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Influence of Temperature from 20 to 100 °C on Specific Surface Energy and Fracture Toughness of Silicon Wafers

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Abstract

The influence of temperature in the range from 20 to 100 °C on the specific surface energy and fracture toughness of standard silicon wafers of three orientations (100), (110) and (111) was studied. Silicon wafers were heated on a special thermal platform with an autonomous heating controller, which was installed under the samples. At each temperature, the samples were kept for 10 min. The specific surface energy γ after exposure to temperature was determined by atomic force microscopy (AFM). Fracture toughness during and after exposure to temperature was determined by indentation followed by visualization of the deformation region using AFM. It has been established that the specific surface energy γ of Si wafers with orientation (100) and (111) increases with increasing temperature from 20 to 100 °C, and for orientation (110) it increases at temperatures from 20 to 80 °C, and then decreases. The diagonal length d of indentation marks, performed both during the heating process and after heating, decreases by increasing the temperature from 20 to 100 °C. The crack length c decreases on silicon wafers during indentation during heating from 20 to 100 °C, and after exposure to temperature, the length increases. When the plates are exposed to temperature, the fracture toughness K_{IC} increases with increasing temperature: for orientation (100) – up to 1.61±0.08 MPa·m$^{1/2}$, for (110) – up to 1.60±0.08 MPa·m$^{1/2}$ and for (111) – up to 1.66±0.04 MPa·m$^{1/2}$. A direct correlation was established between K_{IC}, measured during exposure to temperature, and an inverse correlation between K_{IC} measured after exposure to temperature and specific surface energy for the (100) and (111) orientations. An inverse correlation was obtained by K_{IC} at the (110) orientation when exposed to temperatures of 20–40 and 80–100 °C, and after exposure, a direct correlation was obtained. At 60 °C there is no correlation. The results obtained can be used to improve the mechanical properties of silicon wafers used in solar cells and microelectromechanical systems (operating at temperatures up to 100 °C).

Keywords: silicon wafers, temperature, fracture toughness, indentation method, atomic force microscopy

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Влияние температуры от 20 до 100 °С на удельную поверхностную энергию и вязкость разрушения пластин кремния

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Проведены исследования влияния температуры в диапазоне от 20 до 100 °С на удельную поверхностную энергию и вязкость разрушения стандартных пластин кремния трёх ориентаций (100), (110) и (111). Пластины кремния нагревали на специальной термоплатформе с автономным контроллером нагрева, которую устанавливали под образцы. При каждой температуре образцы выдерживали в течении 10 мин. Удельная поверхностная энергия γ после воздействия температуры определялась методом атомно-силовой микроскопии (АСМ). Вязкость разрушения во время и после воздействия температуры определялась методом индентирования с последующей визуализацией области деформации методом АСМ. Установлено, что удельная поверхностная энергия γ пластин кремния ориентации (100) и (111) увеличивается с увеличением температуры от 20 до 100 °С, у ориентации (110) – увеличивается при температурах от 20 до 80 °С, а затем снижается. Длина диагонали d отпечатков индентирования, выполняемых как в процессе нагрева, так и после нагрева, уменьшается с увеличением температуры от 20 до 100 °С. Длина трещин c уменьшается на пластинах кремния при индентировании во время нагрева от 20 до 100 °С, а после воздействия температуры длина увеличивается. Во время воздействия температуры на пластины вязкость разрушения K_{IC} увеличивается с увеличением температуры: для ориентации (100) – 1,61±0,08 МПа·м$^{1/2}$, для (110) – до 1,60±0,08 МПа·м$^{1/2}$ и для (111) – до 1,66±0,04 МПа·м$^{1/2}$. Установлена прямая корреляция K_{IC}, измеренной во время воздействия температуры, и обратная корреляция K_{IC}, измеренной после воздействия температуры, с удельной поверхностной энергией для ориентаций (100) и (111). Обратная корреляция K_{IC} с γ получена на ориентации (110) при воздействии температур 20–40 и 80–100 °С, а после воздействия – прямая корреляция. При 60 °С корреляции нет. Полученные результаты могут быть использованы для улучшения механических свойств кремниевых пластин, используемых в солнечных элементах и микроэлектромеханических системах (работающих при температурах до 100 °С).

