under the small ring on the load obtained in the experiment and by the simulation. The effect of methods of wafers obtaining and their surface treatment on the strength, as well as the connection between the strength and surface roughness characteristics were shown.

Keywords: silicon, strength, chemical polishing treatment, surface roughness.

DOI: 10.21883/TPL.2022.09.55077.19244

Due to a number of physical, technological, and economic reasons, semiconducting silicon wafers are getting more and more thin; the thickness of wafers used in power electronics and photovoltaic devices often drops below 100 μm. The issue of mechanical strength of wafers of such a small thickness attracts intense interest. Three- or four-point uniaxial bending of strips [1–3] and biaxial (axially symmetric) bending [4–6] are commonly used as loading methods to estimate the strength of wafers, while indentation [7] and hydrostatic pressure [8] are applied less frequently. Strength calculations are performed either in accordance with the classical elasticity theory formulae specified in ASTM C 1161-02c (for uniaxial bending) and ASTM C 1499-15 (for axially symmetric bending) standards [2,3] or using the finite element method [1,4–6]. Three- and four-point bending techniques have a drawback in that the breakdown of a tested wafer is induced by stress concentrators at its edges. The method for measurement of the strength of thin silicon wafers by biaxial bending with a ring support and a ring loading edge (the so-called ring-on-ring test) is currently being used more and more often. Since wafer edges, which act as stress concentrators, protrude beyond the large ring, they are loaded only lightly and do not affect the initiation of a breaking crack. In the present study, this technique is applied in tests of thin silicon wafers of a small diameter. Their strength is determined, and an attempt to establish a correlation between the obtained values and the specifics of fabrication of wafers and treatment of their surfaces is made.

All the tested samples were fabricated from single-crystalline silicon, but the crystals were grown and doped in different ways, and different types of surface treatment were applied in the process of wafer thinning. Single-crystalline silicon wafers prepared in three different ways were used to fabricate these samples (Table 1).

Samples of the first type were standard (100) Czochralski-grown *p*-type wafers for epitaxy doped with boron to $N_A = (5-10) \cdot 10^{18}\,\mathrm{cm}^{-3}$. Their resistivity was $\sim 5-10\,\mathrm{m\Omega \cdot cm}$, and their diameter and thickness were 100 mm and 420 μm, respectively. Substrate thinning was performed by grinding with a loose abrasive with a base grain size of 14 μm (to a thickness of 200 μm), diamond pastes with grains $3-5\,\mu$m in size (to a thickness of 130 μm), and a diamond paste with 1 μm grains (to a thickness of 110 μm). The end thickness of 100 μm was achieved though finishing chemical-mechanical polishing (CMP).

Epitaxial silicon produced by sequential growth of *p*- and *n*-type layers with a thickness of 50 μm on single-crystalline Si wafers was the second material type subjected to testing. The level of doping of epitaxial *p*-type layers with an acceptor impurity (boron) and *n*-type layers with a donor impurity (phosphorus) was $\sim 2 \cdot 10^{14}\,\mathrm{cm}^{-3}$. These wafers were subsequently thinned to 100 μm by removing the substrate completely through grinding, polishing, and finishing CMP.

Samples of the third type („solar" silicon) were grown by the Czochralski technique, featured *n*-type conductivity, and were doped with phosphorus to $N_D = 1 \cdot 10^{15}\,\mathrm{cm}^{-3}$ (resistivity $5\,\Omega \cdot \mathrm{cm}$). The initial (100) silicon wafers of this type had the form of „pseudo-squares" with a side length of 125 mm and a thickness of 180 μm. Wafers 100 mm in diameter were cut out of these pseudo-squares and thinned to 100 μm by removing layers with a thickness of approximately 40 μm from both sides. SiC powder with a base grain size of 14 and 7 μm was used for grinding.

