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Tensile strength of materials obtained by electric pulse consolidation of powders

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Abstract. The article investigates the possibility of determining the resistance to fracture of materials obtained from powders by electric pulse consolidation method using the test method for bending thin disks on an annular support and testing short cylinders according to the "Brazilian test". Techniques are verified by testing small-sized thin disks and short cylinders made of gray cast iron and graphite. It is revealed a difference in the nature of the destruction of cast iron samples (plastic failure) and samples of graphite (brittle fracture). The good correspondence of the characters of destruction of graphite samples and samples produced from powders by the electric pulse consolidation is shown. The influence of various additives and manufacturing technology of materials on the resistance of the material to brittle fracture is revealed. The optimal modes of high voltage consolidation a heavy alloy based on the results of tests of short cylinders according to the "Brazilian test" scheme were obtained. The effect of the thickness of the cylinders on the strength of aluminum oxide Al_2O_3 is investigated.

1. Introduction
Development of production technology of modern materials by consolidation of powders, welding and sintering regimes [1, 2] is carried out, as a rule, on small samples, the size of which often does not exceed 10–15 mm in diameter, and the thickness can vary from 1 to 10 mm. Standard mechanical test methods are often not applicable to such samples. Alternative test methods have to be used to evaluate the mechanical properties of the material. Examples of such methods can be the bending of thin discs on the annular support and the compression of the disc in the plane of symmetry according to the "Brazilian test" scheme. These methods are commonly used to test brittle materials.

The aim of this work is to substantiate the possibility of their application to assess the tensile strength of materials obtained by methods of electro-pulse consolidation of powders, which are characterized by a low-plastic state.

2. Method of testing of thin disks for bending on the ring support
In these studies, the method of loading the disk on the ring support, presented in [3], was used (figure 1).
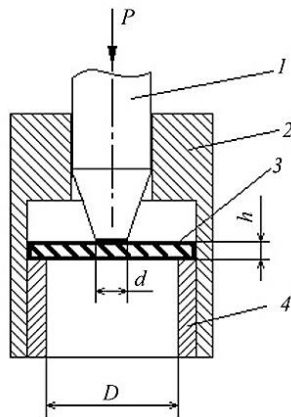
Figure 1. The scheme of testing the disk on the annular support for bending with loading flat indenter: *1* indenter, *2* clip, *3* sample, *4* support ring.

The breaking stress and the resistance of the material to rupture are calculated taking into account the maximum load P_{max} withstood by the sample to failure, with the linear diagram "load P–deflection ω" according to the formula:

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

In equation (1) h is the thickness of the disk; d and D are the diameters of the punch and support, respectively; μ is the Poisson's ratio.

Analysis of the stress-strain state of the model, performed using the verified [4, 5] calculation complex ANSYS Mechanical version 16.2, showed the presence of a three-axis stress state in the contact interaction of the indenter and the disk. On the reverse side of the disk, as expected, there is a flat stress state with maximum circumferential tensile stresses, which cause brittle fracture of the sample. Figure 2 shows an almost homogeneous region of maximum tensile stresses under the indenter.
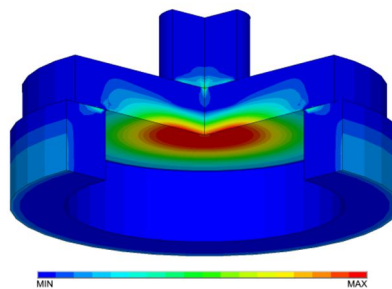

Figure 2. Qualitative picture of the distribution of the first main stresses in the disk sample under the indenter during bending.

The origin of the fracture is possible under the action of maximum shear stresses in the contact loading zone. Verification of methods for small samples was carried out by testing the bending of discs made of gray cast iron and graphite. Mechanical properties of materials are given in table 1.