Ключевые слова: пластины кремния, температура, вязкость разрушения, метод индентирования, атомно-силовая микроскопия

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Introduction

Recently, there has been great interest in the thermomechanical properties of silicon wafers [1] due to their application in the production of solar cells and MEMS operating at elevated temperatures [2]. Under such operating conditions, the formation of cracks, dislocations, and deterioration of the characteristics of semiconductor devices can occur [3]. Elastic-plastic transitions occur when exposed to temperature in silicon [2, 4, 5]. In this case, a change occurs from low-energy brittle fracture to high-energy plastic fracture, which leads to a direct dependence of the mechanical properties of single-crystal silicon on temperature changes [2, 4, 5].

One of the important mechanical properties characterizing the strength and resistance to cracking in a material is the fracture toughness K_{IC} [6]. The fracture toughness of single-crystalline silicon, depending on the orientation, can vary significantly [7–10] – from 0.60 to 3.30 MPa·m$^{1/2}$. In addition to orientation, the influence of temperature on the K_{IC} of single-crystal silicon was established in [5, 8]. Thus, in [5], a change in temperature from 25 to 300 °C leads to an increase in the fracture toughness of (001)-oriented silicon from 0.67 to 3.29 MPa·m$^{1/2}$. In addition to orientation and temperature, fracture toughness depends on the specific surface energy of the material [11, 12].

The purpose of the work was to study the effect of temperature from 20 to 100 °C on the fracture toughness and specific surface energy of silicon wafers with (100), (110) and (111) orientations, as well as to establish a correlation between fracture toughness and specific surface energy.

Materials and methods of research

Standard single-crystalline silicon wafers with (100), (110), and (111) orientations were used. The plates were manufactured at the "Kamerton" (branch of INTEGRAL OJSC, Belarus). Plate dimensions: Ø100 mm and thickness 0.5 mm.

Silicon wafers were heated on a special thermal platform with an autonomous heating controller (Microtest Machines ODO, Belarus), which was installed under the samples. The temperatures for the study were as follows: 20, 40, 60, 80 and 100 °C. At each temperature, the samples were kept for 10 min. During exposure to each temperature, indentation was performed using a PMT-3M microhardness tester (LOMO, Russia). A Vickers type tip (GOST 9377-81) was used as an indenter. The load on the indenter was 0.5 N. Five indentations were made on each sample. Indentation was also additionally carried out at the same load after the samples had completely cooled.

Studies of the silicon wafers surface, the indentation imprints morphology, determination of roughness and adhesion force were carried out on a Dimension FastScan atomic force microscope (Bruker, USA) in PeakForce QNM mode using standard silicon cantilevers MPP-12120-10 (Bruker, USA) with a cantilever stiffness of 5.7 N/m. Surface roughness was determined according to GOST R 8.700-2010[1]. After visualizing the indentation imprints, the diagonal length of the indentations d and the cracks length from the center of the indentation c were quantified.

Fracture toughness K_{IC} was determined using the formulas given in [13]. The choice of formula depends on the value of c/a (crack length to the length of the indent semi-diagonal) and the type of cracks formed (median or Palmquist) [14, 15].

The specific surface energy (adhesion work) was determined according to the formula [16]:

$$\gamma = \frac{F_{ad}}{2\pi R}, \quad (1)$$

where F_{ad} is the force of adhesive interaction between the tip of the AFM probe and the surface, N; R is the radius of the probe tip curvature, m.

The radius of the probe tip curvature R was estimated using the reference sample RS-12M – a polycrystalline titanium roughness sample.

Specific surface energy γ was determined on all plates before and after exposure to temperatures in the range from 20 to 100 °C by AFM on several fields of size: 1×1, 3×3, 5×5, 10×10, 20×20 and 30×30 μm^2. First, the adhesion force was determined, and then the specific surface energy was calculated using Formula (1).

