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The possibilities of using optical coherence tomography as a method for detecting defects in polymer composites

I. V. Zlobina^{1*}, M. V. Alonova¹, N. V. Bekrenev¹, D. A. Zimnyakov^{1,2}, A. V. Pasechnaya¹,
and V. O. Nefedov¹

¹ Yuri Gagarin Saratov State Technical University, Saratov, 410054 Russia

* E-mail: irinka_7@mail.ru

² Institute of Precision Mechanics and Control Problems of the Russian Academy of Sciences,
Saratov, 410028 Russia

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Research has been carried out in the field of using optical coherence tomography to detect defects at the "matrix-fiber" interface in polymer composite materials based on continuous carbon fiber and superconstructive thermoplastic polyesteresterketone. Defective areas were recorded on the received B-scans. Binarization of various degrees has been carried out. The percentage of binarization has been identified, providing visual information content while reducing artifacts and noise typical of B-scans. To form a graphical expression and further quantify the results obtained from binarized B-scans, A-scans were generated. It was found that the binarization of B-scans at the level of 40% makes it possible to informatively display the polymer layer, determine its thickness, reduce the appearance and evaluate the continuity at the polymer-filler boundary.

Keywords: optical coherence tomography, polymer composite materials, binarization, structural defects, matrix-fiber boundary.

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Introduction

Various defects can take place during the production of fibrous PCMs, including surface damage caused by contact with the guide rollers, as well as problems associated with static electricity and uneven coating [1, 2]. Other important defects include issues related to nanoparticle aggregation at the filler–matrix interface, caused by differences in the chemical nature and surface properties of the fibers and the matrix, as well as nonregular distribution of fibers within the matrix [3]. They directly affect the finite properties of the product. Particle aggregation makes it difficult to fully impregnate

the fiber with the binder, resulting in areas with reduced strength [3]. The uneven distribution of fibers in the matrix volume creates local stress concentrations, thereby causing a decrease in strength and toughness, as well as an increased tendency to delamination. The consequence of these defects is a reduction in operational performance under load. Even materials with high strength indices, such as carbon fiber-reinforced plastics, are critically sensitive to this factor due to their low toughness [3, 4].

Due to the fact that the structure of the PCM is complicated, and the materials themselves are multicomponent, it is relevant to control the parts made from them, not only at the

stage of production and release, but also during operation. The presence of defects of various origins, sizes, and localization have a significant impact on the formation of PCM complex of physical and mechanical characteristics [5].

Currently, diagnostic methods and the search for ways to increase their informative value are actively developing. The latter is ensured by increasing the sensitivity of the relevant elements of the research equipment, using confirmed dependencies used for signal processing, and algorithms for information analysis.

One of the promising methods for diagnosing defects in the form of air inclusions (pores, cracks) in polymer composite materials (PCM) is optical coherence reflectometry (OCR). It should be noted that the long-term experience of using this method in relation to biological objects is widely used in medicine [6-10]. Due to the micrometer resolution and the ability to perform cross-sectional imaging in the volume of the material, the OCR provides unique data on the morphology of internal structures, the distribution of reinforcing elements, flaw detection on the subsurface level and the quality of adhesive compounds [8].

The main research vector on using OCR for PCM is the transition from qualitative imaging to quantitative, high-speed analysis and prediction of properties, as well as to a non-invasive method of CM research. Thus, in [11-16], OCR was used to examine an epoxy mixture made of fiberglass, which is usually used to manufacture the spars of wind turbine blades. Cracks and delamination sites were detected through OCR images obtained during static testing of a delaminated fiberglass composite material.

The OCR fundamental principle is based on Michelson interferometry using a low-coherence light source, which is separated from the source by a beam splitter into reference and test beams [12]. The key parameter for the PCM analysis is the scattering capacity. Different components of such materials (polymer matrix, glass/carbon fibers, interfaces) have different refractive indices, which leads to partial backscattering of light. By recording the amplitude and time delay of this signal, the OCR system builds a one-dimensional A-scan. Sequential scanning in two coordinates allows you to reconstruct a two-

dimensional B-scan image (cross-section) or a three-dimensional volume.

