Experimental determination of elastic properties of the loose materials in the field of high pressures after heating

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Abstract. This article presents a physical study of materials’ elastic response (asbestos, slag microspheres, fireclay and corundum powders) at the pressure up to 70 MPa. The research is carried out after heating the samples up to 500 °C and exposure to crimping pressure up to 105 MPa. The graphical dependencies are given for each materials of relative strain from the pressure level. The original research technique of elastic response for materials samples having a loose aggregate state is proposed in laboratory. This technique is based on the use of a special laboratory equipment relatively simple and efficient design which combines power and measurement components. A detailed description of the scheme and principle of operation equipment is given. The calibration of dynamometer component of the special equipment is carried out and graphs of its work are presented. A technique for calculating the elastic moduli of material samples is proposed. The elastic moduli and the relative residual deformation of the material samples are determined. The ability of materials to preserve their loose properties are studied (dismantling ability). The conclusion about the possibility of using materials in the housing of pressure vessels is made as a means of protecting their load-bearing structural elements from unwanted heating and reducing in their strength characteristics.

1. Introduction

There are several studies of mechanical properties materials based on the asbestos [1-4].

The mechanical properties of impregnated asbestos-cements were studied in [1]. Authors investigated of the influence of the degree of impregnation and of the nature of the resin on the impregnation of asbestos-cement by polymeric resins produces composites. The elastic modulus of asbestos-cements was determined equal to 19613.3 MPa. In another study of materials was based on the asbestos fibers given of variation of Young's modulus in direct tension with mass fraction of asbestos fibers at the level 17000 MPa [2] with indicating the exact value for different asbestos-cement mortar composites equal 17200 MPa [3].

The anisotropy of mechanical properties of the asbestos-cements was studied in [4]. The dependence of the ratio of asbestos cement strength along the preferred orientation of asbestos fibers to the strength in transverse direction was calculated as a function of the asbestos-fiber orientation.

Elastic modulus studies fireclay is not researched by us. There are some researches of strength properties of fireclay [5], constitution and properties of ceramized fireclay refractories [6, 7] and...
residual properties of fiber-reinforced refractory composites with a fireclay filler [8]. Also recent study about of physico-chemical properties of some plastic fireclays is known [9].

Investigation of the mechanical and elastic properties of corundum and other materials based on corundum are presented in [10-17].

The research of pressure dependence of the elastic properties for Al₂O₃ (single crystalline aluminum oxide) with its densely packed is presented in [10].

The elastic constants of coordination polyhedral are used as input parameters to calculate the single crystal elastic constants of corundum [11]. The average deviation of the calculated elastic constants from the measured elastic constants is within 6%. The corundum crystal structure is composed entirely of AlO₆ octahedra.

In another study the refractive index and elastic properties of single-crystal corundum (α-Al₂O₃) up to 2100 K were studied in [12]. Another work was comparing values of elastic modulus (all directions of deformation) for pure α-Al₂O₃ which were obtained experimentally and calculated in the SCAD program [13].

The mechanical and physical properties of the metal matrix composite particulate reinforced were produced by a stir casting process are presented in [14]. In particular Young's modulus for corundum Al₂O₃ equal 308 GPa and for hybrid particle Al₂O₃/ SiCp Young's modulus is 358.5 GPa.

Investigation of mechanical and elastic properties of bulk corundum bodies (elastic modulus, microhardness, etc.) was obtained as a result of magnetic pulse pressing and sintering of composite powder mixtures Al₂O₃/Al carry out in [15].

In development of technology for durable corundum and zirconium ceramics mechanical activated powders were used [16]. The physicomechanical properties of ceramics were obtained based Al₂O₃ and ZrO₂, sintered from nanodispersed powders. In particular Young's modulus for Al₂O₃ equal 294.4 GPa and ZrO₂ equal 181.6 GPa respectively.

