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CONTENT

X-Ray Structural Analysis of the Irradiated Basalt Composite Microstructure

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Abstract. A study of the microstructure and elemental composition of the components of a composite material based on basalt after its gamma irradiation for the purpose of mechanical hardening was carried out. For this, a scanning electron microscope and a built-in energy-dispersive system were used. Local changes in morphology (nucleation of bubbles and cracks), as well as the elemental composition of the epoxy binder and filler (basalt fibers) were analyzed. Irradiation was carried out in the dose range: 5-15 Mrad. It has been shown that at irradiation doses up to 10 Mrad, new intermolecular bonds are formed and the material is strengthened. At high doses of irradiation, bond destruction and the formation of a gas phase are observed, which leads to weakening of the composite.

Keywords: Basalt composites, microstructure modification, gamma irradiation, SEM, local elemental composition.

1. Introduction

The widespread use of composites based on natural minerals is often limited by their insufficient strength. In the previous works of the authors [1, 2], the results of mechanical testing of samples from a basalt composite modified by gamma irradiation are presented. It is shown that irradiation can affect the elastic and strength characteristics both in the direction of improvement and in the direction of deterioration. In works [2, 3], using IR spectrometry, an attempt was made to substantiate the mechanisms of hardening and softening of the material depending on the irradiation dose (ceteris paribus, except for the diffusion or relaxation time). The main reason is believed to be the appearance of new intermolecular bonds and their destruction. In this case, the IR spectrometry method provides summary information about the molecular structure of the irradiated basalt composite as a whole. However, it is of interest to study the changes that occur under the influence of gamma rays on a microscale, both in the epoxy binder and in the filler (basalt), separately. In this work, for this purpose, we used the method of scanning electron microscopy with a built-in analyzer of the elemental composition, starting with the light elements B, C, O, N… (SEM-EDS) [4]. It allows one to study separately the local elemental composition of the organic and mineral components of the composite, as well as the boundary of their adhesion at increasing doses of radiation.

2. Literature Review

Studies related to the study of the mechanical properties of basalt composites and the use of this material for various products and structures have been the subject of considerable attention of many Russian and foreign scientists, for example [5-13]. Basalt composite has serious potential for its widest application, offering an excellent cost-quality ratio, high resistance to aggressive environments of alkalis and acids, good dielectric properties, heat resistance, radio transparency and simple manufacturing technology for various products. A simplified procedure for the production of continuous basalt fiber is as follows. First the basalt raw materials are heated to melting temperature (1500 C^0). The melt is then transported via a drain device to the platinum-rhodium spinning machines for drawing and winding on the spindles. The energy cost for the production of basalt continuous fiber (BCF) is 20 times less than that of steel and rolled metal, and its specific tensile strength significantly exceeds the same indicator for alloy steels [6]. The production of basalt fiber requires significantly less energy than the production of fiberglass or carbon fibers [13]. It should be taken into account that basalt belongs to the number of minerals widely distributed on the surface of our planet, which makes it very accessible. In fact, basalt is a mineral derived from a solidified volcanic rock that has poured onto the surface of the earth. It is important to note that basalt rocks are among the most durable natural silicate rocks. Unlike the raw materials for the production of fiberglass, for example, basalt rock is a ready-made natural raw material for the production of fibers. This allows us to have comparable production costs for BCF and fiberglass. Basalt fiber has a developed cluster surface and exhibits good sorption properties [14]. At the same time, the hygroscopic properties of basalt fiber are 6-8 times lower than those of glass fibers. This quality is certainly very important for such an industry as shipbuilding. Basalt fibers, in comparison with glass fibers, have a much wider operating temperature range. Despite the fact that BCF has significantly displaced fiberglass, it should not be considered as a direct competitor. Each of these materials has its own area of use. In addition, the idea of their hybrid use is very attractive.

