The automating of the quantitative analysis and characterization of the polymer based films surfaces SEM-images

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Abstract. It is represented a possible way to standardize and to automate the quantitative description and the analysis of the formed by the scanning electron microscope polymer based films surfaces’ images. Also there are the calculating algorithms for the topographic model forming and the degrees of planar and contour digital heterogeneities calculating and the analysis process model with the computer application interface and the corresponded results of the sulfonated and fluorinated low-density polyethylene films SEM-images’ characterization. Due to the universality of the proposed techniques for quantitative image description and simulating they can be used in quality control systems under the design and manufacture of the surface-modified polymer films.

1. Introduction

One of the most promising areas of the chemistry and materials science is the creation and the study of new high-molecular compounds and polymer based composite materials [1]. In some cases, the polymer based products do not yield to their metal or ceramic prototypes in a number of operational and functional characteristics [2]. At the same time, the development and the production of new high-molecular compounds based materials are usually characterized by significantly lower resource consumption. And their functional properties can be adjusted using various modification techniques, in fact, up to the start of finished products operations [3]. The research and control of physical, chemical and, as a result, functional characteristics of synthesized new and modified polymer materials are necessary for the rational decision-making [4] in the possibility of the ceramic or metal products replacing with the polymer and/or polymer-composite ones [5]. The need to confirm environmental safety and the possibility of using the latter in various extreme conditions is also taking into account [6]. The polymer films are widely used in medicine, food industry, agriculture, construction, and other industries as insulating, facing, packaging and other materials [7]. The main advantage of polymer films is a combination of hydro- and heat-insulating properties, chemical and biological resistance, high relative mechanical strength and flexibility, etc. The major ones disadvantage is the need for artificial recycling and/or utilisation of the polymers at the end of their service life, which is associated with increased environmental risks [8]. Recently, polymer biodegradation techniques are considered as one of the promising ways to overcome this problem. The chemical and
biological resistance of polymer made products in general and polymer films in particular is the consequence of their surfaces layers properties: the general mechanism of biodestruction is the formation and settlement of a microcracks grid by biological agents (Algae and Mycota), the growth of colonies and waste products of which simultaneously create a polygradient protruding pressure and change the chemical composition of the surface, contributing to its gradual destruction under the oxygen action. The film material is completely recycled when it breaks down into sub-micro-sized fragments that can be transported by water and captured by organisms of bacteria, fungi, algae and plants before the final assimilation and/or decomposition into material-forming chemical elements [11]. Since the first step in the polymer biochemical decomposition is the adhesion of potentially effective biodestructors to its surface, the study of surface morphologies of polymer and composite materials films will contribute to solving the problem of their environmentally safe processing and/or disposal in the future. The increase in chemical resistance, reducing gas and vapor permeability, improving the adhesion and tribological characteristics of polymer films are also largely determined by changes in the microrelief and chemical composition of their surface layers due to surface and volume modification [12]. Thus, when conducting the systematic experimental studies of the structure and physics-chemical properties of polymer and composite materials, a comprehensive multi-factor analysis of the measured parameters is necessary.

2. Problem statement
It is known that the chemical composition, structure, micro- and nanotexture of the surface and near-surface layers determine the vast majority of functional properties of composite materials formed by surface modification methods on a flexible polymer basis. At the same time, a comparative analysis of the chemical compositions and surface reliefs of polymer films under the influence of biological (bacteria, micromycetes, algae) and physical (mechanical deformation, thermal and electrical treatment) factors shows that with virtually constant chemical composition, the nanotexture and microrelief of experimental samples undergo significant changes. Thus, to solve the problem of the polymer films biodegradability increasing, more useful results should be expected from the microrelief and nanotexture research methods employment. The images formed by scanning microscopy methods allow to visualize and quantitatively analyze the understudying surface structures of the experimental samples. For this purpose, it is possible to employ the stochastic and/or deterministic image analysis techniques which used in solving the certain problems of image recognition and automating processing
of video information streams with ”machine vision” approaches [13]. The decision of the quantitative description of the modified polymer film surface’s micro-relief problem makes it possible to form a map of local values of free surface energy, which allows to calculate the values of the corresponding adhesive, tribological and micromechanical characteristics of new polymer based material. As a part of the system approach to the data processing and analysis, the specialized technologies for data collecting, storage and pre-, primary and initial dataprocessing are required. The most suitable for these purposes are the basic concepts of database theory and, in particular, the recently widely known information (“big data analysis”) technologies, some elements of which are presented in [14] and [15], respectively. The problem is that the subject area and the technical capabilities of the experimental equipment impose quite severe restrictions on the corresponding data model and logical structure of the problem-oriented database. The goals of this article are the development of the approach to the data model formation in field

Figure 2. The block-diagrams of the algorithms cores for calculating a) amplitudes of morphological spectra (topographic characterization) and b) degrees of planar and contour digital heterogeneities (tomographic characterization) of the polymer films’ surfaces SEM-images.
of high-molecular compounds chemistry and materials science and providing an example of the created problem-oriented database application and data processing algorithms. The original computer application’s interface (in graphical form) and the process model used by authors for the analysis and the characterization of polymer films surfaces’ SEM-images will be presented too.

![Image](image.png)

**Figure 3.** The digital models (a, b) and the morphological spectra (c, d) of the samples surfaces’ SEM-images for the sulfonated (a, c) and fluorinated (b, d) LDPE films. The values of image pixel brightness are plotted along the applicate axes. The biharmonic indices of two-dimensional Fourier series expansions are on the abscissa and the ordinate axes (all values are dimensionless).