Results and discussion

The roughness (Figure 1) of the silicon wafers surface varies in the range: from 0.3 to 2.5 nm for the

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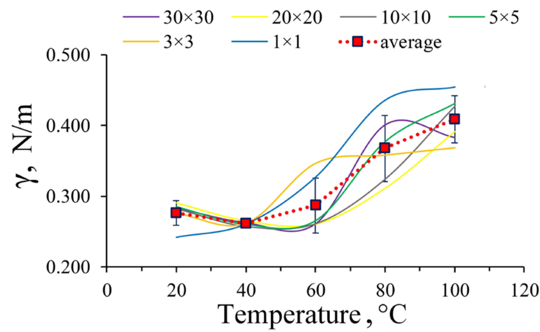
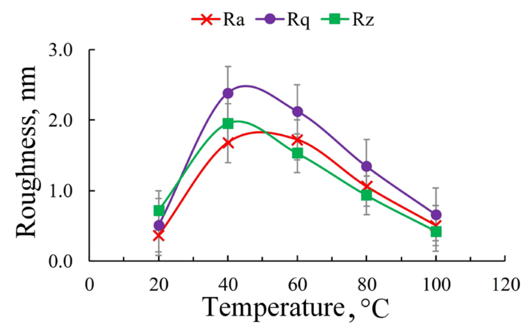
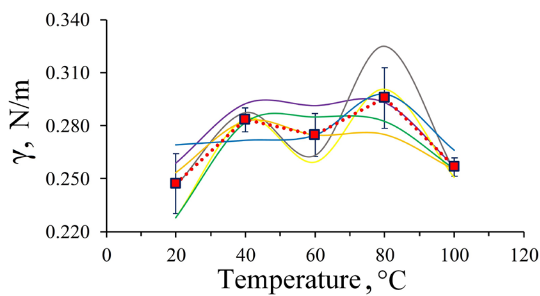
(100) orientation, from 0.3 to 1.6 nm for the (110) orientation, and from 0.3 to 3.6 nm for the (111) orientation. The specific surface energy γ of the surface of the (100) and (111) orientation plates increases with increasing temperature from 20 to 100 °C (Figure 1*a*, *c*). In this case, γ for the (100) orientation varies in the range from 0.276±0.018 to 0.409±0.033 N/m (Figure 1*a*), and for the (111) orientation – from 0.254±0.016 to 0.273±0.007 N/m (Figure 1*c*). For the (110) orientation, γ of the surface has a maximum at a temperature of 80 °C and is 0.296±0.017 N/m, and at temperatures of 20 and 100 °C γ is almost the same and is 0.247±0.017 N/m and 0.257±0.005 N/m, respectively (Figure 1*b*).



a



d



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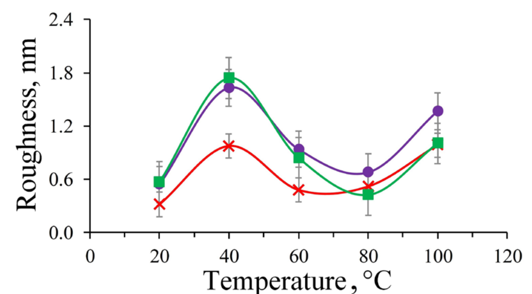

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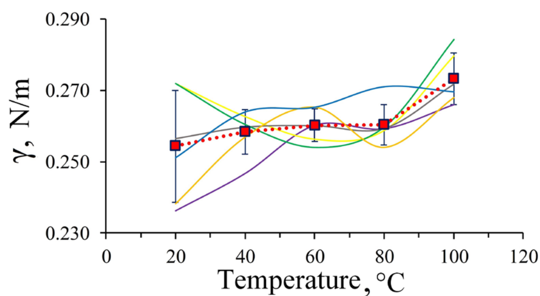
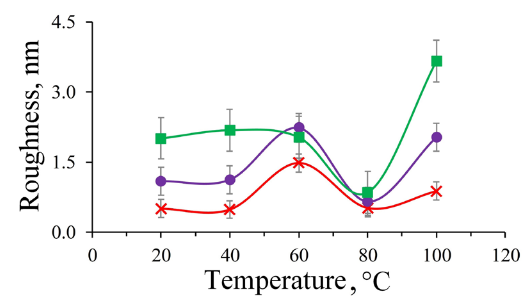


c



f

Figure 1 – Dependence of specific surface energy (*a*–*c*) and surface roughness (*d*–*f*) of silicon wafers before and after exposure to temperature in the range from 20 to 100 °C: *a*, *d* – Si wafer with (100) orientation; *b*, *e* – Si plate with (110) orientation; *c*, *f* – Si plate orientation (111)