Currently, there is very little literature describing the application of this method to a wide range of polymer composite materials (PCMs), which highlights the relevance of this research direction. In this regard, it is relevant to determine the capabilities of OCR in relation to PCM made by fuse deposition modeling of carbon fibers and thermoplastics, which is due to the developed morphology of the filler surface and the short period of increased viscosity of the thermoplastic melt, in particular polyesteresterketone (PEEK).

Materials and methods

In this paper, we studied samples of a monolayer formed on an Anisoprint Composer A4 3D printer from a prepreg reinforced with a bundle of continuous carbon fibers, impregnated with ED-20 epoxy resin and coated with polyesteresterketone (PEEK). The samples top layer was represented by a PEEK layer. Below it was a thin layer of thermosetting polymer (ED-20 epoxy resin cured with polyethylene polyamine (PEPA) hardener), with carbon fibers located in the central part. The structural features of the composite material used are described in papers [17].

For the study, two groups of samples with dimensions of 30 × 10 × 1.2 mm were used – control and experimental, subjected to electrophysical exposure, in particular, those that underwent microwave treatment at a frequency of 2450 MHz. The samples were processed in a microwave electromagnetic field at laboratory microwave processing plants manufactured by SPE AgroEcoTech LLC (Obninsk, Kaluga Region).

An OSC 1300 SS optical coherence tomograph (ThorLabs, USA) with a probe wavelength of 1300 nm, a maximum probe depth of 3 mm, and a scan track length of 5 mm with an image size of 512 by 720 pixels in B-scan mode was used for instrumental implementation of the low-coherence reflectometry method.

After obtaining a set of experimental data, B-scans and A-scans were analyzed.

Results and discussion

The analysis of the obtained B-scan images makes it possible to identify the characteristic features of the modified control samples (Fig. 1). Carbon fiber fibers absorb the probing radiation to a large extent. The upper layer of the material under study, represented by the polymer, is permeable to it, which makes it possible to use OCR to control the state of the upper layer of the polymer and region at the polymer–matrix interface.

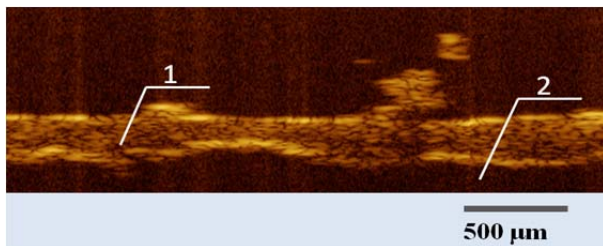


Fig. 1. B-scan of the control sample: 1 – matrix, 2 – fiber

It has been noted that for control samples, there is a clearly expressed region of refractive index step at the "polymer-fiber" interface, which is visually displayed as a bright horizontal line. In the treated samples, the regions with a refractive index step are significantly less extensive.

The analysis of the B-scans revealed a similar pattern of bright lines during the transition of probing radiation across the "air-polymer" and "matrix-filler" interfaces, which determined the expediency of searching for objective methods for their comparison.

The binarization of the generated B-scans was carried out as a quantitative assessment of the results obtained (Fig. 2).

The maximum pixel intensity of the image was considered and a boundary condition was introduced. Pixels with a lower intensity were

assigned a value of zero, while those with a higher intensity were assigned a value of one. The resulting images have greater contrast, highlight areas with the desired response values, and display regions with sharp step in the refractive index. The system of equations (1) has been formed

$$\begin{cases} I_{\text{bin}}(i, j) = 1, & \text{if } I(i, j) \geq I_b, \\ I_{\text{bin}}(i, j) = 0, & \text{if } I(i, j) < I_b, \end{cases} \quad (1)$$

where $I(i, j)$ is the pixel intensity of the original B-scan, $I_{\text{bin}}(i, j)$ is the binarized value of the B-scan intensity, and I_b is the binarization boundary condition.

The boundary condition for binarization is determined by a fraction of the maximum intensity I_{max} of the initial B-scan:

$$I_b = K \times I_{\text{max}}, \quad (2)$$

where K is the binarization coefficient.