Finally, in 2018 the study of Chinese scientists about elasticity of corundum at high-pressures and temperatures was published in paper [17]. The thermodynamic and elastic properties of corundum at high pressures and temperatures were calculated based on density functional theory within the local density approximation. Also found in this study the elastic moduli of corundum, especially its shear modulus, exhibit a noticeable nonlinear dependence with pressure and thereby result in the nonlinear pressure dependences of wave velocities.

To create a new construction of pressure vessels providing the high technological modes (reaction medium temperature and pressure) [18] it is necessary to eliminate the phenomenon of unavoidable heating of the bearing wall reducing its mechanical characteristics. A constructive solution to this problem is placement of a heat-insulating layer between the shell above the heating elements and the bearing part of the vessel shell. With this arrangement the heat-insulating layer transfers force from the central shell to the bearing housing and is to have certain mechanical characteristics (compression elastic modulus).

The choice of material should be made taking into account the compression diagrams. As the alleged materials the asbestos, fireclay powders – traditional heat insulator materials as well as corundum powders and slag microspheres (loose material of gray color, its particles are spheres with diameter 30-110 microns) were investigated.

2. Methods

The research of the elastic characteristics of materials is carried out on a specially designed and manufactured equipment (figure 1) consisting of the base (8) located on a horizontal surface. Side panels are welded to the base (9), on which a support plate is welded on the top (10). The central rod (1) is located on the base plate which is fixed by the loading plate (11) with bolts (12) which pull it to the base plate. The cylinder is mounted on the central rod (5) and piston (6) for compressing material (7) in confined space. Powder compression force is measured by a special device (2) located between the adapter washers (13). The free corner of the central rod is mounted on a support (14). Compression of the test material in the device carries out by screwing on the central rod (1) loading screw-nut (3)
which moves along the threaded section on the central rod. Simultaneously with loading nut the bushing moves (15) and transmits force to the measuring device (2) (figure 2) and on the fixture. Thrust bearing (16) transmits axial force without torque on the movable plate (17), its movement corresponds to the deformation of the test material and is measured by indicator (4). The fit ring (18) drives the piston (6) which moves along the shell of the cylinder (5) with guaranteed gap along outer and inner diameters and compresses the test material (7).

Special dynamometer (2) is calibrated on a tensile testing machine with load change step in 50 kg. Calibration curves for loading \( a \) and for unloading \( b \) is presented on the graph (figure 3).

Figure 1. Constructive scheme of equipment for determination of the elastic properties of materials.
Figure 2. Constructive scheme of the special dynamometer for measuring compression force of materials.

Figure 3. Calibration curves of the special dynamometer (a – loading; b – unloading).

The device (figure 4), in which the test material (4) is located in a closed volume, consists of the cylinder (1), piston (2) and guide bushing (3) which is installed on the central rod (1) (see. figure 1) with guaranteed gap.
Figure 4. The scheme of devices for determination of the elastic properties of material.

To bring the test conditions closer to the operating conditions of the heat insulating layer the investigated materials are pre-processed by the temperature 500 °C and are pressure-tested in the hot state at this temperature by 105 MPa.

The initial thickness of the test material in the device (see. figure 4) is defined as

\[ l = 100 - (80 - x) \]  \hspace{1cm} (1)

The loading and unloading of materials are carried out with step in 5 MPa. The value of the current load is measured on the indicator of special dynamometer (see. figure 2) with precision \( \pm 0.01 \) mm; and deformation of the test material \( \Delta l \) on the indicator (4) (see. figure 1) with precision \( \pm 0.01 \) mm. The value of compression is measured by indicators with precision \( \pm 0.01 \) mm.

To calculate the elastic moduli of materials the methodology described in [19] is used.

The value of the elastic modulus at each loading step is determined by the formula

\[ E_i = \frac{(P_{i+1} - P_i)}{(\Delta l_{i+1} - \Delta l_i)} \]  \hspace{1cm} (2)

where \( P_{i+1} \) and \( P_i \) – value of the loading at the next step and at the current point of determination of the elastic modulus, MPa; \( \Delta l_{i+1} \) and \( \Delta l_i \) – value of the elastic deformation from loading, respectively, mm.

