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Thermal Insulation Properties Research of the Composite Material "Water Glass - Graphite Microparticles"

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Abstract. Research results for the composite material (CM) "water glass - graphite microparticles" with high thermal stability and thermal insulation properties are given. A composition is proposed consisting of graphite (42 % by weight), water glass $Na_2O(SiO_2)_n$ (50% by weight) and the hardener - sodium silicofluoride Na_2SiF_6 (8% by weight). Processing technology of such composition is suggested. Experimental samples of the CM with filler particles (graphite) of a few microns in size were obtained. This is confirmed by a study of samples using X-ray diffraction analysis and electron microscopy. The qualitative and quantitative phase analysis of the CM structure was done. Values of limit load causing destruction of the CM were identified. The character of the rupture surface was detected. Numerical values of the specific heat and thermal conductivity were defined. Dependence of the specific heat capacity and thermal conductivity on temperature during monotonic heating was obtained experimentally. Studies have confirmed the increased thermal insulation properties of the proposed composition. The CM with such properties can be recommended as a coating designed to reduce heat losses and resistant to high temperatures. Due to accessibility and low cost of its components the proposed material can be produced on an industrial scale.

1. Introduction

Currently one of the most important problems is the creation of materials and coatings designed to reduce heat loss and resistant to high temperatures. One way to solve this issue is to develop a technology of preparation and application of composites, which when applied to a surface slow down its heating [1]. Over the past decades, composites have been extensively put into practice and replaced traditional materials in energy, transportation, electronics and other fields of endeavor. The difference between the traditional materials and most of the composite materials is that the production process of the latter can be combined with manufacturing process of a product [1]. The use of graphite as a filler [2] is substantiated by its high temperature and chemical resistance. There are widely known composite materials (CM) based on magnesium and aluminum oxides [3-6] and other alumina-components that are similar in composition but have different reactivity with respect to water glass. There is also international experience with composite technology based on carbon fibers [7-12].

The authors of this paper designed, produced and experimentally investigated a composite material of a new structure with high thermal stability and thermal insulation properties.

2. Investigation of the source filler material

The phase composition of the original graphite was investigated using X-ray diffraction analysis performed on X-ray diffractometer DRON-3 using FeKa radiation. The intensity at each fixed angle was recorded every 20 seconds. The images were taken in reflection geometry within the angular range 3-145° with the step size of 0,05°. From the analysis (figure 1) it was concluded that the modification of graphite is hexagonal. The material revealed the presence of SiO_2 impurity up to 3% by weight.