The aforementioned qualities of basalt and basalt-composites allow us to speak about good prospects of their use in medicine. Primarily in the field of external fixation systems for bone fractures and injuries. Basalt fibers are used in the development of high-performance, flexible and low-cost prostheses [15]. Its use in the development of industrial exoskeletons would be reasonable. There is a successful experience in the use of basalt in dentistry [16]. Titanium and chromium alloys with the use of basalt are quite common materials for hip and knee arthroplasty. Meanwhile, the required fatigue strength and surface compatibility are still the subject of research.

Numerous active attempts of researchers to improve certain characteristics through the introduction of nanotubes into the binder, gamma irradiation, fiber milling, as well as the use of various compositions of the binder itself attract attention [17-23]. For example, in [21], the effect of modification of the material under study is achieved by microwave radiation and the introduction of carbon nanotubes into the material. In [22], crushed ochre is used as a filler. In this paper, the modification is carried out by processing the material with gamma-ray streams at different doses. The purpose of such modification is usually to achieve a high level of adhesion between the fibers and the polymer matrix. The properties of the filler, basalt fiber, are fully realised when this is achieved.

The development of hybrid polymeric materials seems very promising [24,25]. For example, the composition of carbon filaments and basalt fibers allows you to obtain strength characteristics 60% higher than those of fiberglass. In addition, such hybridization increases water resistance by 70% and significantly reduces the cost [25].

The purpose of this study is to investigate the effect of gamma radiation dose on the mechanisms of molecular destruction and intermolecular crosslinking, leading to changes in the mechanical properties and structure of basalt composite. This work is a follow-up to the previously performed works on the study of the mechanical characteristics of modified basalt composites [26]. In the current study, an attempt is made to substantiate the physical effect of gamma irradiation on the mechanical properties of basalt composites.

There are two points of view on this issue in the scientific literature. The first one (which may be conventionally called "structural") suggests that the process of "cross-linking" and the formation of a new structure characterized by new mechanical properties takes place due to the impact of gamma-quanta on the material [27]. At the same time the process of "cross-linking" is accompanied by a process of bond failure ("destruction"). As the absorbed dose from radiation exposure increases, the degradation process begins to dominate and the material gradually breaks down. As the experiments of the authors [1,26] have shown, noticeable degradation of investigated basalt-composite is evident at irradiation doses higher than 15-20 Mrad. The second point of view (conditionally, it can be called "energetic") is that due to the influence of a concentrated stream of gamma quanta, additional energy is "pumped" into the material, allowing the process of structuring the material to continue, which also leads to a change in mechanical properties. In the authors' opinion, both points of view can be used to justify the effect of gamma irradiation on material properties. However, only additional research and well-founded hypotheses will fully understand and explain the physical nature of this phenomenon.

Various methods have been used to study the structure of the material. In particular, in [28], a scanning electron microscope (SEM) was used to study the structure of a polymer composite material after its modification by introducing carbon nanotubes and processing with high-frequency radiation treatment. However, the application of this method requires high surface quality of the samples and is convenient for the material that is electrically conductive. Therefore, in this work, the method of infrared spectrometry (IR) was used to assess the level of intermolecular bonds. It has proven itself in many studies. For example, in [29], the use of spectrometry made it possible to qualitatively assess the appearance of new structural bonds under radiation exposure to the material under study. In [2], IR spectrometry is used to study the structure of the irradiated basalt-composite as a whole.

However, in these works there are no results describing the evolution of the structure of the components of the composite in the process of exposure to concentrated fluxes of gamma rays in various doses. The availability of information on changes in the composition and structure of epoxy resin and basalt at different doses of irradiation will make it possible to more convincingly substantiate the mechanisms of strengthening and softening of the material at the molecular level. And also understand the conditions of their competition. This, in turn, will make it possible to more accurately select the conditions for the radiation modification of composite materials based on basalt in order to improve their mechanical properties. Therefore, the topic of the article seems to be important and relevant for the problems of radiation materials science.