3. **Methods and materials**

There are several ways to analyze and control the morphological characteristics of polymer films (IR Fourier spectroscopy (IRFS), X-ray photoelectron spectrometry (XPS), scanning electron microscopy (SEM), atomic force microscopy (AFM) and others) [16]. Apparently, the most complete information about the free energy of the surface which determines its’ adhesive properties can be provided by scanning electron microscopy (SEM) in the secondary electron beam scattering (SEBS) registration mode under the different observation scales. When interacting with the sample, the primary beam electrons ionize the surface atoms to a depth of
10 nm, so that the secondary electrons with an energy of 50 eV are scattered directly above the surface in the detection zone and are registered even with small voltages. The result signal is interpreted as an effective microrelief of a chemically homogeneous surface compared with a real chemically heterogeneous sample surface with a resolution of up to 5 nm. Thus, SEM in SEBS registration mode allows experimentally investigating the free surface energy of a sample as a homogeneous material and not as a nanodispersed mixture of molecular aggregates of material-forming chemical elements. The SEM-images of the initial, gas-phase sulfonated and fluorinated low-density polyethylene films (LDPE) are performed in Figure 1.

![Image](image.png)

**Figure 4.** The functional dependencies of planar (a, b) and contour (c, d) digital heterogeneities of the sulfonated (a, c) and fluorinated (b, d) LDPE films’ SEM images.

The physical and chemical properties of the surfaces for the initial, fluorinated and sulfonated polyethylene differ significantly from each other [3,8–10,12]. The quantitative description of the obvious visual differences between the experimental samples surfaces’ SEM-images cannot be limited to the values of their average roughness (0.06±0.04, 0.28±0.05 and 0.26±0.03 microns, respectively), since the last two coincide within the statistical error. Thus, in order to enable quantitative structural and functional modeling, it is necessary to use more "suitable" tools for the microrelief characterizing than the average value of deviations from a certain microrelief level. Within the framework [17–19] of a unified quantitative deterministic description of surface morphologies for a set of polymer and composite materials’ films obtained as a result of gas-phase modification, the algorithms for topographical (Figure 2a) (amplitudes of morphological spectra) and tomographic (Figure 2b) characterization of the corresponding SEM-images were developed and software implemented.

The topographical approach assumes the possibility of forming a smooth model surface
describing sample’s micro-relief which is a superposition of any basic functions of two variables, for each of which the structure-functional modeling and the surface shape influence for the material properties evaluation are representable in an analytical form [17, 18]. In this paper the basic functions are the two-dimensional Fourier series biharmonics. When using the topographic approach for the digital SEM-images analyzing the analytical models were obtained as the superpositions of 16×16 biharmonics of the corresponding two-dimensional Fourier series expansions. The digital model and the obtained morphological spectra amplitudes are shown in Figure 3. It’s clearly that the morphological spectra are highly sensitive to the peculiarities of the microrelief of the analyzed surfaces which should allow the effective structural and functional modeling in the future.

Figure 5. The IDEF0 graphical model of the polymer films surfaces’ SEM-images analysis and characterization process.

The tomographic approach to the SEM-image analysing suggests the possibility of a step-by-step review and characterization of the individual equipotential surfaces, corresponding to equal image pixels’ brightness levels, and, as a result, to the same local values of free surface energy. This technique allows the subsequent visualization of some characteristic dependences calculated from image pixels brightness values. In this case, the parameters that characterize the equipotential level of the surface are the degrees of planar and contour digital heterogeneities of the corresponding images. They are defined respectively as the ratio of the total area and the total perimeter of the plane shapes bounded with the equipotential lines to the total area and to the total perimeter of the analyzed images entirely [19]. Under the tomographic approach
it is possible to correspond the planar and contour digital heterogeneities functions to each of the concerned images. The appropriate functional dependencies are shown in Figure 4. It can be seen that the digital heterogeneity functions also differ sufficiently from each other so that they can be used in the future when forming structural and functional models of new polymer films based materials as well. These or similar functions (formed, for example, under the optical images analysis) are more convenient to use for solving problems of operational diagnostics and control, since the implementation of the corresponding algorithms requires less time and computational resources than simulating with two-dimensional Fourier series expansions.

4. Results
When automating the polymer films surfaces’ SEM-images analysing and characterization, the triad model of the form “conversion = ï¿½ formatting = ï¿½ modeling” (visualized in the Figure 5) has proven itself well. The model input receives an image generated by a SEM, which is transformed into a pixels’ brightness table. The necessary information is extracted from the resulting spreadsheet and applied to create the text and the table files in formats that allows using the developed algorithms of data analysing. The results of calculations are saved to the hard disk as the xlsx files containing the 3D-diagrams. The interface of the computer application [20] that implements the automated experimental samples surfaces SEM-images characterization with the developed algorithms is shown in Figure 6.

Conclusion
The interface of the developed computer application and the process model of the characterization procedure for the polymer based films surfaces’ SEM-images analysing are presented. The algorithms of the topographic model forming and the degrees of planar and contour digital heterogeneities calculating for the gas-phase modified (fluorinated and sulfonated) polymer films surfaces’ SEM-images automating analysing have been developed and software implemented. The obtained results make it possible to automate the procedures of the experimental samples surfaces’ SEM-images analyzing. Due to the universality of the proposed techniques for quantitative image description and simulating they can be used in quality control systems under the design and manufacture of surface-modified polymer films processes.
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