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TECHNOLOGICAL ASPECTS OF CREATION LIGHT-WEIGHT COMPOSITE MATERIALS FOR AERO-SPACE APPLICATIONS

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Modificated of the surface of EG-particles, it was proves the possibility change of deformation-strength properties and mechanism of thermal oxidative destruction EG-based moulded materials. The synthesis at the surface of particles of EG of complicated compounds like aluminium silicate increase degree of deformation-strength properties and gives high rises degree of protection of compact specimens from the thermal oxidative destruction.

Introduction. One of the actual trends of developing modern materials science is an elaboration and employment of new lightweight composite materials. Modern tendencies of decreasing the weight of various constructions and details give the rapid demand increase to using the lightweight composite materials for the aerospace applications and it makes such materials more and more marked objects for the fundamental and applied researches. As it is shown by the analysis by the analysis of theoretical and practical principles of creating composites materials the basic theories of creating the composite materials are not applicable to describe the components interaction in a proper way, but the employ of these theories concerning the superlight composite materials, with have the multileveled super-heterogeneous structure, is a total problem in general. This work present using perspective trends in formation of exfoliated graphite (EG)-based composition materials by synthesis of non-organic combinations on the surface of EG-particles, it was proves the possibility of purposeful change of properties both of EG-powder surface and EG-based moulded materials.

Materials. The powder of crystalline graphite was subjected to processing by sulphuric acid H_2SO_4 with an introduction of ammonium as an oxidizer. As a result compounds of intercalation of graphite (CIG) by sulphuric acid were obtained. After the excess of sulphuric acid was washed out, the remains of CIG were collected. Aluminium oxide modificated EG was carried out by one minute thermal processing of mixture

of CIG remains and of aluminium sulphate solution at the temperature of 900°C. Under these conditions, simultaneously with the formation of wormlike particles of EG, the pyrolysis of aluminium sulphate occurs with the formation of aluminium oxide smoothly lodged on the surface. EG modificated by silicon dioxide was obtained by thermal processing of mixture of CIC and tetraethoxilanium solution during one minute at the temperature of 900°C. EG modificated by the intricate compound in the form of aluminium silicate was received by thermal processing at the temperature of 900°C of the mixture of CIC remains, tetraethoxilanium solution and aluminium sulfate in the ratio SiO_2:Al_2O_3=2. Under these conditions imultaneously with the formation of wormlike particles of EG, the pyrolysis of the aluminium sulphate and tetraethoxilanium with synthesis of aluminium silicate which is lodging on the surface of EG particles. The find product comprise the EG powder containing kaoline of polywedged structure smoothly distributed on the surface of graphite's particles. Such technological conditions of obtaining the TEG led to the particular kind of deformation of the plane carbon layers. It caused the formation of vermicular powder particles with a honeycomb structure, which has a bulk density of 50−1200 kg/m^3.

Methods. Small-sized test specimens, manufactured from the powders foamed material and exfoliated graphite outlined above, were shaped into a cylinder of 20 mm in diameter and 20 mm in height. Identification of the obtained modificated powders was carried out using the X-ray phase analysis. Then processing the obtained powders by means of a one side compaction method, cylindrical specimens of 20 mm diameter were made. Evaluation of thermaloxidative stability was exercised by determination of loss in mass (M= ($\Delta m/m$) x100%) at the same initial sample of compact specimens (Δm) of the same density, but which are differ by the initial mass of specimens (m) per the time unit (t). Calcinations was carried out at the temperature of 750°C during 20 minutes.

The study of processes of deformation of specimens with density of 1000 kg/m^3 was exercised under the monoaxial compression in the regime of continuous and repeatedly static loading with recording of diagram deformation (Fig.1). During the testing the stress influencing the specimen's bulk (G), deformation of a specimen (ε), energy of elastic deformation (E_e), energy of plastic deformation (E_p) and energy of non-elastic deformation (E_n) were registered. Energy of non-elatic deformation is nothing but the energy of absorption (dispersal) by the material

volume of part of total deformation energy (E_t=E_e+E_p). Such representation of deformation diagrams obtained in the regime of repeatedly static deformation with rising load made it possible to introduce structural criteria D which complies with the part of deformation total energy absorbed (dissipated) by the material as a result of its structural rearrangement (D=E_n/E_t). Experimental investigation of structure of specimens was conducted using the visual and optical microscopy method.