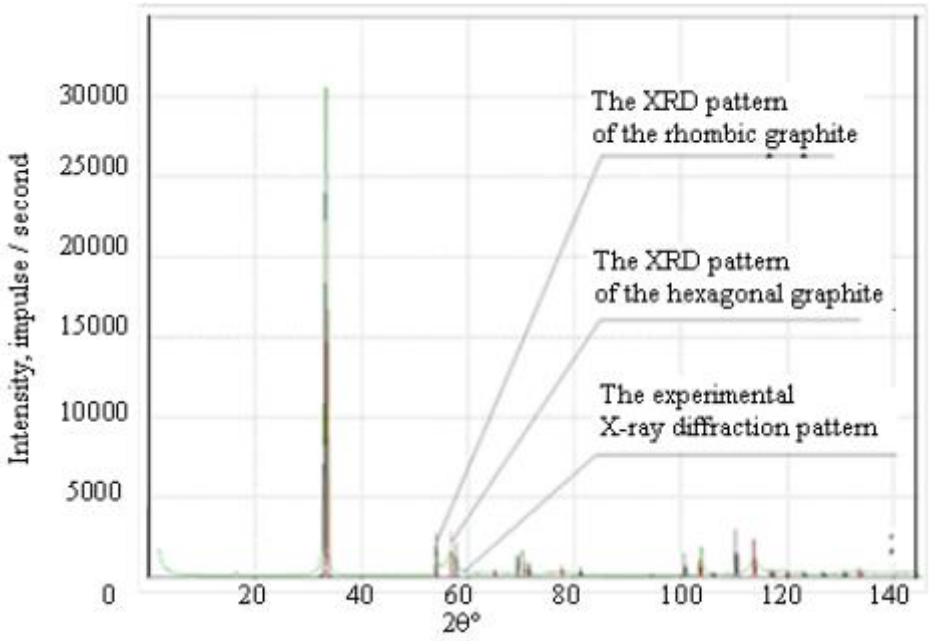

Figure 1. General view of the theoretically calculated X-ray diffraction of the rhombic and hexagonal graphite, as well as the experimental X-ray diffraction pattern of the graphite sample in the range of 20=40°-80°

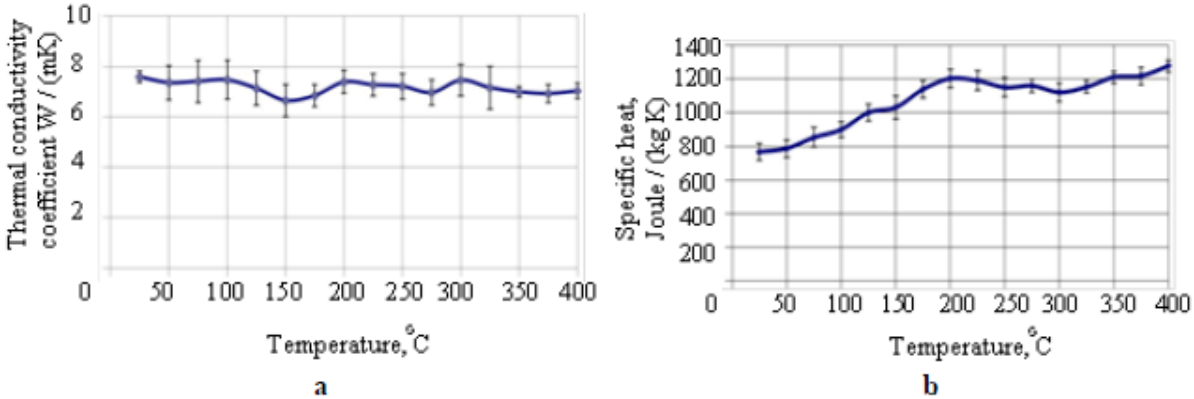

Figure 2. The values of the thermal conductivity coefficient X of graphite (a) and the values of the specific heat *c* of graphite (b) when the temperature changes from 25°C to 400°C

In order to determine the thermal stability of the initial samples and evaluate thermal insulation properties, the thermal conductivity coefficient (Figure 2a) and the specific heat (Figure 2, b) were measured at temperature ranging from 25°C to 400°C using the IT-X-400 and the IT-c-400 in

monotonic heating mode [13-15]. The results of the measurement were processed using the methods of mathematical statistics. Random error in measurements with Student's coefficient equal to 2.9 and probability of 0.90 is shown in the graphs (Figure 2).

The Fig. 2a shows that the thermal conductivity coefficient of the CM when temperature is increased from 25°C to 400°C does not change significantly and remains in the range of 6-8 W/(m-K), taking into account random error in measurement. Fig. 2b illustrates the piecewise linear nature of the increase in the specific heat. With temperature changing from 25°C to 200°C there is a significant increase in the specific heat from 800 Joule/(kg-K) to 1200 Joule/(kg-K), compared with heating at temperature changing from 200°C to 400°C, when the specific heat increases slightly from 1200 Joule/(kg-K) to 1270 Joule/(kg-K). Such dependence can be explained in particular by evaporation of the internal moisture in the first section of heating of the CM.

3. Preparation of composite material based on water glass with graphite filler

At the first stage of production of the micro composition, micro powder of graphite with a particle size of 1-10 microns was obtained by milling the initial samples, which, in our opinion, provides a larger wetting surface, increases adhesion and expands the area of phase contact. Milling was performed on a laboratory planetary centrifugal mill - Hephaestus activator-2 (AGO-2U).