The standard value of the elastic modulus is calculated as

\[ E_n = \frac{1}{n} \sum_{i=1}^{n} E_i \]  \hspace{1cm} (3)

The standard deviation of this characteristic has the form

\[ S = \sqrt{\frac{1}{n-1} \sum_{i=1}^{n} (E_n - E_i)^2} \]  \hspace{1cm} (4)

and the coefficient of variation is calculated as

\[ V = \frac{S}{E_n} \]  \hspace{1cm} (5)

According to the formula (3) the average accuracy indicator is calculated \( \rho_n \). For one-side confidence probability 0.85 coefficient value is adopted \( t_n = 1.16 \).
\[ \rho_a = \frac{t_a V}{\sqrt{n}}. \]  
(6)

Reliability coefficient is calculated by the formula

\[ \gamma_g = \frac{1}{1 - \rho_a}. \]  
(7)

then the calculated value of the elastic modulus of the sample is found as

\[ E_p = \frac{E_n}{\gamma_g}. \]  
(8)

3. Results and discussion

The compression curves for four materials were received by us and presented at figure 5-8. The solid curve means the pressure loading process and the dashed curve is unloading process.

On the figure 5 compression curves of fireclay powder for primary and elastic loading-unloading are shown. The deformation of fireclay powder after the primary cycle is 0.7%. Starting from the third loading cycle the material behaves elastically and having significantly less strain. The elastic modulus of fireclay powder in the elastic duty cycle up to 5 MPa is 3369 MPa and when unloading 2702 MPa. With a load more than 5 MPa the elastic modulus is 21738 MPa and when unloading 36775 MPa.

Asbestos deformation already in the primary loading cycle is practically elastic (figure 6). Its permanent deformation is 0.04%. After the third loading cycle the elastic deformation of this material follows a more monotonous curve and also there is its significant decrease. The elastic modulus of fireclay powder in the elastic duty cycle up to 5 MPa is 4459 MPa and when unloading 4361 MPa. With a load more than 5 MPa the elastic modulus is 19833 MPa and when unloading 46848 MPa.

Corundum powder has a permanent deformation of 0.21% in the primary loading cycle (figure 7). Also after the third cycle the corundum powder has an elastic deformation. The elastic modulus of fireclay powder in the elastic duty cycle is 57965 MPa. When unloading happen up to 5 MPa the elastic modulus is 59177 MPa and lower the value 5 MPa is 13746 MPa.

The slag microspheres work like other materials. Their residual deformation after the first loading cycle is 0.15% (figure 8). Elastic modulus of slag microspheres in an elastic duty cycle up to 15 MPa is 6777 MPa and when unloading 5464 MPa. With a load more than 15 MPa the elastic modulus is 32205 MPa and when unloading 81506 MPa.
Figure 5. Fireclay powder compression curves. Primary loading – 1; Elastic loading – 2.

Figure 6. Asbestos compression curves. Primary loading – 1; Elastic loading – 2.
4. Conclusion
The experiment shows that significant residual deformations observed in fireclay powder are 0.7%. Starting from the third loading cycle materials behave resiliently. Moreover, there is a significant decrease in deformations at the same pressures.

The elastic modulus with a load more than 5 MPa for fireclay powder, corundum powder and asbestos and also more than 15 MPa slag microspheres significantly increase. Also it is necessary to note that the elastic modulus determined from the linear part of the curves of elastic deformations of materials are different when loading and unloading (the elastic moduli increase upon unloading).

Thus, the application of the investigated materials as a heat-insulating layer in the construction of pressure vessels of the type [18, 20] is possible only with the presence of a stage of preliminary preparation of the material for the working technological loads by mechanical action after the final assembly of the entire vessel or cylindrical housing. Such mechanical action can be realized by pre-compressing the heat-insulating layer during mounting and technological crimping pressure which creates residual contact pressure more than 5 MPa for fireclay powder, corundum powder and asbestos and also more than 15 MPa for slag microspheres.

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