The material testing by the tribo –spectral method involved the penetration of a loaded indentor into the specimen surface and motion of the specimen in the regime of the elasto-plastic strain (Fig. 2). In the process of the specimen motion, the indentor performs the forced vibrations, whose character is caused by the different physical resistance of the structural components of the material surface layer. The change of the resistance to the development of the elasto -plastic, strain within the fragments and on the fragment boundaries of the structure is random in its behaviour and corresponds to the instantaneous change of the forces (P_t) and (P_h) (Fig. 2) which is measured in real time scale and is transformed by means of laser sensor into electrical signal.

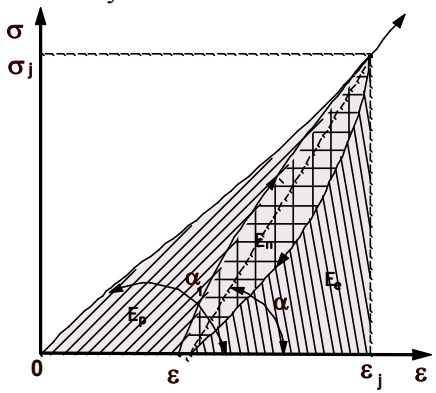

Fig.1. Two-coordinate diagram of the repeated static loading: *1* – loading; *2* – unloading. (*b*) Normal tribogram. (*c*) Tangential tribogram

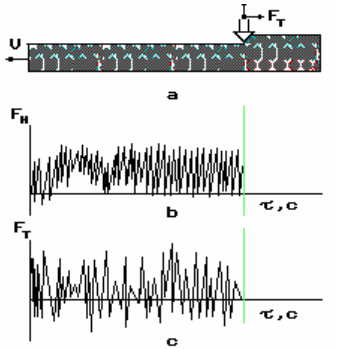

Fig. 2. Physical background on the tribo–spectral method:(*a*) Process scanning material's surface

As result, the process of the forces change in the course of time may be thought of as a set of polyharmonic vibrations processes with periods which are related to the sizes of fragments, blocks, and other structural components of the material. The spectral dens structural components of the materiality (S_t) and (S_h) of such a process will have

maximal at the frequencies which correspond to the sizes of fragments, blocks and other. Before testing, specimens were sequentially fixed in holders on the anvil. A tetrahedral diamond indentor was forced into the material tested at the load of 10−50 mN. After holding under the load during 10−15 seconds, the specimens were moved at the speed of 20−80 mm/s, and the value of the tangential force (P) acting on the indentor which vibrated at the frequency of 20 Hz was registered. The amount of the experimental performance for the probability process was more than 1200 values. The tests were made on different areas of the specimen surface layer in order to obtain an enable of the probability process characteristics. The treatment of the experimental time sequences was carried out using the Fourier transformation method which allows one to perform a simple interpretation of the results obtained through their spectral analysis using a computer. The result of the experimental studies is the values of the tribospectral characteristics $S = (F)$, where S is the spectral density, F is the frequency of the indentor forced vibrations.

The tribo –spectral method with step-by-step control in the process of specimens calumniation in the range of 500°C to 1000°C (every 50°C) and time interval of 10 minutes has been used. Simultaneously, the estimation of thermo – oxidation stability of specimens has been carried out by means of determination of specimen mass loss $(\Delta m/m)$ at 100 percent of the same density with different initial mass.

Results. Carried out initial micro-mechanical tests by the nanotribospectral test tribo–spectral method at 20°C have allowed to see non-monotonous changes in tribospectral characteristics depending on the percentage of aluminium oxide in EG materials 3% of modificator corresponds to the maximum heterogeneity of deformation– strength characteristics of modificated specimens surface which is followed by the increased resistance to contact deformation, (Fig. 3).

Results of experimental studies of thermo – oxidation stability of EG specimen are shown in Fig. 4. As it can be seen, EG composite materials with aluminium oxide increase thermo–oxidation stability of specimens with the sme density in the range of temperatures from 600 °C to 900°C. The correlation between tribo–spectral characteristics and the degree of burning out of specimens shows that at *3* – percent modificator the deformation stress condition of the structure is not changed under the temperatures up to 850°C (Fig. 5, curve*1*). The structure of original specimens and that of specimens with low content of the modificator have

some are of considerable increase of heterogeneous properties n the range of temperatures from 500°C to 800°C.