At the second stage the composite material was prepared. For the binder component we selected soda water glass Na_2O (SiO_2) n with the silicate modulus 2, 8. Water glass composition was prepared at ambient temperature by manually mixing in a mortar graphite powder (42 wt%), water glass (50% by weight) and hardener - sodium fluorosilicate Na_2SiF_6 (8% by weight). The ratio of components was chosen by the authors experimentally upon condition that there is no destruction of the samples when heated to 100 °C, making it possible to conduct studies at elevated temperature. Percentage ratio by weight was determined by weighing on an electronic balance. The process of wetting the particles with the binder component lasted 7 days. Pictures of the surfaces of the samples taken with electron microscope Hitachi SU 1510 (Figure 3) confirmed that the graphite particles in the composition have a size of several micrometers. The surface structure is clearly visible.

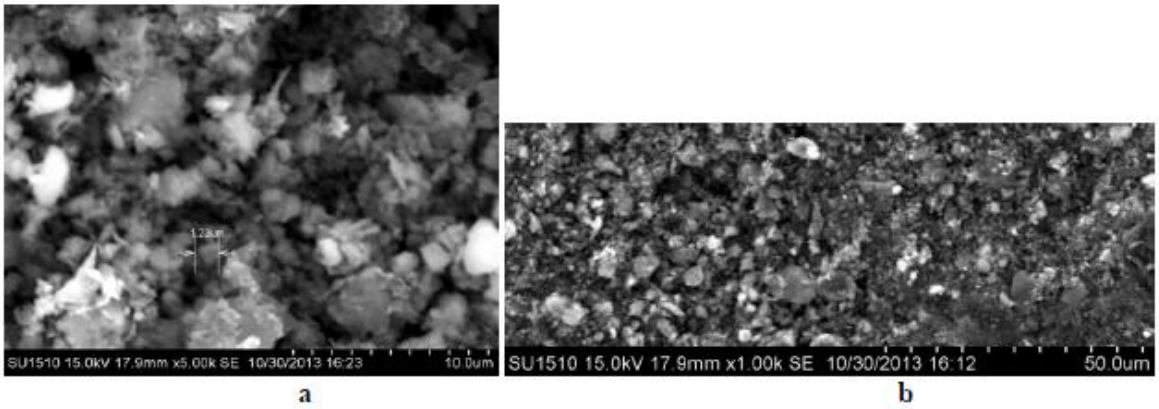

Figure 3. Photomicrographs of the surface of the water glass composition with graphite filler with the horizontal picture size: 10 microns (a); 50 microns (b)

4. Mechanical testing of the composite material

An important characteristic that determines the mechanical properties of the composite material is adhesion [6] This characteristic greatly affects reaching the limit state under loads. In order to investigate the processes that occur during mixing graphite with water glass in the presence of a hardener, we conducted mechanical tests of the composite material [16, 17] to determine the load required to tear the test coverage off the base surface. The finished composition was applied on wood samples (thickness 2 mm). After thorough drying, the samples were torn off measuring the load at the breaking point with accuracy up to 10 N/m^2.

