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BWO based imaging for control of MWCNTs polymer composites homogeneity

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Abstract — **Terahertz imaging system based on backward wave oscillator for the defectoscopy of composites in the industry is presented. At a frequency of 874 GHz images of multiwall carbon nanotube agglomerates in a composite are obtained. The possibility of detecting inhomogeneities using level filtering is shown.**

I. INTRODUCTION

Medical prostheses[1], microelectronics[2], gas storage, heavy-duty filaments are increasingly using composites with the addition of carbon nanotubes in their composition. At the production of these composite materials, it is necessary to control their quality. One of the basic criteria of the quality of materials is their homogeneity. Currently, the problem of controlling the parameters of composite materials based on multiwall carbon nanotubes (MWCNTs) is an urgent problem. [3]. It is of interest to use terahertz (THz) radiation to analyze the homogeneity of composites during production.

II. RESULTS

To control the homogeneity of manufactured materials, the author's system of THz diagnostics of material heterogeneity [4], integrated into the quasi-optical path of the backward wave oscillator (BWO) THz spectrometer, was applied (Fig. 1).

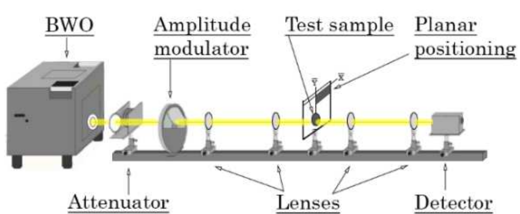

Fig. 1. Schematic diagram of the diagnostic system based on the BWO THz spectrometer.

The experimental sample was located near the diaphragm of the positioning mechanism of the THz diagnostic system. Then it was moved relative to the beam of continuous radiation generated by the BWO. An optoacoustic detector (Golay cell) registered the radiation power transmitted through the sample. In a two-dimensional plane using a point-by-point method with a fixed step the sample was moved. As a result of imaging the intensity distribution of THz radiation transmitted through the sample was registered.

The technology of producing composite samples based on MWCNTs consists of several stages. First of all, 1 mass % MWCNTs was added to the epoxy resin, then the mixture was mixed for 5 minutes with an ultrasonic treatment at a frequency of 28 kHz and a power of 75 Watts. The mixture was then polymerized for 48 hours at room temperature. After polymerization, the sample was machined to achieve plane-parallelism. Four samples were manufactured for the research. Sample No.1 was not subjected to ultrasonic treatment, in turn samples No.2, No.3 and No.4 were exposed to ultrasonic for 1, 2 and 5 minutes, respectively.

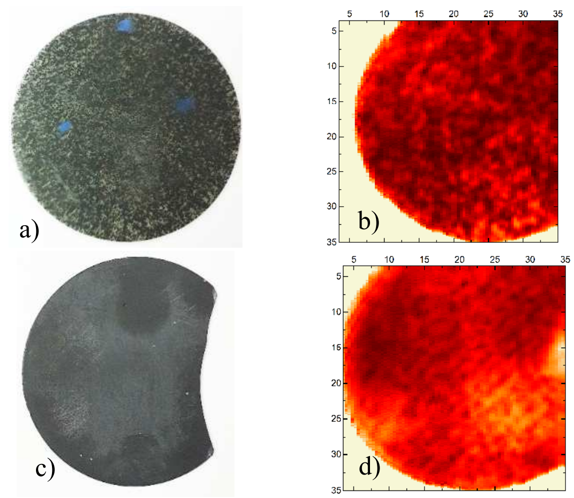

Fig. 2. Optical image of MWCNTs samples and THz image: a, b – sample No.1 (without ultrasonic treatment); d, c – sample No.4 (with ultrasonic treatment during 5 minutes).

Imaging of distribution of radiation transmitted through the samples at a frequency of 874 GHz was obtained. For sample No.1 without ultrasonic treatment the image is shown in Fig. 2b, and for sample No.4 treated by ultrasonic during 5 minutes the image is shown in Fig. 2d. The results of the experiment were normalized to the maximum value (transmitted radiation in free space).