The typical diagrams of static bending of cast iron and graphite samples MPG-6 on the Instron test machine, which indicate the absence of brittle fracture of cast iron samples and brittle fracture of graphite, and the nature of their destruction were presented in [6]. The results of the test of cast iron disks with a diameter of 10–15 mm and a thickness of 1–1.5 mm showed that the level of stresses calculated by equation (1) is in good agreement with the tensile strength of cast iron [6].

Table 1. The values of ultimate strength in tension/compression and Young's modulus of the materials studied.

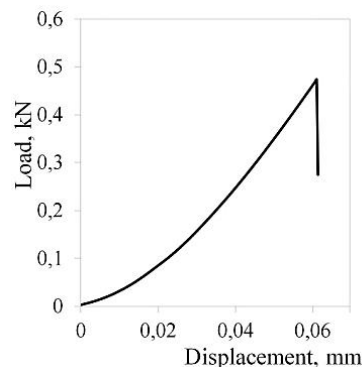| Material	σ_u^t, MPa	σ_u^c, MPa	E, GPa
Cast iron SCH 10-40	104	404	100
Graphite MPG-6	25	98	10
Graphite ARV-1	13.8	37	11

A completely different picture is observed in the destruction of disks of graphite MPG-6 in the same range of thicknesses. With almost linear machine diagram of static bending up to destruction, there is a brittle dynamic destruction of graphite into numerous parts. The level of destructive stresses calculated by equation (1) is about 20% lower than the tensile strength of the material.

3. Testing of thin disks obtained by spark-plasma sintering
The above method of bending thin disks on the ring support was used to assess the brittle strength of a number of materials obtained by spark-plasma sintering (SPS) of powder materials.

A comparative study of the compacts obtained by the SPS method from nano and ultrafine (UF) aluminum oxide powders with a spherical particle shape [7] showed a higher density, strength, microhardness and homogeneity of the structure of the compacts from the UF powder than the compacts from the nanopowder. In particular, the fracture resistance of UF-powder disks was higher than that of nanopowder, 174 and 141 MPa, respectively. The use of pre-pulsed magnetic pressing of UF-powder increased the strength of the samples to 202 MPa.

For the SiAlON material obtained by the SPS method from powder synthesized in the process of self-propagating high-temperature synthesis (SHS), the influence of various additives and the thickness of the disks on the bending strength were investigated. Discs with a diameter of 10 mm ($D = 7.5$ mm, $d = 3.75$ mm) and 15 mm ($D = 11.5$ mm, $d = 3.75$ mm) were tested. Figure 3 shows a characteristic diagram of the bending disk of SiAlON. It is almost identical to the graphite bending diagram.

Figure 3. Engine diagram of a bending disk of SiAlON with a diameter of 15 mm.

The destruction of all samples was brittle with the separation of many small fragments. The effect of the sample thickness and various additives on the SiAlON strength is presented in the work [8].

4. Calculation analysis of short cylinder compression according to the "Brazilian test" scheme
The test discs according to the "Brazilian test" scheme [9] is used to determine the tensile strength of rocks when testing samples with a diameter of at least 50 mm with a thickness-to-diameter ratio in the range of 0.2÷0.75. The purpose of this work in relation to this technique was to justify the possibility of its application to assess the tensile strength of materials obtained by the methods of electro-pulsed consolidation of powders.

Simulation of the disk sample loading process was performed in the verified [4, 5] calculation complex ANSYS Mechanical version 16.2. The full calculation model is shown in figure 4.

ASTM D3967-95a standard offers to determine the strength of the tested material by the formula:

$$\sigma_t = \frac{2P}{\pi t D}.$$

(2)

In equation (2) P is the maximum load sustained by the specimen, t is the thickness of the specimen, and D is its diameter.

