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RESULTS OF THE SURFACE MORPHOLOGY STUDY OF ELASTIC SELF-ADHESIVE RADIATION SHIELDING COATINGS BY ATOMIC FORCE MICROSCOPY

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Abstract. An algorithm for studying the structure of radiation shielding materials using the atomic force microscopy (AFM) method has been developed and described. Using the proposed method, the structure of tungsten-containing radiation shielding materials was studied and the difference in the microstructure of the samples and the nature of the distribution of the filler was revealed.

Keywords: composite material, radiation shielding elastic self-adhesive coatings, linear absorption coefficient, microstructure

1. Introduction

Materials made from two or more components with different properties, which, in combination, lead to the emergence of new material with characteristics different from those of the original components, are composite materials. The peculiarity of composite materials to acquire unique combinations of various properties opens a wide field of application for them. The emergence of lightweight and durable polymeric structural materials leads to the displacement of traditional steel and aluminum in auto and aircraft construction. The creation of various radiation shielding polymeric materials leads to the displacement of heavy concrete and lead, which have been used for many years to protect against ionizing radiation [1,2,3]. Refusal to use lead protection is associated with a large mass of protective screens, the complexity of their installation and dismantling. In addition, lead is a toxic element and can cause various diseases [4].

The desire of scientists to avoid negative consequences from the use of traditional materials underlies the development of polymeric radiation shielding materials. In order to facilitate the installation and dismantling of radiation shielding materials, an elastic self-adhesive matrix was developed [5]. The second part of composite materials, in addition to the polymer matrix, are various fillers. Chemical elements with a high nuclear charge number are chosen as fillers. Composites with tungsten, barium, bismuth, etc. act as fillers absorb gamma quanta most effectively [6,7,8]. When adding fillers into the matrix, it is extremely important

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to achieve its uniform distribution in the matrix. The distribution of the filler depends on many factors: the nature of the filler, its dispersion, and the properties of the polymer matrix itself. In [9], the quality of the filler distribution is determined visually using an optical microscope. Works [8,10] describe the application of such methods as X-ray diffraction, transmission electron microscope, and spectrometer.

The filler distribution depends on its interaction with the polymer matrix. But it is worth remembering that even in its pure form, a polymer matrix has a multilevel hierarchical structure, i.e. well-pronounced structural heterogeneity at the micro-, meso- and nanoscale. The atomic force microscopy (AFM) method makes it possible to study the structure of the material at these levels. The use of atomic force microscopy to study polymer-based composites has a number of advantages over other microscopic methods, in particular, electron microscopy, since this method provides high contrast at a sufficiently high spatial resolution.

The possibility of using the new method to study the structure of polymer composite materials and their radiation resistance is limited by a small number of works in this field of investigation.

In [11], it is noted that, with the exception of the relief, the interpretation of the information obtained in the AFM study is often rather difficult. The explanation of this situation is associated both with the complexity of real objects and with the lack of systematic studies of simple systems, on which one could rely on the study of more complex ones.

The structural state of self-adhesive protective coatings has not yet been studied using AFM. In this regard, the purpose of the work is to refine the algorithm for studying REM samples using AFM.

2. The experimental procedure

Two tungsten-containing samples were studied, which differed from each other in the type of polymer matrix: sample 1 (B52T) and sample 2 (SM4B5). Linear absorption coefficient (μ) at an energy of 59 keV is 22.283 cm-1 and 23.197 cm-1, respectively [5].

Investigations of the surface morphology of the samples by atomic force microscopy method were carried out on a scanning probe microscope (SPM) "Solver Next" manufactured by NT-MDT, OJSC in Zelenograd. Surface scanning was carried out by cantilevers from NSG10 / W2C series using the tapping-mode method in the topography mode and in the phase mode in the air under normal conditions. The AFM topographic mode provides data on the surface topography; phase – about the heterogeneity of the chemical composition. The analysis of surface structure parameters was carried out on the area of 90×90 μm2; 30×30 μm2; 10×10 μm2 and 3×3 μm2. The scan results were processed by the Image Analysis P9 – image processing software module, which has great functionality, is mostly automated, and does not require the use of third-party software.