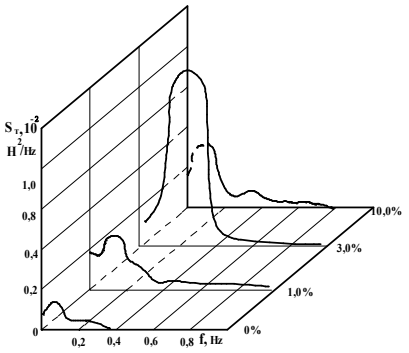

Fig.3. EG specimens decrease of mass with different content of the modification

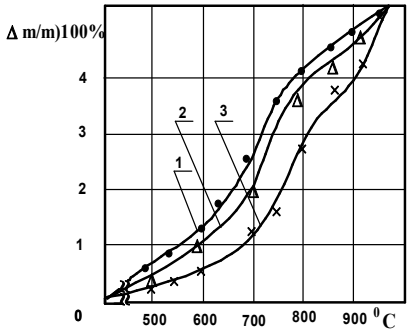

Fig. 4 Thermal oxidative stability of EG compact specimens: *1*–initial; *2*–1% Al_2O_3; *3*–3% Al_2O_3

From the point of view of physics it characterises the intensive process of oxidation (destruction) of specimen material in active centres which are badly protected (Fig. 5, curve 2) or which are not protected at all (Fig. 5, curve *3*). The results of the thermal oxidative of EG powder specimens and compact specimens, and their modificated forms are shown (Fig. 6). As it can be seen the ambiguous influence of the processes of synthesis of simple and intricate compounds on the EG-particles surface, directed towards the thermal oxidative stability of investigated specimens is evident. Thermal oxidative stability of specimens of EG modificated by SiO_2, practically does not differ of specimens from the powder of initial EG. Compact specimens of poders modificated by aluminium oxide and aluminium silicate have the less velocity of burn-out than initial EG compact specimens. The protection of EG from oxidation is evident. The synthesis of modificator in the form of intricate compounds, aluminium silicate, therewith produces a greater protection of EG from oxidation, than synthesis of simple modificators in the form of aluminium oxide and silicon oxide. Thermal oxidative stability of compact specimens essentially depends on the density, with an increasing of the effect of protection of EG compact specimens from oxidation with it.

The analysis of diagrams of continuous deformation specimens bulk shows that the process of modification of EG powder by an alu-

minium silicate at different concentrations ambiguously influences the resistance of specimens with equal density (1000 kg/m^3), Fig. 7. Visual and optical microscopy shows that the initial EG has a homogeneous, porous and fine micro structure, and graphite surface are disoriented. The area of the curve of deformation of the initial specimens of EG has a positive curvature.

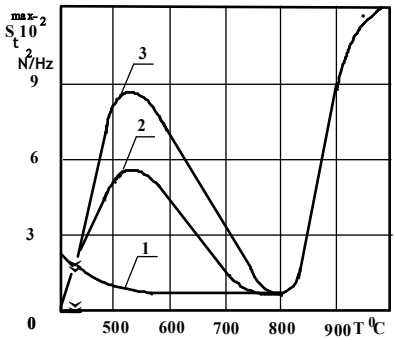

Fig.5. Maximal spectral density in the process of thermo-oxide destruction of EG specimens with content of the Al_2O_3:3%(*1*);1%(*2*); 0%(*3*)

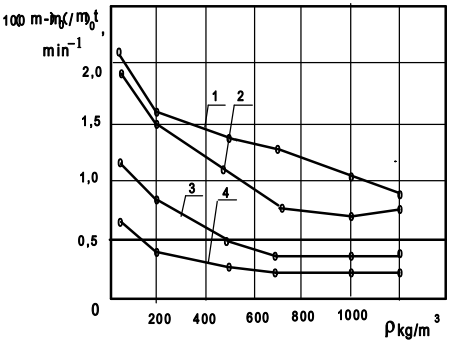

Fig.6.Thermal oxidative stability of EG compact specimens: initial EG(*1*) and modifiacated by SiO_2(*2*); Al_2O_3(*3*); (SiO_2+Al_2O_3)(*4*)

The collapse of pores leads to that the area of subsequent increasing of tension and the deformation of compression has the linear character till the initial moment of plastics deformation of inner pores and microdefects. It testing that the defects in continuity of materials in the form of pores with the active external load definitely resisting the specimen, and "collapsing" in the process of next plastics deformation, is available in the non-modificated EG.