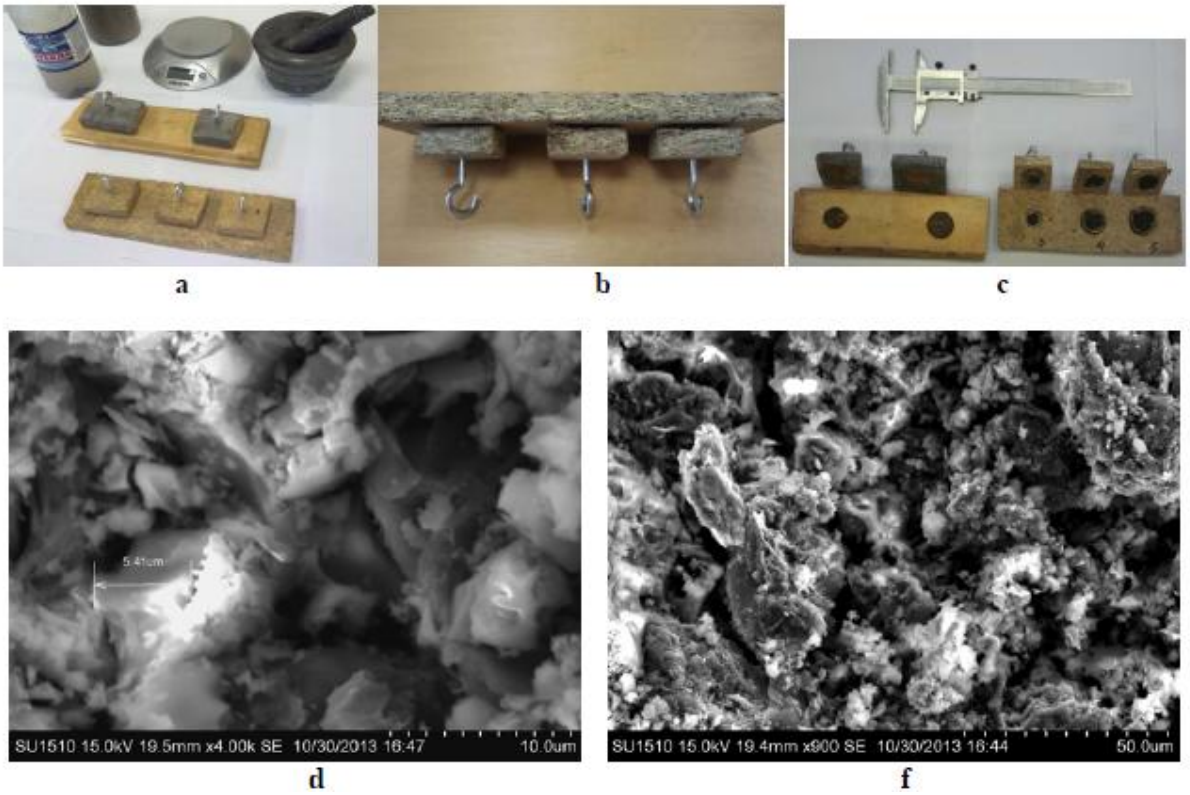

Figure 4. Tools to measure the load required to tear off the coating and pictures of the rupture surface after the experiment (Electron Microscope Hitachi SU 1510): wooden tool (general view) (a); wooden tool (side view) (b); type of the rupture surface (c); micrographs of the rupture surface (scale 10 microns) (d); micrographs of the rupture surface (scale 50 microns) (f)

The load was applied to the sample by hanging weights on hook tools (Figure 4a, b). The destruction of the samples (Figure 4,c-f) occurred along the boundary of the composite material and the base surface at the structure in the longitudinal section. Cracks in the samples were not formed. Limit fixed load value was 1,22 MPa.

5. Investigation of structure and physical properties of the composite material based on water glass with graphite filler

In order to ensure reliability of the results of the experiment four identical samples were investigated. To determine the thermal stability of the composite two samples were monotonically heated from 25° C to 400° C. The other two samples were control samples. We have performed X-ray analysis of the samples on DRON-3 diffractometer (FeKa radiation, wavelength 1,93597-10^{-10} m) (Figure 5).

Comparison of the radiographs of all four samples - two heated up to 400° C (Fig. 5, a, b), and two that have not been heated (Figure 5, c, d) - makes it clear that the position of the diffraction peaks coincide. Qualitative analysis showed that the samples are multiphase. Graphite phase belongs to hexagonal graphite. Silicon dioxide SiO_2 is in the crystalline state of a-quartz. Change of the background, when 20° = 27, indicates that the composition also contains amorphous silicon dioxide SiO_2, and sodium fluoride NaF in the form of crystals. The presence of sodium silicofluoride Na_2SiF_6 phase in crystalline form shows that the chemical reaction of silica gel, which binds the filler phase, did not go completely.

