QUESTION OF DETERMINING THE COMPOSITION OF COMPOSITES BY A TOMOGRAPHIC METHOD

V. I. Barakhov, V. A. Chernyaeva, V. S. Kiselev,
and L. D. Shestopal

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One possible method of determining the volume content of composite components by using x-ray calculational tomography is examined.

The volume content of the material components, the bonding filler, binder and medium in the pores as well as its variation over the thickness of composites, must be known to solve many practical heat and mass transfer, strength, and stability problems of shells from composite materials. This is especially important for preloaded shells having residual strains after removal of the loads. Porosity of the material grows in them mainly because of the cracking of the binder as the weakest link, and can exert considerable influence on the operability of the structure.

One means for determining the composition of composites can be based on the application of x-ray calculational tomography (XCT). The crux of the method is the scanning and measurement of the photon intensity of x-radiation after its passage through the substance of the object. The intensity of the radiation being recorded will here depend on both the radiation energy and on the structural inhomogeneities of the substance of the object on the ray path. Measurement of the radiation field parameters after interaction with the material carries information about is physicochemical properties, including the density. After mathematical processing of the measurement results we obtain a matrix of numbers characterizing the radiation absorption and scattering by each elementary cell of the specimen section being investigated. Each matrix element is a linear coefficient of attenuation (LCA), the Houndsfield number. The LCA for each elementary cell (a parallelepiped) is the integral characteristic. Using a calibrating dependence, each LCA value can be converted into a density. Therefore, converting the matrix $F(f_{ij})$ into the matrix $P(\rho_{ij})$ we can obtain the value of the density of each elementary cell of the internal structure of the specimen. Its dimensions are characterized by the scanning layer thickness d and the magnification coefficient z.

Investigations of organic plastic specimens on a tomograph showed that the correspondence between the true density and the LCA f(x, y) for them can be expressed with a certain approximation in the form of the following formula

$$\rho = (1 + 97{,}75 \cdot 10^{-5} f) \cdot 10^{-3}\ \text{kg/m}^3, \tag{1}$$

where $1 \cdot 10^{-3}$ is the density of water in kg/m³. The LCA of water equals zero ($f_{\text{water}} = 0$) for the Houndsfield scale.

As is known, the density of a composite material can be represented in the form

$$\rho = \rho_{fi}\mu_{fi} + \rho_b\mu_b + \rho_p\mu_p\,. \tag{2}$$

Tomographic investigations of organic plastic specimens as well as composite components, binder and filler in the form of rings obtained by a sintering method, and comparison of scanning results with traditional density measurement methods, showed that an additive law is valid even for the linear attenuation coefficients of x-rays. We therefore obtain for the LCA

$$f = f_{fi}\mu_{fi} + f_b\mu_b + f_p\mu_p. \tag{3}$$

Formula (3) permits going over to the determination of the volume content of material components. Let us assume that we have a matrix of LCA values $F(f_{ij})$ of the section of dry

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specimen from an organic plastic under investigation and the same section of a specimen with fluid-filled pores. Then the following system of equations is valid for the material layer

$$\begin{gathered} f_{di}=f_{fi}\mu_{fi}+f_{b}\mu_{bi}+f_{p}\mu_{pi}; \\ f^*_{di}=f_{fi}\mu_{fii}+f_{b}\mu_{bi}+f_{im}\mu_{pi}; \\ \mu_{fi}+\mu_{bi}+\mu_{pi}=1. \end{gathered} \tag{4}$$

The solutions of the system (4) for μ_{fii} and μ_{bi} in the i-th layer of the composite take the following form

$$\begin{gathered} \mu_{fi}=\frac{(f_{di}-f_{p})(f_{b}-f_{im})-(f^*_{di}-f_{im})(f_{b}-f_{p})}{(f_{b}-f_{fi})(f_{im}-f_{p})}; \\ \mu_{bi}=\frac{f^*_{di}-f_{im}}{f_{b}-f_{im}}-\frac{f_{fi}-f_{im}}{f_{b}-f_{im}}\mu_{fi}; \quad \mu_{fii}=1-\mu_{pi}-\mu_{bfii}. \end{gathered} \tag{5}$$