To study the surface microrelief, the Roughness Analysis method was used. To analyze the dispersion of filler particles, we used the Grain Analysis program, which allows getting a large set of quantitative data on the surface structure, such as area, volume, and size of particles/pores, their center of gravity coordinates, maximum size, maximum height, perimeter, and average values of the listed parameters. In addition, the program allows building histograms of the distribution of the selected geometric parameter by image objects.

3. Results and discussion

It is better to begin a qualitative assessment of the structure by analyzing the panoramic scan of the surface of REM samples on a scanning field of 90×90 μm. Figure 1 shows AFM images of the surface of the studied samples. The surface structure is a structureless elastic matrix (dark color in the image), in which a metal-polymer frame of varying degree of

ordering and density is placed. Survey scans revealed a difference in sample morphology. The frame in sample 1 has a rough relief and a loose globular-fibrillar structure. The filler in the polymer matrix forms limited, linearly oriented sections consisting of particle agglomerates (Fig. 1a). In sample 2, the structure of the polymer frame is represented insufficiently. The finely dispersed spherical filler and a few aggregates have a statistical distribution in a dense matrix (Fig. 1c).

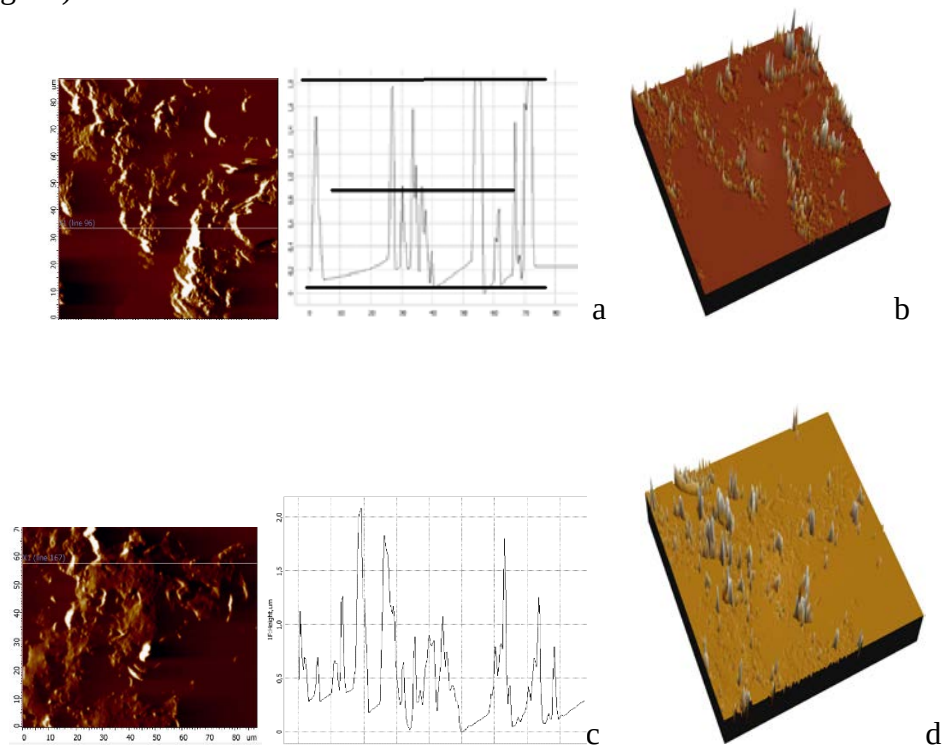

Fig. 1. AFM scan, field size 90×90 μm (a – 2D image of sample 1 with a section profile; b – 3D image of sample 1 in the phase of contrast mode; c – sample 2 with a section profile; d – 3D image of sample 2 in the mode of phase contrast)

As the analysis of the microrelief of the surface of the samples on scans of different sizes / different scale levels showed, the microrelief parameters of sample 1 are higher than those of sample 2 (Table 1).