After visualization and quantitative determination of the indentation parameters (diagonals length and cracks length), it was found that with an increase in temperature from 20 to 100 °C, the length of the indentation imprints diagonal, performed both during the heating process and after heating, decreases (Figure 2*a*, *d*). During heating, the values of the diagonal length d decrease from 10.21–10.57 μm to 9.85–10.07 μm when the temperature changes from 20 to 40 °C. Further, the values practically do not change and are in the range of 9.96–10.04 μm (Figure 2*a*).

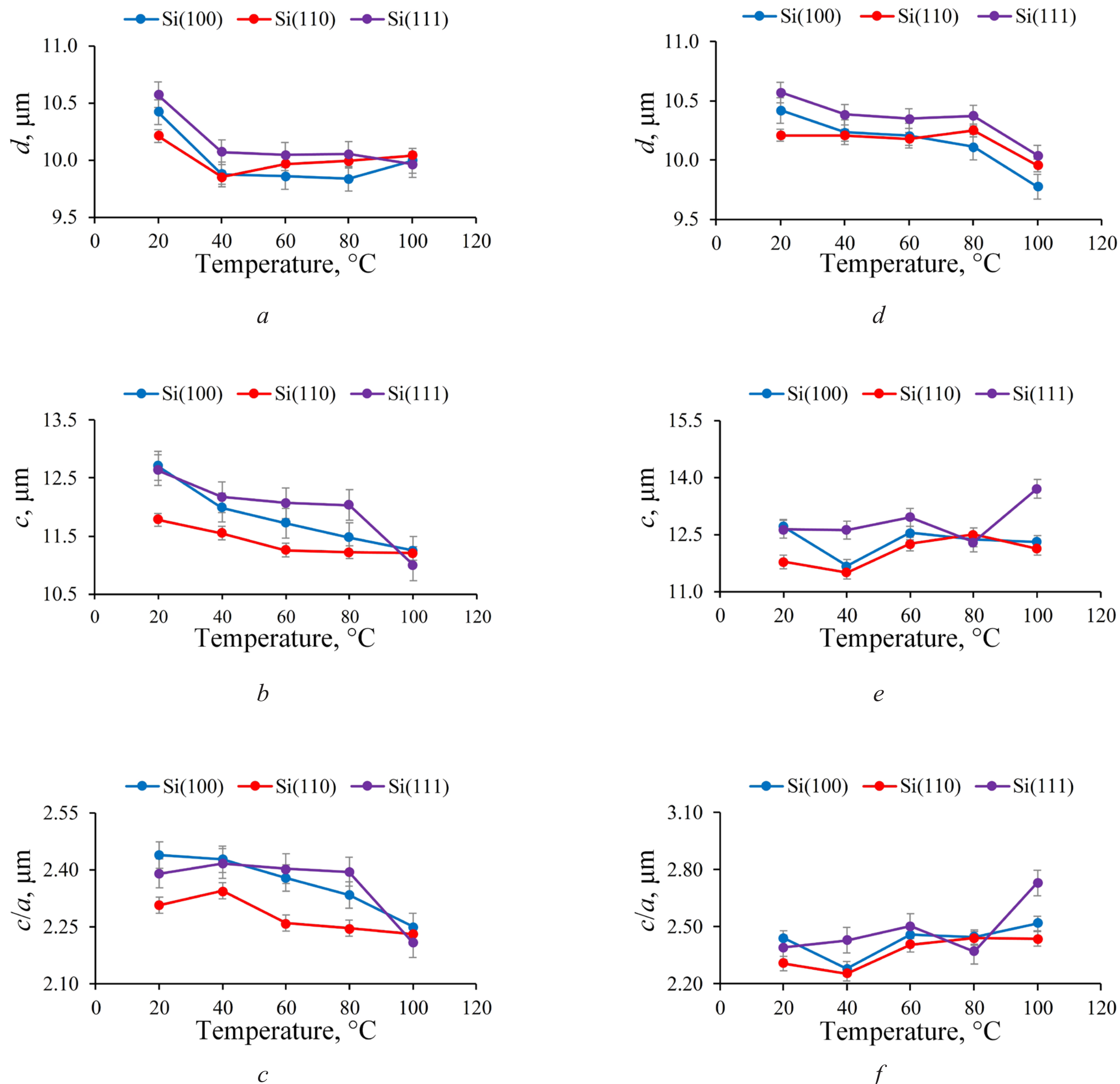


Figure 2 – Dependences of the diagonal length (*a*, *b*), crack length from the center of the indentation imprint (*b*, *e*) and the c/a ratio (*c*, *f*) during (*a*, *b*, *c*) and after (*d*, *e*, *f*) exposure to temperature

The diagonal of the indentation imprints after heating decreases from 10.21–10.57 μm to 10.21–10.38 μm when the temperature changes from 20 to 40 °C (Figure 2*d*). In the temperature range of 40–80 °C it remains practically unchanged, and at 100 °C it decreases to 9.77–10.04 μm (Figure 2*d*).

The crack length c decreases on silicon wafers upon indentation during heating from 20 to 100 °C (Figure 2*b*). The c values for all silicon orientations at 20 °C are in the range of 11.78–12.71 μm (Figure 2*b*). Exposure to temperature causes the range to decrease to 11.00–11.25 μm. The length c on silicon wafers of orientation (110) and (111) after heating increases at 100 °C to 12.13 and 13.71 μm (Figure 2*e*), and on the (100) wafer it decreases to 11.25 μm compared to the value of 12.71 μm at 20 °C.