The results of the analysis with the study of the influence of the ratio of the cylinder thickness to its diameter on the maximum tensile stresses are presented in [10]. It has been shown that the σ_t parameter determined by the ASTM D3967-95a method is a good estimate of the first principal stress averaged along the x-axis of the disk. Changing the ratio of the disc thickness to its diameter in the range of 0.3–1 has little effect on the maximum tensile stress. It has also been found that the maximum principal stresses at individual points can exceed the average stresses σ_t by 2.5 times, which should affect the applicability of equation (2) for concentration sensitive brittle materials.
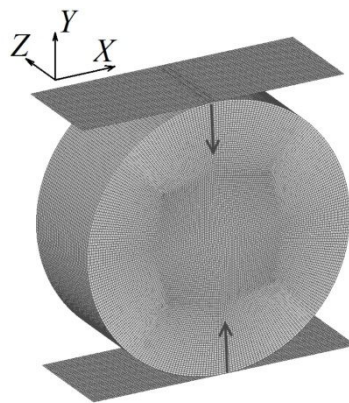

Figure 4. Full geometry of the computational model.

5. Results of experimental study of short cylinders made of model materials
Short cylinders made of gray cast iron and ARV-1 graphite, the characteristics of which are given in table 1, were subjected to experimental study according to the scheme of the "Brazilian test".

5.1. Test cylinders of gray cast iron
The results of testing of small-size discs made of cast iron given in [9] confirmed the possibility of using the calculated equation (2) to determine the tensile strength of the material. Discs made of grey cast iron were about the size of $D \times t$: 10 × 4 and 15 × 4 mm. The diagram of compression of the cast iron disc is presented in figure 5a.

Initial cracks in the cast iron sample are formed in the contact area where the maximum shear stresses are applied. The beginning of crack growth is associated with the maximum load on the sample. Smooth load reduction after the maximum-slow crack propagation is the departure of one half of the sample from the other. No explosive fracture characteristic of a brittle rupture from normal stresses was observed.

5.2. Test cylinders of graphite
Graphite disks were of three sizes $D \times t$: 8 × 4, 8 × 8 and 8 × 12 mm. The characteristic compression diagram of a graphite disk 8 × 8 mm in the plane of symmetry is presented in figure 5b.

(a)   (b)
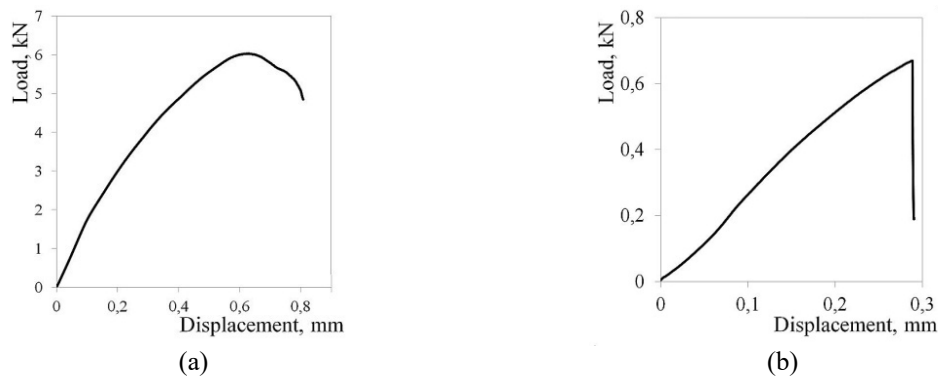
Figure 5. Compression diagrams of discs of (a) cast iron with a size of 10 × 4 mm and (b) graphite ARV-1 with a size of 8 × 8 mm according to the scheme of "Brazilian test".

The destruction of the graphite disk occurs dynamically on a virtually linear portion of the diagram at maximum load with the separation of the sample sometimes into several fragments. Initial cracks in the contact area were also found in the graphite sample with a size of 8 × 8 mm, which served as a source of destruction of the sample.

Sample tests showed a significant difference in the behavior of these two materials. Graphite turned out to be a more brittle material, the destruction of the samples of which was explosive in the linear deformation diagram. The ratio of strength limits of materials and destructive stresses determined by equation (2) are given in table 2.

Table 2. Ratio of breaking stresses and tensile strengths of cast iron and graphite.