The concept of porosity is related directly to the body density. Comparing values of the theoretical and actual densities of the specimen under investigation, we can determine its porosity by neglecting the small contribution from other defects [1, 2]

$$C=\left(1-\frac{\rho}{\rho_T}\right)\cdot 100. \tag{6}$$

The porosity is here separated into open and closed, the open is measured by filling the specimens with liquid. Dry specimens are weighed in air then soaked in a vacuum in a fluid whose selection is determined by the composite being investigated. The soaked specimen is then weighed in air and in water. The open porosity is calculated from the formula [2]

$$C_{op}=\frac{(m_1-m_{fi})\rho_w\cdot 100}{\rho_{im}(m_1-m_2)} \tag{7}$$

and the closed porosity is

$$C_d=C-C_{op}. \tag{8}$$

Let us assume that the volume content of closed pores is α and of open is β under the condition α + β = 1 in the organic plastic under investigation. Then the system of equations for the linear attenuation coefficients of the i-th layer of dry and saturated specimen can be written as follows in the case of pores filled by a fluid

$$\begin{gathered} f_{di}=f_{fi}\mu_{fii}+f_{b}\mu_{bi}+f_{b}\mu_{pi}; \\ f^*_{di}=f_{fi}\mu_{fii}+f_{b}\mu_{bi}+(\alpha_i f_{p}+\beta_i f_{im})\mu_{pi}; \\ \mu_{fii}+\mu_{bi}+\mu_{pi}=1. \end{gathered} \tag{9}$$

Solutions of the system (9) for μ_{fii} and μ_{bi} take the form

$$\begin{gathered} \mu_{fii}=\frac{(f^*_{di}-\alpha_i f_{p}-\beta_i f_{im})(f_{b}-f_{p})-(f_{di}-f_{p})(f_{b}-\alpha_i f_{p}-\beta_i f_{im})}{(f_{b}-f_{fi})(f_{p}-\alpha_i f_{p}-\beta_i f_{im})}; \\ \mu_{bi}=\frac{f_{d}-f_{p}}{f_{b}-f_{p}}-\frac{f_{fi}-f_{p}}{f_{b}-f_{p}}\mu_{fi}. \end{gathered} \tag{10}$$

A tomographic investigation of the composition of the organic plastic by the proposed method encounters a number of difficulties. The first is the circumstance that from a definite time the composite starts to swell noticeably during saturation. An increase in the specimen mass and the growth and then decline in the density are observed. It can be assumed