3. Data and Methodology

Samples of a composite material based on basalt, made at the Ural Research Institute of Composite Materials, were studied. The constituent parts of the composite were the epoxy binder EDT-10P and basalt roving. Their characteristics are given in [2]. Before testing, the samples were divided into four groups. The first group included comparison samples that were not subjected to the procedure of gamma irradiation, but passed all measurements under identical conditions. The ^{60}Co radioisotope was used as a source of ionizing electromagnetic radiation. It emits gamma rays with energies of 1.17 and 1.33 MeV. The remaining three groups of the basalt composite were irradiated with gamma rays in the open atmosphere at doses of 5, 10, and 15 Mrad, respectively, at a dose rate of 12.5 rad/s. The exposure time certainly depended on the planned dose and was about 14 days for 15 Mrad. The small size of the samples compared to the geometry of the gamma irradiator suggests that the irradiation was uniform [30]. It is known that under intense high-energy irradiation, the material absorbs energy and leaves the thermodynamic equilibrium state. After the cessation of irradiation, in accordance with the laws of thermodynamics, the material relaxes in the direction of lowering the internal energy to a state of equilibrium. In order to stabilize the state of the material, samples were tested three months after irradiation (5 times the exposure time). During this period, the samples were stored under normal conditions at room temperature in a dark room, to exclude photochemical processes. To ensure the surface quality required for SEM-EDS, the material was poured with an epoxy binder and the required area was finely polish (Fig. 1).

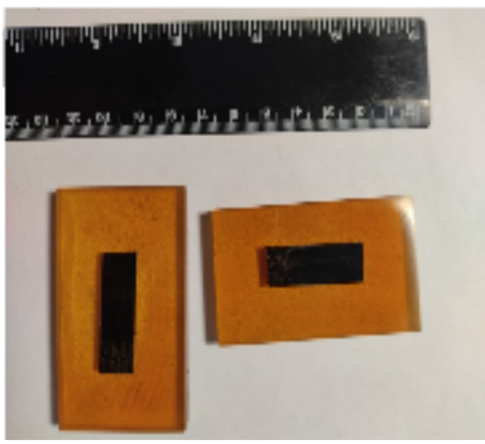

Fig.1 Samples prepared for SEM-EDS and IR spectrometry. Source: [2].

Scanning electron microscopy (SEM) and local elemental composition - SED (electron dispersion spectrum) were carried out on the polished surface of the samples (viewing area about 1 mm^2) at the Japan Technology Center of the Tashkent Polytechnic University on a JEOL JSM-IT 200 microscope. Measurement conditions: electron beam voltage - 20 kV, aperture - 3 (wide), magnification - x80, color map mode.

4. Results

Let us consider the elemental composition of some components of the composite before irradiation. The non-irradiated epoxy binder is characterized by the ratio C/O = 2/1. The composition of the non-irradiated area of basalt is characterized by the ratio C/O <½. Silicon (Si), aluminum (Al) and magnesium (Mg), which are part of basalt, usually correlate in the proportion 3/2/1. Note that, due to the different valences of the cations, the structural positions of oxygen and the bonds in them are not symmetrical.

The visible region is enriched in Na and Ca. The visible grain is aluminosilicate crystallites. A more detailed complete composition is given in tables 1 and 2. After irradiation, the elemental compositions of the epoxy binder and basalt undergo changes. Figures 2 and 3 show typical SEM images and SED spectra of the epoxy component and basalt, respectively.

