Elastic moduli of alumina nanoceramics

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Abstract. In this study we investigated the physico-mechanical properties of alumina nanoceramics, that is used as substrates in micro- and optoelectronics chips. The elastic characteristics (Young's modulus E, shear modulus G, Poisson's ratio ν, microhardness H) are determined by measuring the longitudinal and transverse wave velocities. The results suggest that the nanoceramics have unique properties that can be used in the future microelectronics to improve the stability of chips and to optimize the production technology.

1. Introduction
Currently, nanostructured materials that have special electrical, optical and mechanical properties represent considerable interest for micro- and optoelectronics. Nanoceramics studied in this work is used in microelectronics for chip substrates. For the stable operation of the device, the electrical properties must be combined with other physical and mechanical features, which can be obtained by measuring the sound velocity. In this work, we determined the elastic characteristics (Young's modulus E, shear modulus G, Poisson's ratio ν, microhardness H) of nanoceramics based on Al₂O₃.

2. Samples
In this study we investigated three samples of nanoceramics with alumina nanostructured phase received from plant of electronic instrumentation "Elion" (Zelenograd, Russia). For certification of the samples, the scanning electron microscopy and X-ray analysis were carried out. Investigation of the structure of the surface of the samples was performed by scanning electron microscope SIGMA VP (Carl Zeiss) production using secondary electron detector (In-lens) in high vacuum at an accelerating voltage of 5 keV. Pictures with a scanning electron microscope were obtained at different scales at several points samples cut. Images of the sample 2 and the sample 3 obtained with a scanning electron microscope are shown in Figures 1 and 2.

The particle size was determined by all the images using the analyzer of solid bodies microstructure fragments Clinker C7 by SIAMS company and the particle size of sample 1 was 200-400 nm, sample 2 had particle size of 1-3 micron, while the particle size of the sample 3 was 80-200 nm.
Another method to determine the particle size and phase composition performed was the X-ray analysis carried out on a diffractometer XPERT PRO in the $K_{\alpha}$ copper radiation. The qualitative composition and size of coherent scattering regions (CSR) for samples was determined from the broadening of X-ray diffraction reflections. By the decoding results it was revealed that the main phase for all the samples was $\alpha$-Al$_2$O$_3$ (corundum), and the average size of coherent scattering region for the sample 1 and sample 2 was more than 200, and CSR of the sample 3 was 100-150 nm.

The density of the samples was measured by the helium Pycnometer AccuPyc II 1340 with high accuracy: $\rho_1 = 3.7462 \pm 0.0006$ g/cm$^3$, $\rho_2 = 3.7563 \pm 0.0008$ g/cm$^3$, $\rho_3 = 3.7616 \pm 0.0006$ g/cm$^3$.

3. Methods

Determination of the propagation velocity of the longitudinal waves was performed on the device consisting of two piezoelectric transducers (5 MHz) and a sample between them. One of transducers was used as the signal source and the other was the receiver. The signal received by the second transducer was registered by a personal computer. Using a special program written in LabView, the value of the longitudinal ultrasonic velocity in the sample was calculated.

The size of nanoceramics sample was quite small, that is why a scheme was developed that allows single point measurements, in which the transverse wave reflected perpendicularly from the sample face in order to determine the velocity of shear waves. We used a specially shaped prism that conducts transverse waves. Aluminum was chosen as the material for the prism because its acoustic impedance is close to the acoustic impedance of the ceramic. The scheme of shear velocity measurements is shown in Figure 3.

The longitudinal ultrasonic wave $l$ emitted in angle beam transducer $I$. On the border of the aluminum prism 2 it is transformed into a transverse wave $t$, which penetrates between the first $\alpha'$ and second critical angles $\alpha''$. The shape of the aluminum prism 2 was calculated so that the transverse wave propagating through the prism goes through a thin contact layer 3 and the beam is perpendicular to the sample 4. The silicate glue is used as the contact layer 3. The signal from the pulse is received by ultrasonic measurement system. To determine the transverse velocity it is required to know the thickness of the sample and the time at which the ultrasonic wave has passed through it. Time is determined by the difference between the arrival times of the two pulses. The first impulse is a reflection from the near face of the sample. The second pulse is a reflection of the far face of the sample. The speed of transverse waves in this method is calculated as follows:

$$C_t = \frac{2D}{t},$$

(1)
where $D$ is specimen thickness, $t$ is time between two pulses.

Measurements were performed using ultrasonic flaw detector PCUS-10 in pulse mode and the inclined transducer with 65° angle and 5 MHz frequency.

Figure 3. The scheme of shear velocity measuring is. Where 1 is ultrasonic flaw detector PCUS-10, 2 – tilted transducer, 3 – aluminum prism, 4 – contact layer, 5 – the sample, 6 – computer, $l$ – longitudinal wave, $t$ – shear wave.

4. Results

The results are shown in Table 1. Values of the elastic characteristics were calculated according to well known formulae (2)-(5) [1-5]:

- shear modulus:
  \[ G = \rho \cdot C_t^2, \quad (2) \]

- Poisson's ratio:
  \[ \nu = \frac{(C_l^2 - 2 \cdot C_t^2)}{2 \cdot (C_l^2 - C_t^2)}, \quad (3) \]

- Young's modulus:
  \[ E = \rho \cdot C_t^2 \cdot \frac{3 - 4 \cdot \frac{C_l^2}{C_t^2}}{\left(1 - \frac{C_l^2}{C_t^2}\right)^2}, \quad (4) \]

- microhardness:
  \[ H = \frac{(1 - 2\nu)E}{6(1 + \nu)}, \quad (5) \]

where $\rho$ is density of the sample, kg / m$^3$, $C_l$ and $C_t$ is velocity of longitudinal $l$ and shear waves $t$, m/s.
Table 1. The properties of nanoceramics.

| Sample 1 | Sample 2 | Sample 3 | Accuracy |
|----------|----------|----------|----------|
| Density $\rho$, kg/m$^3$ | 3746 | 3756 | 3762 | $\pm$ 0.8 |
| Longitudinal waves velocity $C_l$, km/s | 9951 | 9948 | 8728 | $\pm$ 3 |
| Shear waves velocity $C_s$, km/s | 4158 | 4030 | 4915 | $\pm$ 50 |
| Young's modulus $E$, GPa | 170 | 162 | 229 | $\pm$ 8 |
| Shear modulus $G$, GPa | 61 | 58 | 90 | $\pm$ 3 |
| Poisson's ratio $\nu$ | 0.39 | 0.4 | 0.28 | $\pm$ 0.01 |
| Microhardness $H$, GPa | 4.6 | 4 | 14 | $\pm$ 0.2 |

The values of the elastic parameters of the sample 1 and the sample 2 are equal within the experimental error. However, they are very different from those for the sample 3. The value of longitudinal wave velocity decreases with increasing samples density. Such dependence is anomalous, because the ultrasonic wave velocity usually higher for more dense samples [6]. In contrast, the dependence of shear wave velocity on the density is normal. The Young's and shear moduli, as well as the calculated microhardness of the sample 3 increase significantly, while the Poisson ratio decreases. This fact indicates that the sample is hard but more fragile than other samples. The data collection of X-ray diffraction and electron microscopy suggests that these unusual changes of the elastic properties may be associated with a size factor, i.e. with the presence of nanometer particles. Therefore, mechanical properties can be controlled by varying the particle size.

5. Conclusion
In this study we investigated the physico-mechanical properties of aluminum oxide nanoceramics. The findings suggest that the nanoceramics have unique properties that can be used in the future in microelectronics to improve the stability of chips and to optimize the production technology.

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