Modificated EG has a layer structure the fragments of EG-particles are oriented perpendicularly to the acting external load. They have a lens-like pores and micro-separation. The beginning of the curve of deformation of modificated EG has a negative curvature. In modificated EG the presence in the layer structure of lens-like pores and micro-separation is connected with the S-like character of the curve of repeated static deformation. During the deformation of material with such structure the elastic resistance is similar to the resistance of lens like springs. The slope of line at the origin of he diagram of deformation (α_1) (Fig. 1) characterises the initial rigidity framework structure of the ma-

terial and does not bear the physical meaning as the elongation modulus (the Young's modulus) of continuous materials.

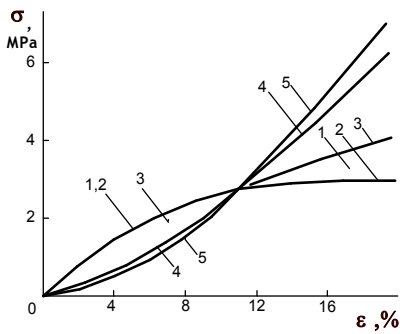

Fig. 7. Diagrams of the bulk deformation of specimens EG with various modification(SiO_2 +Al_2O_3) concentration (c):0%(1);1,0%(2); 6,0%(3);16%(4);25,0%(5)

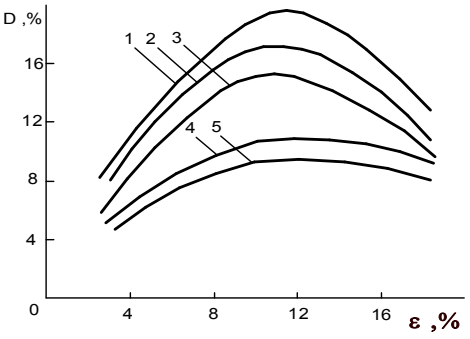

Fig. 8. Dependence of the structural criteria at the bulk deformation of specimens EG with various modification (SiO_2 +Al_2O_3) concentration (c): 0%(1); 1,0%(2) 6,0%(3); 16%(4);25,0%(5)

After the initial stage of deformation, a completion of the slope of axis of hysteresis (tgα) characterises the ability of he material being deformation impregnated and disimpregnated. For non-modificated EG and the EG-specimens with low content of modificator (c) we note an insignificant changes of tangent of slope of the hysteresis loop axis (tgα) during their bulk deformation. In this case the material is called as deformation stable. With the increasing of the modofocator's content in EG, the tangent of the slope of hysteresis loop axis (tgα) is magnified, i.e. the material remains deformatively-sealed.

Changes structural criteria D of the bulk deformation of specimens has a non-linear and stage character (Fig. 8). The first stage by the growth of D criteria is determined by the ability of he EG to absorb the parts of the total deformation by the internal bulk of the material. Absorbed energy is connected with the deformation and the failure of defects of continuity in the form of pores. The intensity of energy processes taking place on this stage of deformation an the structural rearrangement of the material attended upon them are tended to decrease as the content of modifator in EG increases. It testifies that the number of defects of continuity is user now as well as the changes in their types in

the material structure as the concentration of modificator in EG increases. At the second stage when criteria D is maximised on the one hand the external stresses cause the high air pressure and gas accumulated in the remnant defects of material bulk continuity and on the other hand the energy of activation of the process of deformation of the solid part of the material structure because of changes of the potential barrier decreases. In this case the bulk structure transition into the balance is evident. At the third stage the level of D criteria is decreasing in response to the general energy processes of the structural failure. The Fig.8 shows that the kinetics of changes of structural criteria D of the bulk deformation the limiting structural balanced condition of material, essentially depends on the content of the modificator. It proves that the process of synthesis of aluminium silicates on the surface of EG leads to the formation of the composite powder structure energetically more resistant to the deformation.

Conclusions:

1. Developed the methodology of supervised creating the lightweight composite materials of the exfoliated graphite (EG) with the employ of principles of estimating tribological and deformation-strength properties, with can used for defining the features of interrelation of the processes structural transformations of their creation can change the mechanism of thermal oxidative destruction.

2. The investigations conducted reveals that the synthesis of simple and intricate compounds on the surface of particles of EG can changes the deformation properties and thermal oxidative stability of formatted materials on its base.

3. The synthesis at the surface of particles of EG of intricate compounds like aluminium silicate gives a high degree of protection of compact specimens from the oxidation and increase degree of deformation rises.

4. Prepared lightweight products for aerospace assignment with high deformatively strengthened and high thermal oxidative stability.

5. Tribo –spectral method can be applied for the supervised of the technological processes of modified EG based-materials production.