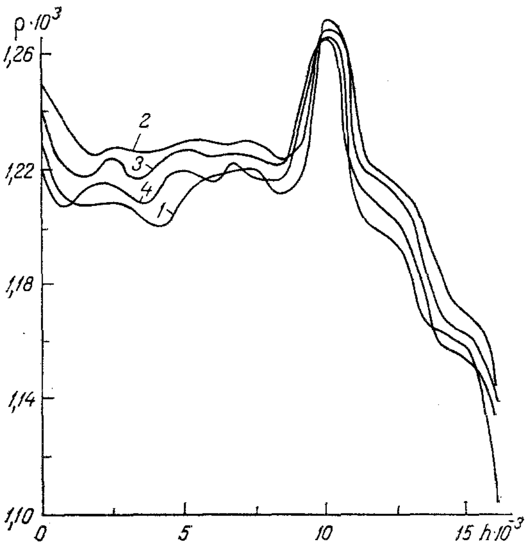

Fig. 1

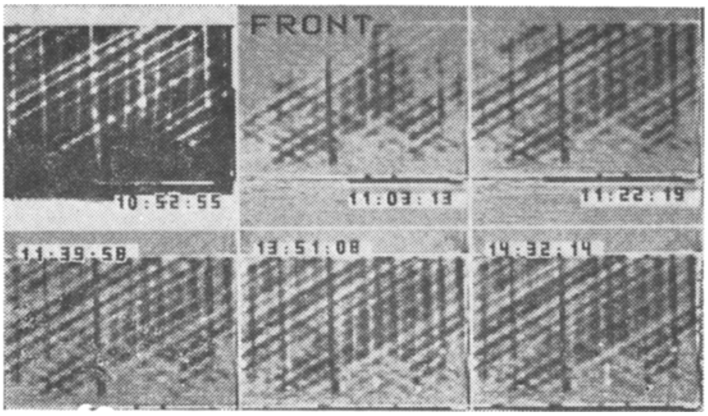

Fig. 2

Fig. 1. Curves of the density ρ, kg/m^3, over the composite specimen thickness h, m, during its saturation by water: 1) original state; 2, 3, 4) after 7, 14, 28 days of saturation by water, respectively.

Fig. 2. Tomograms of the winding layer of a composite displaying water propagation therein: original "dry" state corresponds to the time 10 h 52 min 55 sec; the tomograms in the subsequent five times are executed by using image subtraction operations.

that although growth of the density proceeds, filling of the open pores occurs. Then because of the swelling the volume starts to increase and the density drops. The characteristic curves for one of the organic plastic specimens are shown in Fig. 1. The systems of equations considered for the LCA are valid at the time the saturation process goes over from one stage to another, i.e., when all the open pores are filled with fluid and there is no displacement of specimen layer sections because of swelling. This time can be determined by periodic scanning of the identical section during saturation and processing the matrix of LCA values.

The second important feature of the tomographic method of investigation is the special mathematical processing of the scanning data on an electronic computer. It is in the tomograph special processor and is a "black box" for the researcher. Numerous contrasting experiments on determining the density of organic plastics by tomographic and ordinary measurement methods showed that the most acceptable modification of scanning data processing when using the applied tomograph is the high resolution regime (H1) characterized by emphasizing

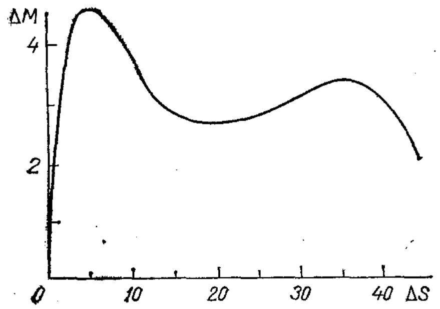

Fig. 3

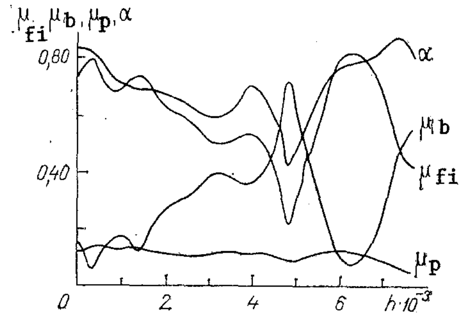

Fig. 4

Fig. 3. Dependence of increments in the mathematical expectation of the LCA values ΔM on the diminution of their rms deviation ΔS.

Fig. 4. Curves of the variation in the volume content of organic plastic components over the specimen thickness.

the contrasts of the internal structure. A diminution in its contrast is observed when filling the pores and capillaries of the inner structure of the organic plastic with water, i.e., when the process of water displacing the air proceeds. The structure becomes more homogeneous in density, which is characterized by a diminution in the value of the rms deviation. If the process of filling the capillaries of one inner layer (Fig. 2) with water is observed by periodic scanning then it can be seen how the water starts to fill the capillary being formed during winding, heat treatment, and loading of the shell between braids, by penetrating it. A quantitative analysis of water propagation over the layer can be performed if the operation of subtracting the matrix of LCA values $F(f_{ij})$ is used. By having the m matrix of LCA values for the layer under consideration in the initial state $F_0(f_{ij})$ and an analogous matrix $F_t(f_{ij}^*)$ at the time t from the beginning of saturation, we can obtain the matrix of the difference $F_{t-0}(f_{ij}^{(-)})$

$$F_{t-0}(f_{ij}^{(-)}) = F_t(f_{ij}^*) - F_0(f_{ij}), \tag{11}$$

where $f_{ij}^{(-)} = f_{ij}^* - f_{ij}$ ($i = 1, 2, \ldots, n$; $j - 1, 2, \ldots, m$).