Low values of the boundary conditions – $K = 30\%$ binarization – preserve information about the inhomogeneities formed in the B-scan in the polymer layer (Fig. 2), and high values – $K = 50\%$ binarization – leave only the brightest areas of the boundaries and do not allow obtaining information about the polymer layer (Fig. 2). The intermediate variants, respectively, leave part of the heterogeneity in the polymer layer.

The initial and binarized A-scans were also evaluated. The evaluation of a single A-scan along a vertical line is also carried out in accordance with the boundary conditions of binarization, which makes it possible to identify the boundaries and distribution of signal intensity in the polymer volume.

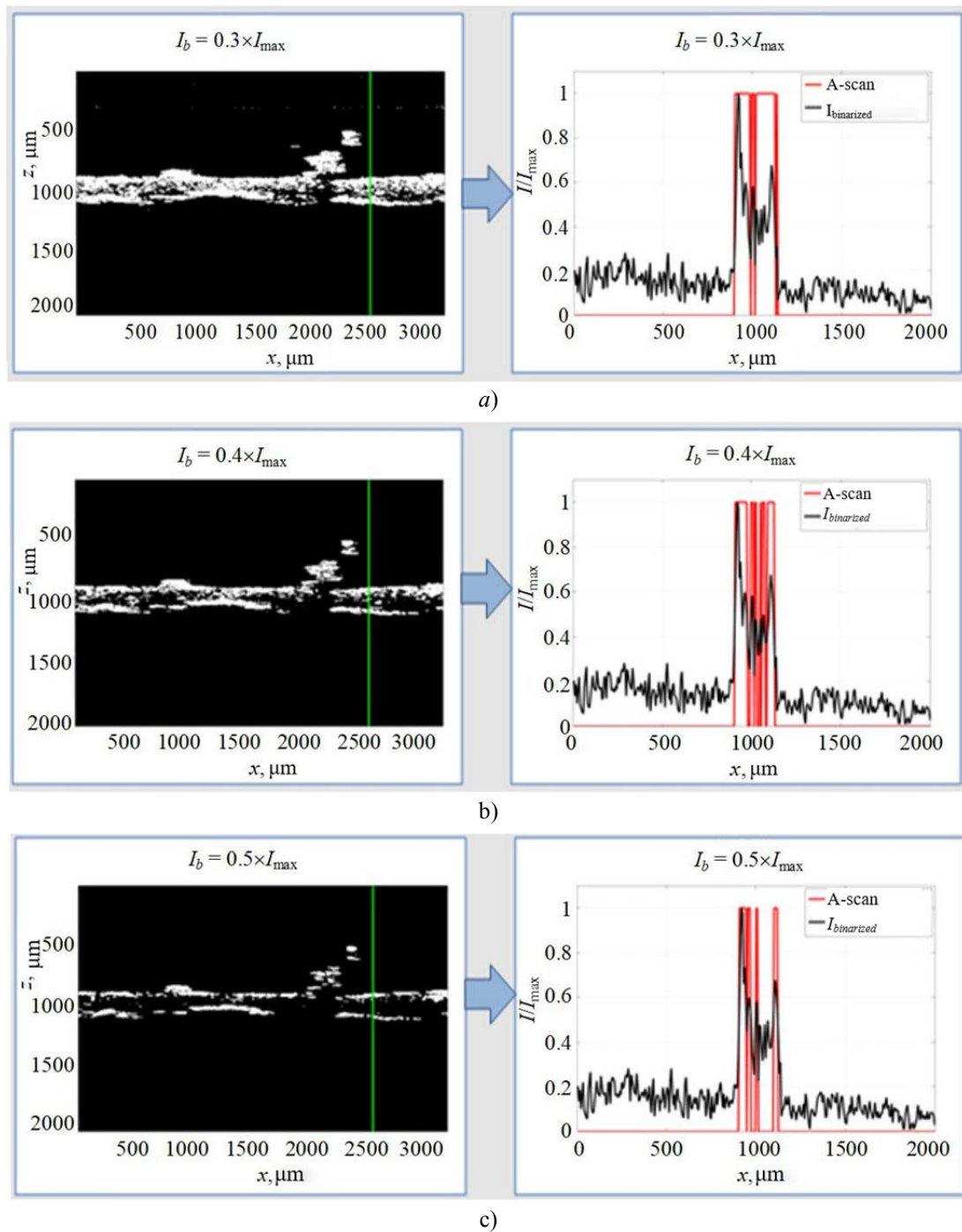


Fig. 2. The results of binarization of B-scans (on the left) and their corresponding A-scans (on the right). Binarization of 30% (a), 40% (b) and 50% (c)

To obtain a graphical representation enabling quantitative assessment of the results from binarized B-scans, A-scans were allocated.

