Research of Structure of Elastic Self-Adhesive Radiation Shielding Coatings

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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 were revealed.

1. Introduction

The use of polymeric materials and composites based on them for protection against various types of radiation has gained widespread popularity in recent years. The materials studied in this paper differ from traditional polymer radiation shielding composites. To create such materials, an elastic self-adhesive matrix was developed, which allows to make fast installation and dismantling of radiation shielding coatings [1].

The technologies for creating X-ray protective polymer-matrix composites are based on adding particles of materials with high absorption coefficients of gamma rays (Pb, W and their compounds) into the polymer matrix [2, 3]. In this case, it is important to have an equal distribution of the filler in the polymer matrix, which depends on the nature of the filler, dispersion and filler-matrix bond. Even in pure form, the polymer matrix has a multi-level hierarchical structure, i.e. well-defined structural heterogeneity at the micro-, meso- and nanoscale. Investigation of the structure at these scale levels allows to conduct atomic force microscopy (AFM) method. The use of atomic force microscopy to study polymer-based composites has several advantages over other microscopic methods, in particular electron microscopy, because this method provides high contrast with a sufficiently high spatial resolution.

However, despite the opportunity to study materials at a qualitatively new level, there are very few studies on the structure of polymer-based composites and their radiation resistance using AFM.

In [4], it is noted that, with the exception of the relief, the interpretation obtained by the AFM study of information is often quite 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 that could be relied upon in the study of complex ones.
Studies of the structural state of protective self-adhesive coatings with the use of AFM have not been conducted yet. In this regard, the purpose of the work is to develop an algorithm for studying REM (radiation shielding materials) 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 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 μm\(^2\); 30×30 μm\(^2\); 10×10 μm\(^2\) and 3×3 μm\(^2\). The scan results were processed by the Image Analysis P9 – image processing software module, which has great functionality, is most 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 to get 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 to build 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 90x90 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).
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 in representing the relief as the sum of several reliefs [4]. 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 [6,7].

Table 2 shows the results of processing of AFM scans of the surface of the samples in a 30x30 μm field using the “Graine Analysis” program. At this size of the scan, both individual particles and aggregates are well resolved.

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.
Table 2. Characteristics of filler particles.

| Sample number | 1 | 2 |
|---------------|---|---|
| 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

For a detailed study of the supramolecular structure, we investigated the surface of the samples on a scanning field of 3 x 3 μm (Figure 2). By reducing the scan size, one can observe the thin structure of the material. So, in Figure 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).

![Figure 2. AFM scan, size 3x3 μm, phase contrast: a - sample 1; b - sample 2.](image)

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 is determined.

5. References

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