Table 1. Characteristics of wafers and silicon strength

Type of Si wafers	Finishing treatment	Strength, MPa
p-Si (100) (Czochralski technique)	CMP	1010 ± 456
p/*n*-Si (100) (epitaxy)	CMP	754 ± 506
	Diamond polishing	152 ± 59
n-Si (100) (Czochralski technique)	Grinding with SiC M7 powder	144 ± 33
	Grinding with SiC M14 powder	127 ± 13

The samples were ground and polished with diamond pastes at AO „PK „FID-Tekhnika“ with the use of equipment produced by Peter Wolters GmbH. Glass grinding disks 500 mm in diameter were used for wafer processing. The axial rotation rates of disks and wafers were 18–20 and $\sim 10\,\mathrm{min}^{-1}$, respectively. The downward pressure on the wafers subjected to grinding was $\sim 50\,\mathrm{g/cm}^2$. A suspension of diamond particles $3-5\,\mu$m in size was used for wafer polishing. This suspension was prepared by mixing one part (by weight) of ASM 5/3 diamond powder and 50 parts of a mixture of equal amounts of synthanol and glycerol. Polishing was performed with the use of Unipol-1202 machines and Simba-N polishing disks 300 mm in diameter produced by MetCata GmbH. The rotation rates of the polishing member and wafers were roughly equal to 100 and $20\,\mathrm{min}^{-1}$, respectively, and the specific pressure applied to the processed wafer was $80-100\,\mathrm{g/cm}^2$. A polishing suspension of fumed silica in KOH or ethylenediamine with a pH of 10–12 was used for CMP, which was performed with Unipol-1202 machines and Simba-N polishing disks at a polishing member rotation rate on the order of $200\,\mathrm{min}^{-1}$ and a specific pressure of $250-350\,\mathrm{g/cm}^2$. At the first polishing stage, a suspension containing 10–20 wt.% of silica nanoparticles 10–40 nm in size removed a material layer with a thickness on the order of $25-30\,\mu$m. Amorphous SiO_2 nanoparticles 7 nm in size were used at the second stage. The polishing member rotation rate and the specific pressure were reduced to $\sim 80\,\mathrm{min}^{-1}$ and $\sim 100\,\mathrm{g/cm}^2$, respectively. This two-stage finishing treatment allowed us to reduce considerably the rate of removal of the wafer material at the second stage and thus obtain silicon wafers with the highest possible surface smoothness. The thickness of the silicon layer removed at the second stage of finishing treatment normally did not exceed $3-5\,\mu$m.

Samples for strength tests in the form of disks 11.8 mm in diameter were cut out of thinned wafers by pulsed laser cutting. A „MiniMarker 2“ fiber laser with a wavelength of $1.06\,\mu$m operated in the ablation mode was used for the purpose. The temperature in the cut region in the chosen cutting mode (pulse duration: 12–30 ns; mean luminous power: 5–10 W, pulse repetition rate: 20–40 kHz; luminous spot motion rate: 400–800 mm/s) did not exceed 100°C, and the thickness of cut layers was on the order of $10\,\mu$m.

The thickness of wafers with a diameter of 100 mm and disks 11.8 mm in diameter was measured with an accuracy no worse than $1\,\mu$m, which was achieved using an LIR-19A digital linear displacement transducer with a measuring rod, an LIR-500A digital readout unit, and an S-III M optical measuring stand with a table. The thickness of silicon disks 100 mm in diameter after thinning was $100 \pm 5\,\mu$m. Disks 11.8 mm in diameter were cut out of the central part of thinned wafers, which was the most uniform in thickness. The variation of thickness of individual disks within their diameter of 11.8 mm did not exceed $1\,\mu$m, while the spread of mean thickness of these disks within groups selected for measurements was $\pm 2\,\mu$m.

The strength of wafers was determined using an Instron 1342 multi-purpose testing machine with an attachment for axially symmetric bending designed at the Ioffe Institute. A set of support and load rings with diameters $2b = 8.4$ mm and $2a = 4.4$ mm, respectively, was fabricated for these tests. The loading rate was 0.2 mm/min. Force F was measured in experiments as a function of displacement $\Delta l = w(a)$ of the measuring rod of the testing machine (i.e., of the wafer deflection under the load ring). The CMP-treated side was subjected to tension in tests of samples of the second type. Wafer stresses were calculated using the finite element method (FEM). Calculations were carried out in Comsol Multiphysics. An axially symmetric model of a wafer with rectangular finite elements and a characteristic mesh size of approximately one quarter of the wafer thickness was used. The lack of displacement along the contact perimeter of the support ring was set as the boundary condition. Since stresses are rather hard to determine directly in an experiment[1] and displacement $w(a)$ under the small ring is provided by the testing machine, the validity of the chosen model was verified by comparing the experimental dependence of wafer deflection $w(a)$ on load F to the one derived by FEM. The dependence of the maximum stress in the wafer (i.e., radial stresses under the small-radius ring) on load and the loading diagram are presented in Fig. 1. The characteristics of the sample material and the mechanical strength of samples, which was characterized by the calculated breaking stress value, are listed in Table 1. The mean strength value and its root-mean-square deviation were calculated based on the results of 10–30 tests of the same kind.