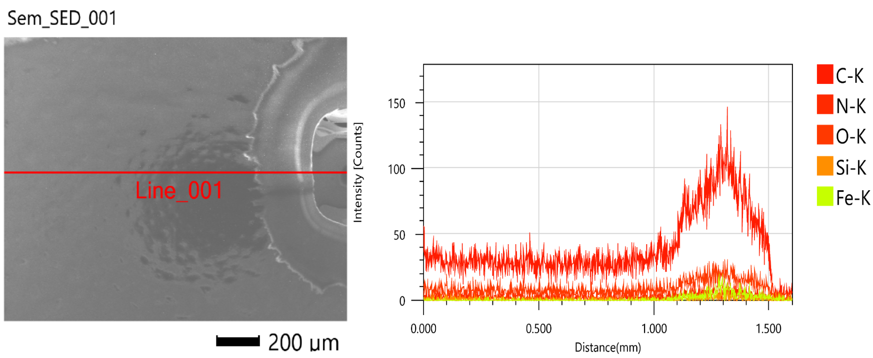

Fig. 2. Example of SEM and SED results of the epoxy component of the composite after gamma irradiation with a dose of 10 Mrad (along line 001 1.5 mm long, over which the composition was averaged. The area includes a microbubble).
Source: Authors' elaboration.

The formation of microbubbles and gas release in resins during gamma irradiation was noticed even in the first experiments due to the increase in pressure in sealed ampoules. The first assumption, that this is the result of the radiolysis of the C-H bond with the release of H2, has not been confirmed. The appearance of the C-O bond in the IR spectra was attributed to the interaction with atmospheric oxygen, but it was also refuted by irradiation in an evacuated ampoule.

The results of SED for the components of the composite were summarized in table. 1 and 2. It can be seen that after irradiation of the material with a dose of 5 Mrad, the mass fraction of carbon in the epoxy binder sharply decreases, but the fraction of oxygen increases and silicon appears. In basalt, on the contrary, the proportion of carbon increases, while that of oxygen and silicon decreases. The decrease in C and at the same time an increase in O were not detected until the appearance of spectral analyzers of light elements of the SED or EDS type.

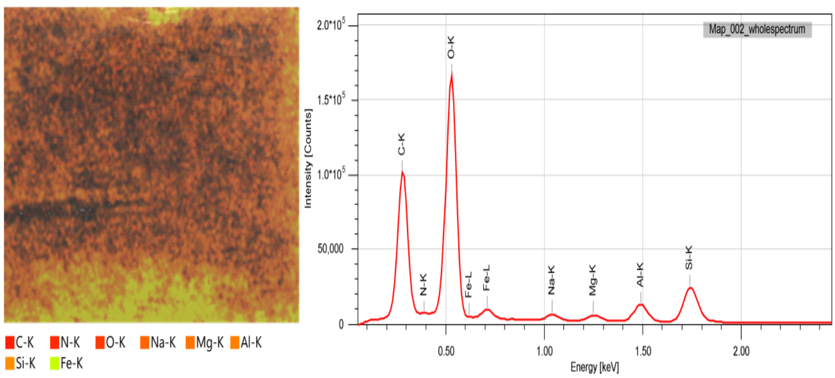

Fig. 3. An example of the results of SEM and SED of the basalt component of the composite after gamma irradiation with a dose of 10 Mrad (visible grains are aluminosilicate crystallites, micropores and a microcrack on the left are black).
Source: Authors' elaboration.

This indicates that the process of formation of new intermolecular bonds (crosslinking) occurs in the material with the formation of organo-silicate compounds –C–O–Si-. According to the authors, this is due to the fact that the silicate group of basalt is the most widespread and active, and the C–H bond in the epoxy binder breaks quite easily even under UV irradiation. Gamma rays have an energy above 1 MeV, which is certainly enough to excite any nucleus and chemical reaction. Thus, three-dimensional structures with chemically embedded basalt filler are formed, including the formation of an aluminosilicate compound Si-O-Al, which is observed during SEM (Fig. 3). These results confirm the conclusions made by the authors after studying the irradiated basalt composite using IR spectrometry [2].

