Development of technology and investigation of structure and properties of composites on the basis of shungit and polymer resins

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Abstract. The technology for manufacturing of composites based on polymer (epoxy) resins and shungites was developed by using of the electron irradiation and experienced samples with different percentages of the components a) 90 and 10 %, b) 70 and 30 %, c) 50 and 50 %, were manufactured respectively. It was carried out the series of experimental studies of the structure and properties of the obtained materials, including the atomic force microscope images were obtained and Raman spectra of these composites were measured and their analysis was performed.

Introduction

It is known from the literature that carbon materials have a high mechanical strength. In the case of mixing them with various polymeric materials, it is possible to obtain sufficiently thin plate with high strength and resistance to external influences, particularly to radiation. Such materials can be used in conventional and in the aviation industry, medicine, agriculture, engineering, space objects; in home appliances, car industry, etc. In this regard, it is evident that carrying out such works is very important as the creation of new materials with sufficiently stringent requirements (high mechanical stresses, large changes in temperature, aggressive media, acids, alkalis, organic solvents, etc., the ambient humidity, etc.) is caused by the their practical application in various industries, including Kazakhstan, Russia, CIS and non-CIS countries. The use of high-energy electron fluxes gives undoubtedly a substantial industrial and economic effect. Effect of ionizing radiation on solids for such materials is specific and has a number of significant features associated with the properties of the solid state: by a regularity of the structure (crystallinity and defectiveness), by the electronic interactions between the components of the solid particles, by shortness of diffusion of large particles on the surface and volume, etc. Taken together these factors determine the kinetics of chemical reactions generated by ionizing radiation, as well as characterize the output of stabilized intermediate and final products of transformation [1 - 4]. In contrast to the photochemical reactions at radiative initiation of transformation occurs not only on the surface and in the subsurface layers, but also in the whole volume of the material and first of all in the fields of micro-, macro- and nanodefects.

The experimental technique

We have developed the technology for manufacturing of composites based on polymer (epoxy) resins and shungites, which includes the following steps: 1. The choice of powder on the basis of shungit. 2. The qualitative analysis of the granulometric composition of the powder and study of its physical and mechanical properties. Additional grinding of the material. 3. The analysis and selection of the type of binder (resin, etc. to improve the parameters by pressing and sintering). 4. A detailed analysis and sub-

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sequent selection of the parameters of electron irradiation (energy of high-energy electrons, beam intensity and dose of electron irradiation). 5. Fabrication the target holder and adaptations. 6. The analysis of beam parameters and the development of a technique of irradiation components of composite material with high-energy electrons. 7. Development of a technique of cooling material under the beam of high-energy electrons. 8. Radiation processing of components of the composite material. 9. The analysis of parameters and selection of the type of a pressing apparatus for producing composite materials with given parameters.

The analysis and modification of the individual blocks of the installation relation to specific technological modes of pressing and sintering of the experimental samples. 10. Fabrication of the press form, working in a given range of pressure, temperature and time. 11. The oil lubrication of the pressform and filling components of the future composite material into the press- form.12. The analysis and verification of the parameters of the press installation in different intervals of temperature, time and mechanical loads. 13. Development of measurement techniques of the temperatury.14. The analysis and choice of intervals of the mechanical loadings (stresses). 15. The analysis and choice of time intervals of pressing to achieve the necessary homogeneous structures of the composite by volume of the material. 16. The trial test works, pressing and agglomeration of powder. The recess samples from the press-form and their drying in air or in a gas atmosphere at a specific temperature for a given time (from several hours to several days). 17. The mechanical treatment and manufacturing of the experimental samples of the composite of a given shape (cutting, grinding, polishing, and others.), 18. Studying the physical and mechanical properties of obtained composite and revealing the features of the material deviations from a given characteristics. 18. The semi-industrial tests and correction techniques and modes of manufacturing of the composite material (temperature, size and mechanical load). 19. Establishment of the parameters and making an experimental batch. 20. The analysis and giving specific advice. In view of developed technology the experimental samples with the following percentages of the components a) 90 and 10 %, b) 70 and 30 %, c) 50 and 50 %, were made respectively.