Table 1. The value of the parameters of the microrelief

Sample	Scan size, μm	Sa, μm	h, μm	max μm
BMT	90x90	0.278	2.434	1,597
	30x30	0.212	2.405	1.671
	3x3	0.042	0.899	0.761
SM	90x90	0,220	2,400	1.726
	30x30	0,127	1,946	1.541
	3x3	0,018	0,654	0.423

To analyze macroscopic homogeneity (the quality of mixing a polymer with a filler) of composites, it is important to be able to correctly determine particle aggregates on the surface reliefs of a material. When analyzing this material, a multilevel visualization technique was

used, which consisted of representing the relief as the sum of several reliefs [11]. Analysis of the cross-sectional profile of AFM images showed that both samples have irregularities of various sizes. High peaks on the cross-sectional profile correspond, as studies in phase mode showed, to the filler; the middle level corresponds to the metal-polymer frame, and the lower one corresponds to the elastomeric matrix (Fig. 1b and 1d). The next step is to obtain quantitative characteristics of the filler.

It is known that the quality of composites largely depends on the dispersion of the filler distribution in the polymer matrix [12,13].

Table 2 shows the results of processing of AFM scans of the surface of the samples in a 30×30 μm field using the "Graine Analysis" program. At this size of the scan, both individual particles and aggregates are well resolved.

Table 2. Characteristics of filler particles

Sample number		1	2
Parametres of filler particles	Aspect ratio *	4	1.7
	Fraction of particle area under the microscope,%	2.084	2.65
	Average number of particles under the microscope, pc.	75	171
	Transverse size range boundaries, μm		
	Upper	1.27	0.63
	Lower	0.16	0.16
	Height range boundaries, μm		
	Upper	2.41	1.94
	Lower	1.02	0.33

*aspect ratio characterizes the size of aggregates - the ratio of the maximum size to the effective width

Analysis of the data in Table 2 showed that the filler in sample 2 is more dispersed. The samples have a different number of agglomerates (75 and 171 pieces under the microscope), and the area occupied by the filler is comparable. According to this study, the filler in sample 1 is dispersed slightly worse than the filler in sample 2.

For a detailed study of the supramolecular structure, we investigated the surface of the samples on a scanning field of 3×3 μm (Fig. 2). By reducing the scan size, one can observe the thin structure of the material. So, in Fig. 2a, one can clearly distinguish the domain structure of the surface of sample 1. In the phase image, the location of the filler on the periphery of the structural formations in the form of extended aggregates is clearly visible. The structure of the metal-polymer frame of sample 2 is fundamentally different. The finely dispersed filler is randomly distributed in a dense, poorly structured matrix (Fig. 2b).

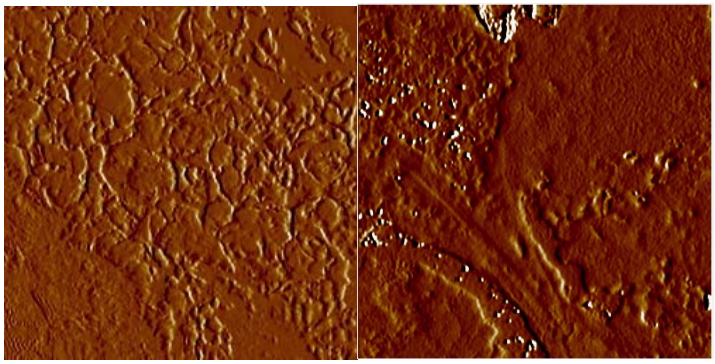

Fig. 2. AFM scan, size 3×3 μm, phase contrast: a – sample 1; b – sample 2

Thus, the AFM method allows obtaining information at a qualitatively new level and is very useful in the structural characterization of radiation shielding polymer-based composites.

4. Conclusion

The AFM algorithm has been developed - studies of samples of self-adhesive radiation shielding coatings using multilevel imaging techniques.

As a result, high-quality AFM scans of the surface of REM samples at different scale levels were obtained.

A formalized description of the AFM image is given. The difference in the microstructure of the samples and the nature of the filler distribution was revealed.

Some standard programs from the Image Analysis P9 package have been tested. The characteristics of the microrelief, filler dispersion; the nature of its distribution in REM samples are determined.

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