Table 1. Mass fraction of epoxy binder elements before and after exposure, %

Element	Radiation dose, Mrad			
	0	5	10	15
C	82,0	68,49	63,53	70,10
O	18,0	31,39	29,98	22,60
Si	0	0,12	0,09	0,07
Al	0	0	0	0,05
Mg	0	0	0	0
Na	0	0	0	0.05
Ca	0	0	0	0
Fe	0	0	1,08	0
N	0	0	5,32	7,13

Table 2. Mass fraction of basalt elements before and after irradiation, %

Element	Radiation dose, Mrad			
	0	5	10	15
C	23,56	29,53	38,17	32,01
O	69,50	64,32	56,58	63,46
Si	4,68	3,51	2,50	2,30
Al	1,12	1,85	1,32	1,05
Mg	0,56	0,74	0,49	0,32
Na	0,24	0,3	0,78	0,56
Ca	0,36	0,06	0	0
Fe	0	0	0,17	0,15
N	0	0	0,05	0,12

With further irradiation up to 10 Mrad, the crosslinking process continues, but its rate decreases. Microbubbles are observed in the epoxy binder, which indicates an excess of irradiation energy necessary for the process of formation of new intermolecular bonds (Fig. 2).

At an irradiation dose of 15 Mrad, the reverse process (destruction) is observed, in which the destruction of intermolecular bonds and, consequently, the weakening of the composite, which was previously noted in [1], occurs.

The study revealed an interesting fact associated with the appearance in the area of a highly irradiated epoxy binder large enough to detect the amount of nitrogen and iron. The appearance of nitrogen could be explained by adsorption from the atmosphere, but this option is not acceptable for iron. It seems logical to consider the possibility of initiating nuclear reactions by ^{60}Co gamma quanta. An absorbed dose above 10 Mrad corresponds to $\sim 10^{17}$ gamma quanta/cm2, which can at best form the same number of product nuclei. The authors suggest that during gamma irradiation of the polymer CH, a hydrogen proton enters the carbon nucleus and it transmutes into the nitrogen nucleus according to the formula:

$$(^{12}C_6 + {}^{1}H_1) + \gamma\ (1.17+ 1.33\ \text{МэВ}) = {}^{13}N_7\ (9.96\ \text{min}\ \beta^+,\ \text{no}\ \gamma).$$

However, to create a stable $^{14}N_7$ isotope, the positron-active $^{13}N_7$ isotope must capture the neighboring hydrogen (proton + electron). Hydrogen, due to the annihilation of an electron with a positron, will turn into a neutron in the excited $^{14}N_7^*$ nucleus, which then relaxes to a stable state and appears in small amounts in the EDS and FTIR spectra.

Another option is possible: it is known that the nuclei of oxygen and carbon are α-cluster nuclei, where bonds between α-clusters have an energy of about 2 MeV [31]. Thus, an intense ^{60}Co gamma field can lead both to the decay of $^{12}C_6 \rightarrow 3\alpha$ and $^{16}O_8 \rightarrow 4\alpha$, and to the capture of $^{12}C_6 + \alpha \rightarrow {}^{16}O_8$. These reactions can explain the observed decrease in the content of C and the increase in O, as well as the formation of microbubbles filled with helium (He). Under conditions of high ionization in a gamma field, it is quite easy for an alpha particle to attach 2 electrons and become an He atom.

5. Conclusion

The conducted studies allow us to understand and substantiate the mechanisms of the effect of gamma rays on composite materials based on basalt. It can be asserted with a high degree of certainty that, upon gamma irradiation of the composite with doses up to 5 Mrad, an intensive process of cross-linking of molecules occurs with the formation of a three-dimensional framework and the material is strengthened. In the range from 5 to 10 Mrad, the crosslinking process continues with the simultaneous destruction of intermolecular bonds. Irradiation of the material with doses greater than 10 Mrad leads to intense destruction of intermolecular bonds, softening of the composite, formation of micropores, gas microbubbles and microcracks. Thus, gamma irradiation can be recommended as one of the methods for modifying basalt-based composite materials in order to strengthen them. However, the radiation dose should not exceed 10 Mrad, and the radiation power - 12 rad/sec.

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