The most informative is the A-scan corresponding to the B-scan, which is binarized by 40%. The polymer boundaries and the contrasting area of the polymer-fiber boundary are clearly shown. The condition for this binarization was to decrease the intensity by a factor of e relative to the maximum value. The

average binarization condition value of approximately 40% of the maximum intensity provides an informative display of the matrix-fiber boundary (without loss of information resulting from $I_b = 0.5 \times I_{\max}$, and without unnecessary noise, which persists at a low binarization threshold).

A high value of the response peak at the polymer-fiber interface characterizes the refractive index step of a substance reaching

values close to the refractive index of air. Taking into account the manufacturing technology of such composite materials and the specifics of the surface structure of carbon fibers, it can be concluded that part of the contact area between the matrix and the filler contains air, which partially dissolves in the polymer under modifying action on the samples, which is consistent with the results of a comparative analysis of the physico-mechanical characteristics of samples of a similar composition [17, 18].

Conclusion

Binarization of B-scans with a low threshold makes it possible to informatively display the polymer layer, determine the thickness of the polymer layer and estimate its distribution over the surface of the studied PCM.

Binarization of B-scans with a high threshold makes it possible to identify areas with a refractive index step, and identify defects in the polymer and carbon fiber connection.

An intermediate binarization level preserves information about the polymer layer thickness and the presence of contrast fragments at the "matrix–fiber" interface, enabling a comprehensive assessment of these parameters.

Considering the signal intensity distribution with exponential decay, the binarization boundary condition is proposed to be set at the intensity level reduced by a factor of e (Euler's number) from the maximum value.

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About authors

Zlobina Irina Vladimirovna, Candidate of Technical Sciences, Associate Professor, Yuri Gagarin Saratov State Technical University (77 Politechnicheskaya st., Saratov, 410054, Russia). E-mail: irinka_7_@mail.ru SPIN code: 6730-8099, AuthorID: 686083

Alonova Marina Vasilyevna, Cand. Sc. Physics and Mathematics, Associate Professor, Yuri Gagarin Saratov State Technical University (77 Politechnicheskaya st., Saratov, 410054, Russia). E-mail: alonova_marina@mail.ru SPIN code: 3059-2174, AuthorID: 667431

Bekrenev Nikolai Valeryevich, D.Sc. in Engineering, Professor, Yuri Gagarin Saratov State Technical University (77 Politechnicheskaya st., Saratov, 410054, Russia). E-mail: nikolaj.bekrenev@yandex.ru SPIN code: 8948-8116, AuthorID: 271817

Zimnyakov Dmitry Alexandrovich, Doctor of Sciences, Professor, Head of the Chair of Physics, Yuri Gagarin Saratov State Technical University (77 Politechnicheskaya st., Saratov, 410054, Russia).; Institute of Precision Mechanics and Control Problems of the Russian Academy of Sciences (24 Rabochaya st., Saratov, 410028, Russia). E-mail: zimnykov@mail.ru SPIN code: 1918-5220, AuthorID: 29094

Pasechnaya Alina Vladimirovna, student of the Chair of Technical Mechanics and Mechatronics, Yuri Gagarin Saratov State Technical University (77 Politechnicheskaya st., Saratov, 410054, Russia). E-mail: pasechnaya_alina_15.07.03@mail.ru

Nefedov Vladislav Olegovich, student of the Chair of Technical Mechanics and Mechatronics, Yuri Gagarin Saratov State Technical University (77 Politechnicheskaya st., Saratov, 410054, Russia). E-mail: b640018968@gmail.com SPIN code: 4115-8463, AuthorID: 1334364