To localize the MWCNTs agglomerates on THz picture intensity filtering algorithm was applied. Figures 3 and 4 show THz images filtered by the level of electromagnetic radiation of composites.

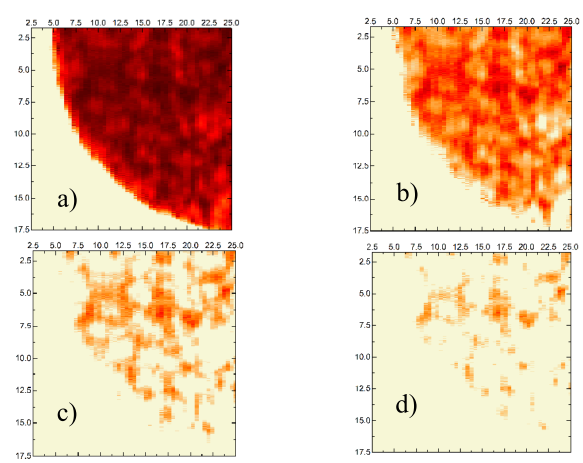

Fig. 3. Filtering by radiation intensity levels of sample No.1 without ultrasonic. a) to 0.17, b) to 0.04, c) to 0.02, d) to 0.015.

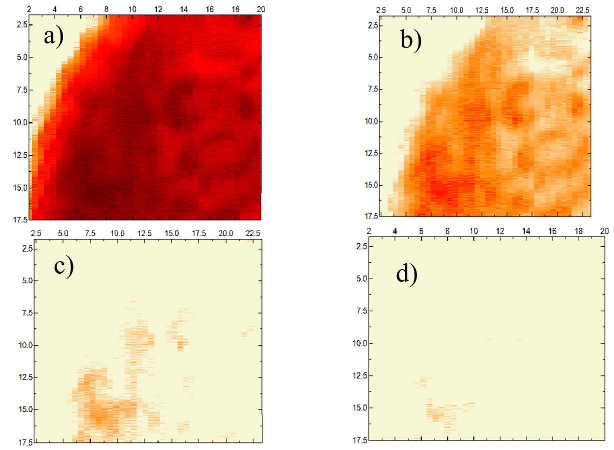

Fig. 4. Filtering by radiation intensity levels of sample No. 4 treated with ultrasonic for 5 minutes: a) to 0.17, b) to 0.04, c) to 0.02, d) to 0.015.

To assess the degree of homogeneity, the method of estimating the areas of inhomogeneities is used. The essence of the estimation method is to calculate the average voltage transmitted by radiation through the sample and recorded using the detector. In the future, the localization of the sample areas in which the electromagnetic response differs from the average value by 30% is performed. After that, the ratio of the localized areas to the area under consideration is calculated.

The average value of the voltage as the arithmetic mean (1) of all the values of the amplitude of the THz radiation transmitted through the sample is calculated. We introduce conditions (2) under which the values will correspond to regions of inhomogeneity, i.e. outside the homogeneous areas.

$$\overline{U} = \frac{1}{m \times n} \sum_{x=1}^{m} \sum_{y=1}^{n} U_{xy} \qquad (1)$$

$$\sum_{x=1}^{s} \sum_{y=1}^{t} U_{xy} = \begin{cases} U_{xy} < 0.7\overline{U} \\ U_{xy} > 1.3\overline{U} \end{cases} \qquad (2)$$

Where m and n are the dimensions of the sample area under consideration, s and t are the dimension of the inhomogeneous sample area, x and y are coordinates.

As a result, we obtain the area of the inhomogeneity regions $\Psi_{in\hom}(s,t)$. Hence, knowing the area of the sample area under consideration $\Psi(m,n)$, it is possible to calculate the deviation coefficient (3), which will depend on the homogeneity of the sample under study.

$$D_{in\hom} = \frac{\Psi_{in\hom}(s,t)}{\Psi(m,n)} \qquad (3)$$

In the process of searching for the average values, the areas on the edges of the sample were not taken into account because the edges were worn off during mechanical processing of the composite. The obtained average values of the voltage recorded by the detector, transmitted radiation for samples No. 1, 2, 3 and 4 are shown in Table 1.