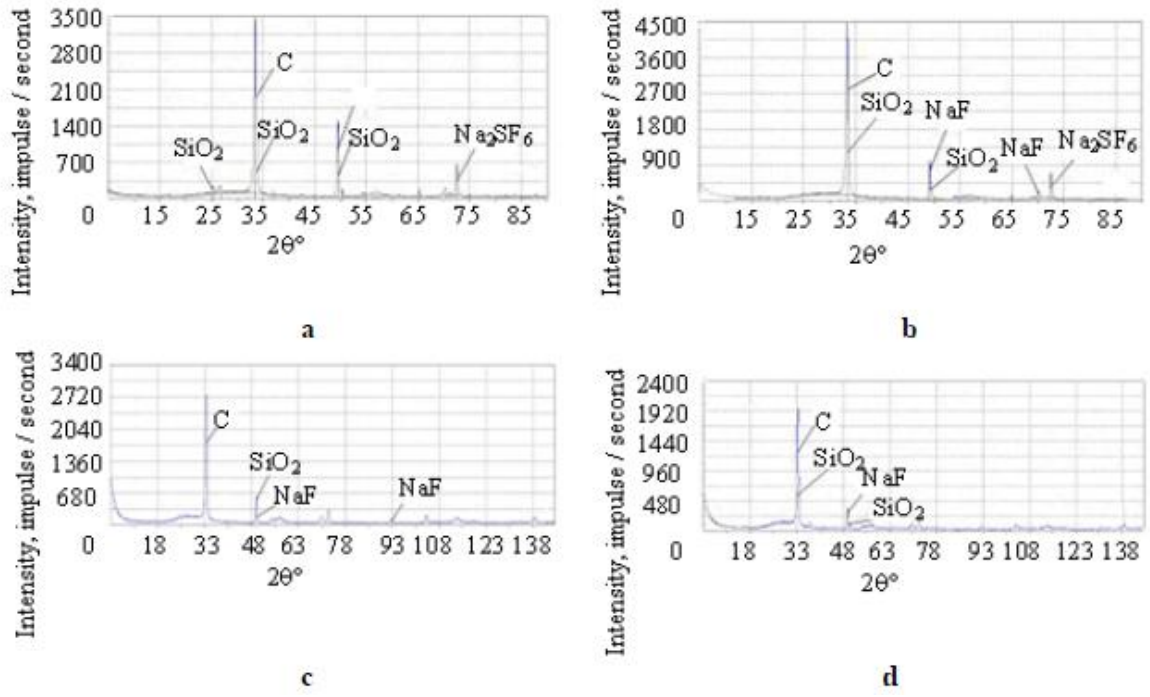

Figure 5. XRD patterns of the samples: No. 1 (a); No. 2 (b); No. 3 (c); No. 4 (d)

Quantitative analysis of the phase composition was performed on electron microscope Hitachi SU 1510 and DRON-3 diffractometer. The analysis is based on the exact position of the diffraction peaks. The results of the CM research are shown in the Table. The emission spectra obtained with the electron microscope are shown in Figure 6.

Table. The values of mass and mole fractions of the components of the CM

Phase	Mass fractions of the components in the samples,%				Mean,%	Molar mass of, g / mol	Mole fraction, *x*
	No. 1	No. 2	No. 3	No. 4			
C	42,1	42	44,2	42,4	42,6	12	0,76
NaF	22,8	21	22,8	21,7	22,1	41,98	0,11
SiO2	33	32,7	30,9	34,2	32,8	60,08	0,12
Na2SiF6	2,1	4,3	2,1	1,7	2,5	188	0,01

Thus, the authors demonstrated that the investigated material has heat resistance and is able to maintain substantially unchanged chemical structure in the temperature range from 25°C to 400°C.