[1] The potential for X-ray determination of stresses was discussed in [6].

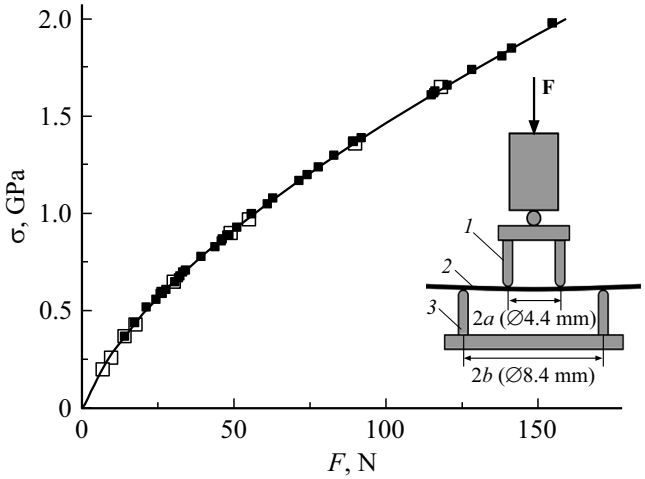

Figure 1. Dependence of the maximum tensile stress in the sample on the applied load calculated by FEM. Symbols denote the strength of epitaxial (open squares) and Czochralski-grown (filled squares) silicon after finishing CMP. The loading diagram is shown in the inset: *1* — load ring, *2* — sample, *3* — support ring.

Solar silicon samples, which were subjected only to grinding with a loose abrasive with a base grain size of 14 and 7 μm had the lowest strength values (127 and 144 MPa). The samples polished with a diamond paste had a slightly higher strength value (152 MPa). However, all of them were inferior in terms of strength to samples subjected to CMP. The method of production of silicon (epitaxy or Czochralski growth) affects its strength parameters. The strength of samples cut out of *p*-type Czochralski-grown crystals after two-stage CMP was ~ 1 GPa. In the case of epitaxially grown silicon layers, the mean strength of wafers after two-stage polishing was 0.75 GPa. The results for samples subjected to mechanical treatment are close to the available literature data [1–5]. The strength of samples subjected to two-stage CMP detailed above was higher than the values reported in [4,5] for samples subjected to common single-stage chemical polishing. This is indicative of the capacity of silicon to withstand high loads.

Weibull distribution $P(\sigma)$ is commonly used to analyze the spread of strength of brittle materials such as silicon. This distribution has the following form:

$$P(\sigma) = 1 - \exp\left(-\left(\frac{\sigma}{\sigma_0}\right)^m\right), \qquad (1)$$

where parameters σ_0 and m characterize the strength value and the width of its distribution. An experimental dependence in coordinates $\ln\big(-\ln(1-P)\big) - \ln\sigma$ is plotted to determine these parameters. It follows from (1) that this dependence is a straight line with slope m, and the second parameter allows one to calculate σ_0. Figure 2 shows such dependences for *p*-type samples grown by the Czochralski technique and epitaxial silicon samples that were subjected to CMP. The lines representing experimental data have

Table 2. Characteristics of surface roughness (nm) of silicon wafers (Czochralski-grown) measured along a base line

Roughness parameter	Diamond polishing ASM 5/3	CMP
R_a	2.41	0.26
$R_{\max} - R_{\min}$	13.42	1.61
R_z	9.91	1.54
R_q	3.11	0.35

significantly different slopes ($m = 1.41$ and 2.28) and are shifted along the stress axis ($\sigma_0 = 0.76$ and 1.11 GPa, respectively). This suggests that the corresponding strength values are different and the distribution widths differ greatly. The low m value is evidently an effect of the smallness of samples, which may yield major strength variations under a slight variation of loading parameters.

The surface irregularity height was measured along a base line to establish the relation between the strength of samples and surface profile parameters. Surface profilometry was performed using an AlphaStep D120 stylus profiler produced by KLA-Tencor Corp. Figure 3 presents the obtained data for samples after mechanical polishing with diamond powder and after CMP. The surface roughness was characterized by the mean absolute roughness height value (R_a), the difference between the maximum and minimum height values ($R = R_{\max} - R_{\min}$), the ten-point (five maximum and five minimum) height of irregularities (R_z), and root-mean-square deviations of the measured height values (R_q). It can be seen from Table 2 that the height of surface irregularities and its spread decrease considerably after CMP. This has a marked positive effect on strength.