The *c*/*a* ratio for all samples is above 2 (Figure 2*c*, *f*). For samples that were indented during temperature exposure, *c*/*a* is in the range of 2.30–2.45 at 20 °C and decreases to 2.20–2.26 at 100 °C (Figure 2*c*). The *c*/*a* ratio for samples after exposure to temperature increases from 2.30–2.45 at 20 °C to 2.40–2.75 at 100 °C (Figure 2*d*).

The fracture toughness K_{IC} increases with increasing temperature from 20 to 100 °C while the plates are exposed to temperature (Figure 3*a*). For the (100) orientation plate, K_{IC} changes from 1.46±0.07 MPa·m$^{1/2}$ to 1.61±0.08 MPa·m$^{1/2}$, for

(110) – from 1.53±0.05 MPa·m$^{1/2}$ to 1.60±0.08 MPa·m$^{1/2}$ and for (111) – from 1.52±0.03 MPa·m$^{1/2}$ to 1.66±0.04 MPa·m$^{1/2}$ (Figure 3*a*). After exposure to temperature, fracture toughness decreases with increasing temperature (Figure 3*b*): for the (100) orientation plate from 1.46±0.07 MPa·m$^{1/2}$ to 1.34±0.03 MPa·m$^{1/2}$, for (110) – from 1.53±0.05 MPa·m$^{1/2}$ to 1.39±0.05 MPa·m$^{1/2}$ and for (111) – from 1.52±0.03 MPa·m$^{1/2}$ to 1.22±0.04 MPa·m$^{1/2}$.

The K_{IC} largely depends on the crack length [15, 16]. It should also be noted that the K_{IC} value is influenced by the energy of crack propagation, which in turn is related to the specific surface energy of the material [11, 12].

