Strength analysis of small-sized thin ceramic discs obtained by spark-plasma sintering

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Abstract. The study develops an original method of mechanical test of thin small-size disks for determining resistance of brittle materials to rupture. Based on this method a test of ultimate flexure strength was conducted on Al₂O₃ ceramics and composites of β-Si₅AlON₇–BN, system obtained by the method of spark-plasma sintering. The obtained test results allowed determining the optimal process parameters for sintering this ceramic materials in terms of their strength level: composition and dispersion of the starting powder mixtures, the maximum sintering temperature, etc.

1. Introduction
Development of advanced technology of material consolidation using electromagnetic fields requires development of appropriate mechanical test methods for determination of the mechanical properties of the produced materials. This is also conditioned by the fact that in most cases the produced compacts have a very small size with diameter 10 mm and thickness 1 to 10 mm. Known standard test methods are not applicable to the samples so small. Consequently, the objectives of this work were to develop the methods for determining the strength of small-sized samples basing on the solution of the flexible small-size disk bending problem, and to test the applicability of the developed bending test method for ceramic disk samples obtained by spark-plasma sintering (SPS).

2. Method of determination of the bending strength of small-size disk brittle material
The scheme applied to determine the strength of the material by testing small-size thin discs placed on a ring bearing is presented in Figure. 1.

Figure 1. Flexure test of the disk supported along the contour: 1 – punch, 2 – cage, 3 – sample, 4 – bearing ring.
This test scheme was previously designed to determine the bending strength of brittle materials for larger samples (about 50mm to 60 mm in diameter and thickness of 3 mm to 6 mm) [1]. To substantiate its applicability to small-sized samples, the elastic bending disk problem has been solved, which was supported along the contour and loaded in the center with a distributed load. A scaling ratio was maintained between the geometric dimensions of large and small-size samples. A solution was obtained for circumferential and radial stresses in the central area of the disc, which coincides with the known, and the allowed relation between the thickness of the disk and the diameter of the bearing surface was defined. The calculation of the failure stress takes into account the maximum load withstood by the sample before the failure, with a linear diagram of load $P$ - deflection $\omega$ by the formula (1):

$$\sigma_0 = \sigma_r = \sigma_{\text{max}} = \frac{3P_{\text{max}}}{8\pi h^2} \left[ 4 - \left( 1 - \mu \left( \frac{d}{D} \right)^2 + 4(1 + \mu)\ln \frac{D}{d} \right) \right]$$

(1)

where $h$ – the thickness of the disk; $d$ and $D$ – diameters of the punch and the bearing respectively; $\mu$ – Poisson's ratio.

Conventional elastic modulus was determined by converting the elastic deflection of the disk center into the circumferential and radial deformations by the formula (2):

$$\varepsilon_\theta = \varepsilon_r = \frac{h}{2R}$$

(2)

where $R$ – curvature radius of the neutral plane of the disk as shown in Figure. 2.

![Figure 2. Calculation scheme of curvature radius of the neutral plane of the disk](image)

The radius $R$ is found by simple geometric proportions and with a sufficient accuracy by the formula (3):

$$R = \frac{D^2}{4\omega}$$

(3)

After the substitution of the formula (3) in (2), a formula for the relative deformations is obtained:

$$\varepsilon_\theta = \varepsilon_r = \frac{2h\omega}{D^2}$$

(4)

By Hooke's law, the deformations for the biaxial stress state are equal:

$$\varepsilon_\theta = \frac{1}{E} \left[ \sigma_\theta - \mu \sigma_r \right] = \frac{\sigma_{\text{max}}}{E} \left( 1 - \mu \right)$$

(5)
By equating the (4) and the (5), a formula for determining the conventional elastic modulus is obtained:

$$E = \frac{(1 - \mu)\sigma_{\text{max}}D^2}{2h\omega}$$

(6)

The conventionality of $E$ characteristic is due to a number of assumptions made in the derivation the formula. Conventional elastic modulus is essentially a characteristic of flexural rigidity of the disk.