Such an operation permits the extraction of the water that has penetrated the individual elementary cells, the consideration of its motion over the capillaries along the winding layers, and the passage to quantitative estimates. The whole difficulty here is that the convolution kernels in the H1 mode used to process the scanning data yield negative values $f_{ij}^{(-)}$ in cells corresponding to the winding braids in addition to the positive values of $f_{ij}^{(-)}$ in cells characterizing the capillaries by seemingly "observing" the change in contrast when filling the emptiness with water. These negative values underestimate the results of the tomographic measurements. We try to correct the work of the convolution kernels in the H1 regime by replacing all the negative values of $f_{ij}^{(-)}$ by zeroes in the matrix $F_{t-0}(f_{ij}^{(-)})$. This is equivalent to the fact that there is no change in density in the winding tapes at the initial time of saturation. Therefore, the imaginary unloading domains are cancelled. We obtain the matrix $F_{t-0}^*(f_{ij}^{*(-)})$ that is characterized just by increments of the LCA values because of water penetration into the elementary cells. Now if we combine the matrices $F_{t-0}^*(f_{ij}^{*(-)})$ and $F_0(f_{ij})$ we then obtain the corrected matrix of LCA values at the time t:

$$F_t^*(f_{ij}^{**}) = F_0(f_{ij}) + F_{t-0}^*(f_{ij}^{*(-)}), \tag{12}$$

where $f_{ij}^{**} = f_{ij}^* + f_{ij}^{*(-)}$.

Each of the matrices under consideration is characterized by the mathematical expectation $\bar{f}$ and the rms deviation S_f. By analyzing the results obtained during performance of tomographic scanning experiments of an organic plastic layer during water propagation over the material, the dependence between the change in S_f (ΔS_f) to the time t and the increment $\bar{f}$ can be determined by taking account of the correction of the work of the convolution kernels

$$\Delta\bar{f} = f(\Delta S_f). \tag{13}$$

TABLE 1. Results of Computations of the Volume Content of Composite Components

Layer number	μ_{fi}	μ_b	μ_p	α	Layer number	μ_{fi}	μ_b	μ_p	α
1	0,600	0,198	0,202	0,808	11	0,598	0,292	0,110	0,880
2	0,600	0,234	0,166	0,898	12	0,600	0,287	0,113	0,881
3	0,600	0,242	0,158	0,873	13	0,602	0,283	0,115	0,850
4	0,599	0,246	0,154	0,760	14	0,600	0,282	0,118	0,825
5	0,597	0,269	0,133	0,810	15	0,600	0,272	0,128	0,798
6	0,600	0,272	0,128	0,827	16	0,600	0,275	0,125	0,791
7	0,600	0,314	0,086	0,877	17	0,595	0,285	0,120	0,810
8	—	—	—	—	18	0,600	0,277	0,123	0,834
9	0,600	0,287	0,113	0,863	19	0,605	0,282	0,113	0,810
10	0,602	0,277	0,121	0,860	20	0,600	0,284	0,116	0,510

An algorithm has been developed to determine the dependence (13) and the program BARR has been compiled in the language PL/1. The results of computations on an electronic computer according to the program for the LCA matrices of one of the experiments in an investigation of water propagation in an organic plastic layer are presented in Fig. 3.