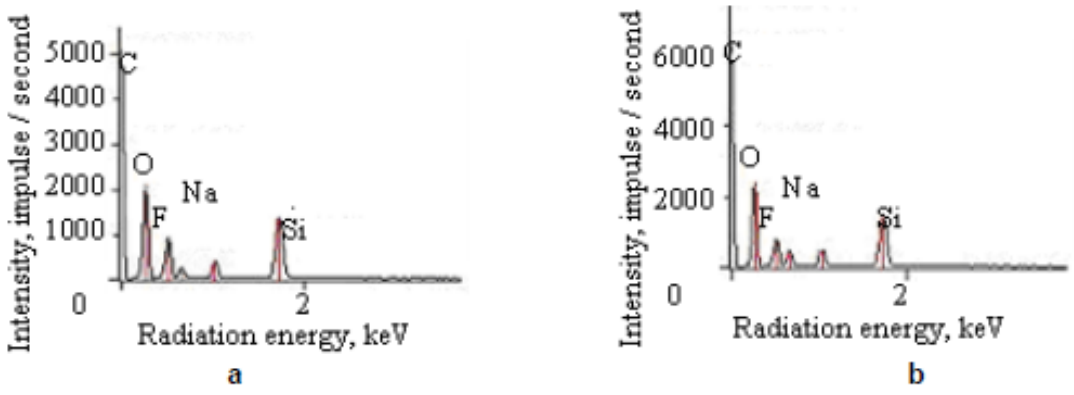

Figure 6. The emission spectrum of the CM samples not heated (sample No. 1) (a) and heated (sample No. 2) (b)

To evaluate the thermal insulation properties of the samples we measured the CM thermal conductivity coefficient X, W/(m-K) (Fig. 7a) and the specific heat c, Joule/(kg-K) (Fig. 7, b) at the temperature ranging from 25 ° C to 400 ° C by using the IT-X- 400 and IT-c-400 in monotonic heating mode [7-9]. The measurement results were processed by the methods of mathematical statistics. Random error in measurements with Student's coefficient equal to 2.9 and probability of 0.90 is shown in the graphs (Figure 7).

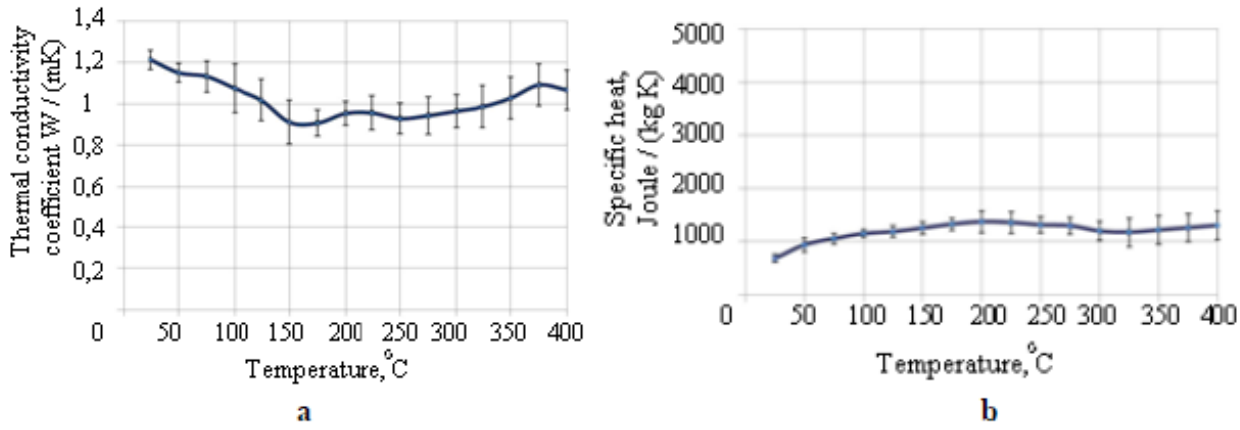

Figure 7. Temperature dependence of thermal conductivity coefficient λ of the CM samples (a) and specific heat ccm (b) in the range of 25°C to 400°C

6. Conclusion

The composite material with high heat resistance and insulating properties was studied [6, 18]. XRD analysis of the samples subjected to heating to 400 ° C showed that the material is able to maintain chemical structure unchanged, which confirms its higher thermal stability. The experimentally obtained dependence of the specific heat capacity and thermal conductivity coefficient on temperature showed good thermal insulation properties of the composite material.

Composite material with these characteristics can be used as a material or coating designed to reduce heat loss and resistant to high temperatures. Due to general availability and low cost of its components, the proposed material can be produced on an industrial scale.

7. Acknowledgment
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