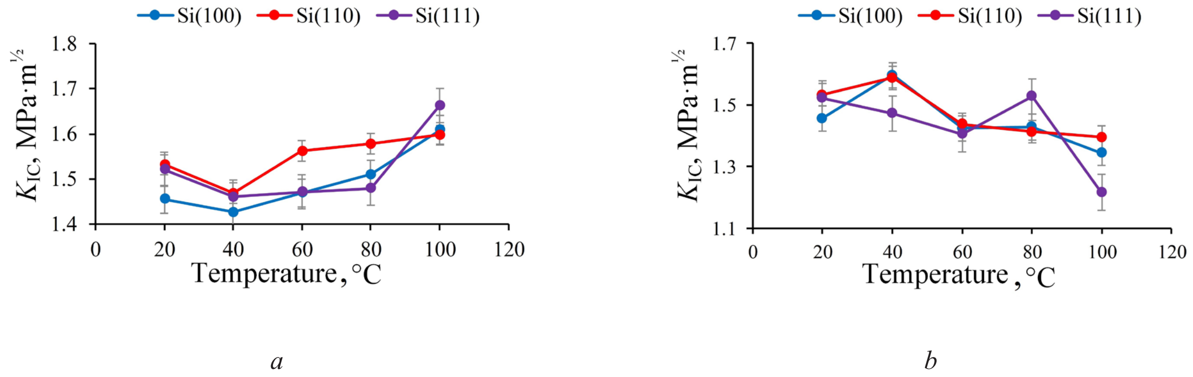


Figure 3 – Dependences of K_{IC} during (*a*) and after (*b*) exposure to temperature

Table

Correlation coefficients between fracture toughness K_{IC} and specific surface energy γ

	K_{IC}					
	(100)	(110)	(111)	(100)	(110)	(111)
	during exposure to temperature			after exposure to temperature		
γ	1.0	-0.1	0.8	-0.8	-0.1	-0.9

The correlation coefficients Ccorr between fracture toughness and specific surface energy of silicon wafers with temperature changes are given in Table. A direct correlation was established (Table) K_{IC}, measured during exposure to temperature, and specific surface energy for wafers (100) and (111) – C_{corr}, respectively are equal to 1.0 and 0.8.

The fracture toughness K_{IC} measured after temperature exposure is inversely correlated with the specific surface energy of the (100) and (111) orientation plates. If we determine C_{corr} over the entire temperature range from 20 to 100 °C for a (110) orientation plate, then there is practically no correlation between K_{IC} (both during and after exposure to temperature) and γ (Table). However, if we consider the ranges before and after 60 °C, then C_{corr} is equal to -1.0 (when exposed to temperature) and C_{corr} = 1.0 (after exposure).

The presence of a difference in the values of specific surface energy and K_{IC} at a temperature of 60 °C may be associated with the appearance of an elastic-plastic transition, the appearance of dislocations in the lattice instead of cracks [17].

Conclusion

The specific surface energy and fracture toughness of silicon wafers of three orientations (100), (110) and (111) were studied during and after exposure to temperatures in the range from 20 to 100 °C using atomic force microscopy and the indentation method.

Determined that the specific surface energy γ of the surface of the (100) and (111) orientation plates increases with increasing temperature from 20 to 100 °C. The diagonal length of the indentation marks (performed both during the heating process and after heating) decreases by increasing the temperature from 20 to 100 °C. The length of cracks on silicon wafers decreases during indentation during heating

from 20 to 100 °C, and after cooling, the length increases.

The fracture toughness K_{IC} behaves similarly. When the plates are exposed to temperature, the fracture toughness K_{IC} increases with increasing temperature: for orientation (100) – 1.61±0.08 MPa·m$^{1/2}$, for (110) – up to 1.60±0.08 MPa·m$^{1/2}$ and for (111) – up to 1.66±0.04 MPa·m$^{1/2}$. A direct correlation was established between K_{IC}, measured during exposure to temperature, and the specific surface energy for the (100) and (111) orientations. K_{IC} measured after temperature exposure is inversely correlated with the specific surface energy of the (100) and (111) orientations. An inverse correlation was obtained by K_{IC} at the (110) orientation when exposed to temperatures of 20–40 and 80–100 °C, and after exposure, a direct correlation was obtained. At 60 °C there is no correlation. The results obtained can be used to improve the mechanical properties of silicon wafers used in solar cells and microelectromechanical systems (operating at temperatures up to 100 °C).

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