3. Bending test results for small-size disks

The developed method is used to determine the strength characteristics of the ceramic composites of the $\beta$–Si$_5$AlON–BN system, obtained by SPS of the mixtures of corresponding powders. These composites consist of solid SiAlON matrix with high elastic modulus and inclusions of low-modulus particles of hexagonal boron nitride. This structure is able to provide an improved resistance to various influences by absorbing and dispersing the elastic energy released during the expansion of cracks.

Another important advantage of ceramic composites of this type is their good processability by traditional instruments. By varying the maximum temperature of isothermal holding during the SPS from 1550 °C to 1750 °C and the composition of the starting powder mixture, the samples were obtained with a relative density of 73% to 99% and h-BN content of 0 wt% to 30 wt%. The samples were disk-shaped with 10 mm diameter and thickness of 1mm to 1.3mm. During the flexure test the bearing ring had a diameter of 7.5 mm.

The performed tests showed that the failure these samples has a pronounced brittle nature with the formation of fission fragments. The key parameter for determining the level of the bending strength of the composite is its relative density (Fig. 3).

![Figure 3](image_url)

**Figure 3.** The dependence of bending strength of the composite $\beta$-Si$_5$AlON–BN on its relative density

It should be noted that determined level of strength characteristics of the studied samples corresponds to the strength characteristics and similar dependencies of ceramic composites with similar composition obtained by other methods[2-4].

It should also be noted that in all mentioned test the bending strength measurement was carried out by the conventional three-point bending method using a standard size samples. It was also determined that varying the h-BN content in the studied composites in the range of 0 wt% to 30 wt% has almost no effect on its strength characteristics (Fig. 4). Concurrently, a notable increase in the strength values
of 30% to 40% can be achieved by substituting 20 wt% to 40 wt% of SiAlON phase in the composite content by other refractory compounds with high values of elastic modulus (SiC and TiN).

Figure 4. Dependency of the bending strength of the composite \(\beta\)-Si\(_5\)Al\(_7\)ON\(_6\) on BN content \((\rho_{\text{rel}}=0,95-0,98)\).

The presented method was also used for conducting comparative mechanical tests of Al\(_2\)O\(_3\) ceramics obtained by SPS of two kinds of nanoscale spherical powder with an average particle size of 45 nm and 150 nm and specific surface area of 35.8 m\(^2\)/g and 10.1 m\(^2\)/g respectively. The heating rate during the SPS equaled 100 °C/min, the maximum temperature of the process amounted to 1400°C, the duration of holding at the maximum temperature equaled 10 min, the diameter of sintered sample equaled 10 mm, its thickness was about 1 mm to 1.3 mm. It was established that ceramics sintered from a larger size powder of Al\(_2\)O\(_3\) has a homogeneous fine-grained structure and a high density and strength characteristics (Table 1).

Table 1. Characteristics of Al\(_2\)O\(_3\) ceramics.

| Average grain size of the starting powder (nm) | Characteristics of the sintered ceramics |
|-----------------------------------------------|----------------------------------------|
| Average grain size (µm)                       | Relative density (%)                   | Ultimate flexural strength \(\sigma_f\), (MPa) | Elastic modulus \(E\), (GPa) | Microhardness \(H\), (GPa) | Structure |
| 45                                            | 1,10                                   | 93,7                                       | 141                          | 66                          | 12,9       | layered |
| 150                                           | 0,65                                   | 95,8                                       | 174                          | 73,7                        | 19,5       | homogeous|

Ceramics sintered from a finer powder of Al\(_2\)O\(_3\) had a pronounced layered structure with higher porosity and grain size (Fig. 5).
There are two factors negatively affecting the strength characteristics of the ceramics: large specific surface area of the starting powder, which promotes the sorption of various gases at room temperature, and the formation of large agglomerates of its fine particles. All this complicates the removal of pore in the process of sintering and leads to the formation of structural inhomogeneities, while also leads to an increase of grain size.

4. Conclusions
The research analyzed the application possibility of the developed method for determining the bending strength of ceramic materials obtained by SPS with mechanical test of small-size thin disks placed on a ring bearing.

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