Sample	Time of ultrasonic treatment (min)	Thickness (mm)	$\overline{U}$ (mV)
1	-	0.77	93
2	1	1.39	72
3	2	0.81	160
4	5	0.6	139

Table 1. Average values of the intensity of THz radiation transmitted through the samples.

Fig. 5 shows images of localized sections of 4 samples based on MWCNTs, where: a) a sample that was not treated with ultrasonic before polymerization; as well as samples that were treated with ultrasonic before polymerization for: b) 1 min, c) 2 min, d) 5 min.

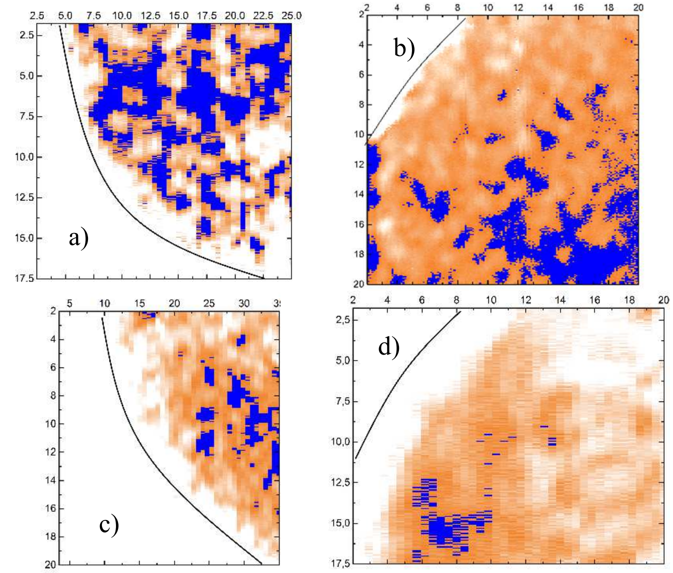

Fig. 5. THz images of composites based on MWCNTs, manufactured according to different technological processes: a) sample No.1 - without ultrasonic treatment; b) sample No.2 - with ultrasonic for 1 min; c) sample No. 3 – with ultrasonic for 2 min; d) sample No.4 – with ultrasonic for 5 min. Agglomerates are indicated in blue, voids are indicated in white.

The results presented in Fig. 5 show the area of inhomogeneities which were formed as a result of the formation of composite mixtures (MWCNT agglomerates and voids) at the mixing stage in sample No. 1 is 24.4%, in sample No. 2-19.4%, in sample No. 3 – 11.1%, and in sample No. 4 – 7.7% of the considered area in the corresponding sample.

III. Summary

As a result of the conducted research, it was shown that the homogeneity of the distribution of MWCNTs in composites depends on the time of ultrasonic processing. The longer the sample was subjected to ultrasonic processing, the more homogeneous it is. The possibility of using a submillimeter imaging system based on BWO to assess homogeneity in the manufacturing of composites is also shown.

ACKNOWLEDGMENT

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References

[1] Medupin R.O. et al. Carbon nanotube Reinforced natural Rubber nanocomposite for Anthropomorphic prosthetic foot purpose //*Scientific reports.* 2019. Vol. 9. No. 1. pp. 1-11.

[2] Ladani L., Awad I., She Y., Dardona S., Schmidt W. Fabrication of carbon nanotube/copper and carbon nanofiber/copper composites for microelectronics. *Materials Today Communications*. 2017. 11, 123-131.

[3] Yakovlev E.V. et al. Nondestructive testing of polymer composite materials using THz radiation, *J. Phys. Conf. Ser.* vol. 486, no. 1, 2014.

[4] Badin A.V., Berdyugin A.I., Vigovskii V.Y., Dunaevskii G.E. System of automated measurement of electromagnetic response of anisotropic materials in quasi-optical beams, *18th International Conference of Young Specialists on Micro/Nanotechnologies and Electron Devices*. IEEE, pp. 301-304, 2017.