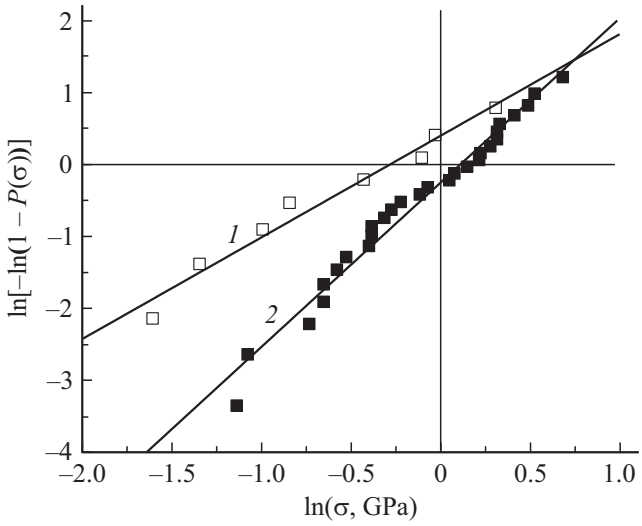

Figure 2. Weibull distributions for the strength of epitaxial (*1*) and Czochralski-grown (*2*) silicon after finishing CMP.

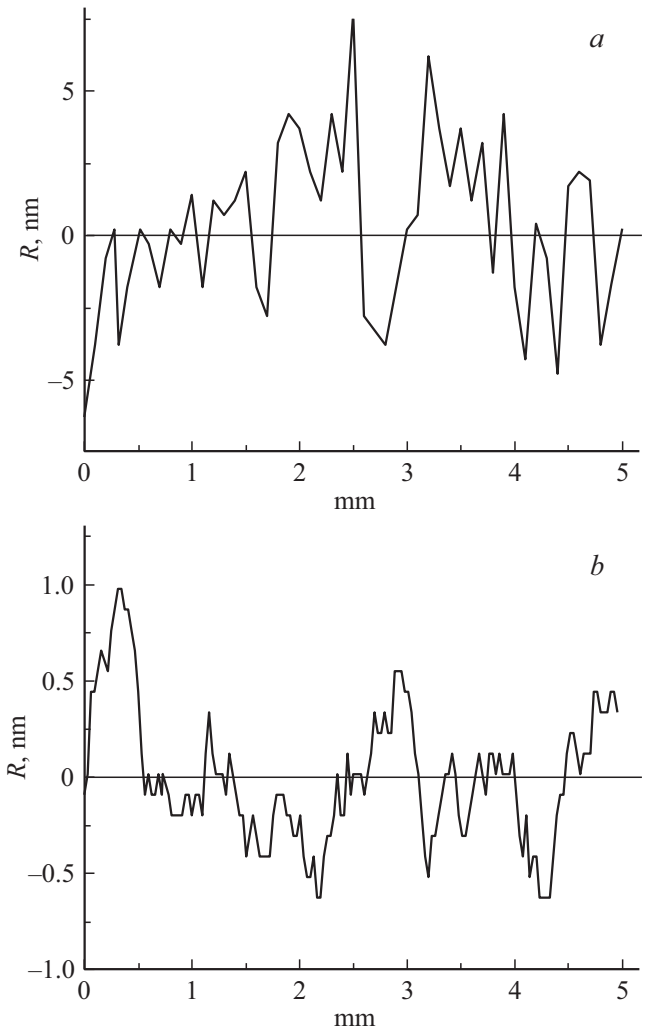

Figure 3. Height of surface irregularities measured along a line for silicon wafers after diamond polishing (*a*) and after CMP (*b*).

The methods and modes of mechanical and chemical-mechanical treatment in the process of thinning exert the primary influence on the strength of thin and ultrathin wafers. The arrangement of optimum conditions of finishing chemical-mechanical polishing, which enable the fabrication of smooth surfaces with minimum roughness values, is crucial for achieving the maximum possible strength. Such conditions may be established in two-stage CMP with „finishing smoothing" that mitigates the mechanical aspect of material removal. Finishing polishing with a weak mechanical impact and a marked chemical aspect of the material removal process smoothes out surface irregularities and leads to a considerable enhancement of strength.

Conflict of interest

The authors declare that they have no conflict of interest.

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