1 – a transformer T1; 2 – generator housing; 3 – compressor; 4 – cover protection **Figure 1.** The appearance of pulsed high-current electron accelerator AS-TRA-M SP

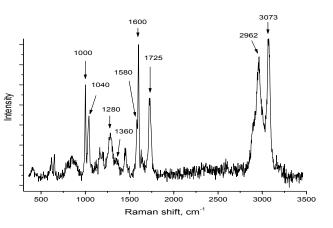


Figure 2. The Raman spectrum of the unirradiated composite on the basis of epoxy resin and shungite (90 and 10 %)

The irradiation was performed on a pulsed electron accelerator ASTRA-M (Figure 1) [5], which has the following parameters: 1. The amplitude of the accelerating voltage is 500 kV. 2. The duration the voltage pulse (on base) is 450 ns. 3. The extracted electron current is up to 1 kA. 4. The duration of the extracted electron current (on base) is 110 ns. 5. The pulse repetition frequency is from 1 to 50 pulses/sec. 6. The energy of the extracted electron beams is 20 J.

The experimental results

Studying of composites on the basis of shungite and epoxy resins was allowed us to conclude that the increase in the concentration of shungite in a composite leads to a slight darkening of the samples, while the irradiation with electrons to different integral doses (103 - 105 Gy) does not cause substantial discoloration. The series of experimental studies were carried out, including Raman spectra were measured (Figures 2, 3) on obtained composites. In the composite 1 (90 and 10 %) were found peaks at frequencies of 1000; 1040; 1280; 1360; 1580; 1600; 1725; 2962 and 3073 cm⁻¹. For the composite 2 (70 and 30 %) on the one hand there are the same bands 1000; 1040; and 1365, 1600; 1725; 2950, 3070 (with a slight shift) cm⁻¹. On the other hand the bands in 1280; 1580 cm⁻¹ were completely disappeared. These two bands were also disappeared for the sample number 3 (50 and 50 %). It was found decrease in band intensity of 1000 cm⁻¹ with increasing concentrations of the second component (several times). AFM images of the non-irradiated and irradiated (by electrons with an energy of 2 MeV) composites to various integral doses (Figure 4) were obtained. It is seen a substantial effect of the concentration of components and dose of electron irradiation on the structure and some properties of the material.

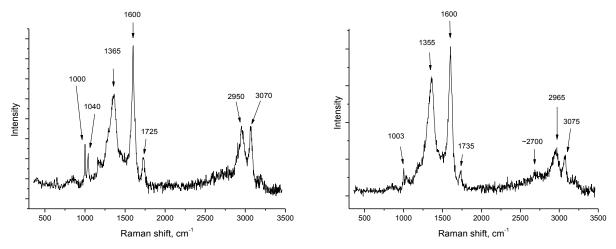


Figure 3. A Raman spectrum of the unirradiated composite based on epoxy resins and shungite (70, 30, 50 and 50 %)



Figure 4. AFM image of the composite on the basis of epoxy resin (50 %) and Kazakh shungite (50 %), irradiated by electrons with an energy of 2 MeV up to integral doses 5 (a) and 10 (b) kGy

Findings

1. The technology for manufacturing of composites based on epoxy resins and shungites with the use of electron irradiation was developed. The experimental samples with different percentages of the components were made.

2. The experimental studies of the properties and structure of composites based on epoxy resins and shungites were carried out. Raman spectra of these composites were measured. The peaks at 1,000;

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1040; 1280; 1360; 1580; 1600; 1725; 2962 and 3073 cm⁻¹ were found. For the composites (70 and 30 %) and (50 and 50 %) on the one hand there are the same bands 1000; 1040; 1365 (there is a slight shift), on the other hand the bands in 1280; 1580 cm⁻¹ were completely disappeared. It was found decrease in intensity of band 1000 cm⁻¹ and others with increasing concentrations of the second component (in several times).

3. Based on the analysis of carried out data it was concluded that the main contribution to the intensity of the bands gives the carbon OH, $CH CH_2$ bonds. With increasing irradiation dose for various concentrations of the second component Raman spectra behave differently - there is also decrease in and increase in the intensities that is connected with competing processes of rupture and cross linking of polymer bonds.

4. AFM images of the non-irradiated and irradiated (by electrons with an energy of 2 MeV) composites up to various integral doses were obtained. It was revealed a substantial effect of the concentration of components and of electron irradiation on the surface structure of the material.

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