Material	Plastic fracture	Brittle fracture
Cast iron	$\sigma_t \approx \sigma_u^t$,	-
Graphite ARV-1	-	$\sigma_t \approx 0.7\sigma_u^t$,

The results given in table 2 indicate that for graphite samples, the fracture resistance estimated from equation (2), 1.5 times less than the true tensile strength of the material.

6. Testing of samples of materials obtained by methods of electro-pulse consolidation of powders

6.1. Testing of samples of alloy WNiFe obtained by high-voltage consolidation

To obtain samples of heavy alloy WNiFe by high-voltage consolidation used industrial powder composition 90W–7Ni–3Fe, obtained by mechanical mixing of the component. Data on the composition and density of the powder are given in table 3.

Table 3. Composition of industrial WNiFe powder.

Element	ρ, g/cm^3	Weight, %	ρ_{theor}, g/cm^3
W	19.25	residue	
Ni	8.902	6.93	17.13
Fe	7.874	3.12	

Various modes of consolidation in the manufacture of samples of alloy WNiFe were investigated. The supplied voltage varied from 4.5 to 5.8 kV and the pressure from 100 to 250 kPa. Testing of samples allowed us to determine the levels of pressure and stress pulses during consolidation, providing optimal strength characteristics of the alloy WNiFe. Figure 6 shows the dependences of the material resistance to rupture on the voltage pulse level for samples sintered at the same pressures of 200 and 250 kPa. In all cases, the material fracture resistance was calculated using equation (2).
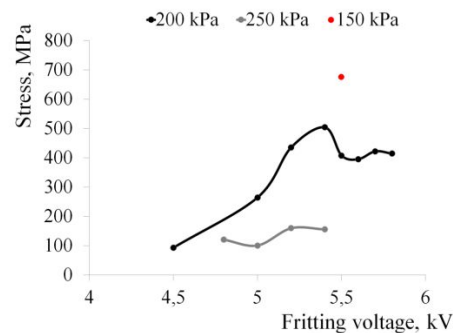
Figure 6. The dependence of the fracture resistance of the applied voltage at sintering.

At a pressure of 200 kPa, there is an increase in the strength of the samples with an increase in the voltage pulse to a value of 5.4 kV, then a decline. The growth and decrease in strength occurred against the background of the development of plastic defo rmations that preceded the brittle destruction of the samples. At a pressure of 250 kPa, we obtained an absolutely brittle fracture without noticeable traces of plastic deformation.

6.2. Testing of samples of aluminum oxide obtained by SPS

The results of the "Brazilian test" of cylindrical samples with a diameter $D = 10$ mm made of aluminum oxide powder by the method of electro pulse consolidation and calculated by the formula (2) are presented in figure7.
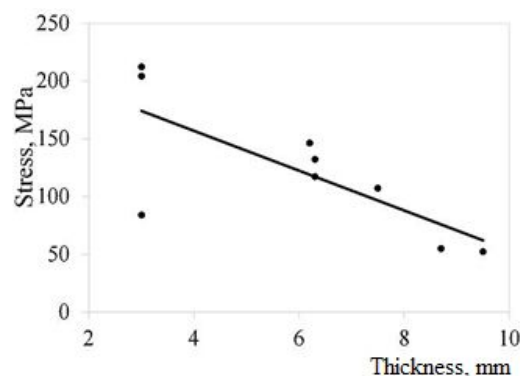

Figure 7. The test results of samples of aluminum oxide.

We note a good correspondence between the results obtained by the test of short cylinders according to the "Brazilian test" scheme and the results of the test of thin aluminum oxide disks with a   diameter of 15 mm and a thickness of 1.5 mm for bending on the annular support mentioned above.

7. Conclusions

Thus, the method, which combines calculations using FEM and experimental test results accor ding to the following schemes: "bending on the ring support" and "Brazilian test", makes it possible to determine the fracture resistance of materials obtained by consolidation from powders using the method of electric pulse action. Small samples in the form of thin discs are tested for bending on the ring support, and short cylinders (thick discs) - according to the scheme of the "Brazilian test". The influence of a number of technological factors on the strength of consolidated materials is studied using the proposed methods.

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