The method elucidated for determining the composition of composites was used to investigate organic plastic specimens cut out of a preloaded shell. The specimens were dried for 150 days in a calcium chloride atmosphere before performance of the tests. In order to fill the capillaries better, water saturation was performed under vacuum evacuation conditions for the tank with the specimen being tested. The results of the scanning and processing of the LCA matrices of a fixed section of material during variation of its density under the effect of water filling the pores and swelling are presented in Fig. 1 in the form of the graphs $\rho = f(h)$. The peaks on the density graphs correspond to a rubber layer between force shells. It is seen from the graphs that filling the capillaries with water proceeded during the first seven days. Later the process of material swelling began. Its density diminished because of growth of the volume. Consequently, matrices of LCA values of the initial state of the material and saturated by water for seven days were used in performing the computations. A program OBJEM in the language PL/1 was developed to determine the volume content of composite components. The results of computations according to the program for the specimen under investigation are presented in the table. Scanning the specimen was performed for $z = 2.5$, which corresponds to a $0.85 \cdot 10^{-3}$ m spacing between layers. By analyzing the table it can be concluded that closed or isolated pores predominated in the organic plastic under investigation. The mean characteristics over the whole volume of the section yield the following results $\mu_b = 0.275$; $\mu_p = 0.123$; $\alpha = 0.830$. An assumption is made in the computations that the volume content of the filler does not vary in practice in the case under consideration.

Specimens from an organic plastic panel were fabricated to compare the results of determining the volume content of composite components obtained by the method proposed to those already known. The comparison was made with an ultrasound method and the method of microstructural analysis. The ultrasound method permits determination of the volume content of the bonding filler in the whole volume of the material. It requires a large quantity of initial formation and, consequently, can yield great errors. The method of microstructural analysis requires the presence of a microsection on the specimen surface under investigation. Polishing the surface disturbs the material structure. Practically all the pores vanish. Consequently, the comparison was performed only in the volume content of the bonding filler. The tomographic method is realized exactly as in the first case by application of drying and evacuation. Scanning was performed from $z = 7$ which yields an elementary cell dimension equal to the nominal thickness of the winding layer. The ultrasound method yielded 54.0% fiber, 29.7% binder, 10.6% pores, 70% of which are closed, for the specimens under investigation. If the methodological errors of the microstructural analysis method are taken into account, a deduction can be made from comparing the results that the XCT yields sufficiently accurate results. Binder interlayers are formed during winding of shells from composites because of the replacement of tension by juncture of the layers with different bonding angles. Microstructural analysis skips these interlayers since the filler volume content therein ≈ 0.

The results of processing the LCA matrices, obtained during the comparison experiments described above, are represented in Fig. 4 in the form of curves over the thickness of the specimen under investigation. As is seen from the figure, application of the XCT permits obtaining not only the volume content of the filler but also of the binder and the pores by separating the porosity into open and closed and retaining the completeness of the specimen. Analysis of the curves in Fig. 4 shows that the porosity is reduced in layers with elevated binder content. The arrangement of the interlayers over the material thickness is seen from the curves, the tendency to increase the volume content of binder with the approach to the specimen outer surface is noticeable because of its extrusion during winding due to the contract pressure of the layers. Closed pores predominate as in the first experiment.

Therefore, the proposed tomographic method of determining the composition of composites permits computation of the volume content of the original components, the porosity, the volume content of the open and closed pores in each elementary layer of material, and clarification of the regularities of its structure.

NOTATION

ρ, ρ_T, ρ_{fi}, ρ_b, ρ_p, ρ_w, ρ_{im}, material densities, theoretical, fiber, binder, pore, water, impregnating fluid; μ_{fi}, μ_b, μ_p, fiber, binder, pore volume contents; f_{fi}, f_b, f_p, f_{di}, f_{di}^*, f_{cij}, f_{im}, linear coefficients of attenuation of the fiber, binder, air of the i-th layer of the dry specimen; i-th water-saturated layer of the specimen; j-th cell of the i-th layer of the specimen under investigation, impregnator; n, quantity of cells in the i-th layer, m_{fi}, m_1, m_2, weight of the dry and impregnated specimens in air and in water; C_{cl}, closed porosity; S_f, rms deviation of the linear attenuation coefficients; and α, volume closed pore content.

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MEASUREMENT OF THERMAL DIFFUSIVITY OF DIAMOND BY THE LIGHT-INDUCED THERMAL LATTICE METHOD

V. V. Gorbachev, V. M. Durasov, R. B. Zezin,
E. V. Ivakin, and A. S. Rubanov

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The thermal diffusivity coefficient of natural diamonds is measured by optical induction of thermal diffraction lattices.

Diamond is the unique natural material which combines thermal and electrical conductivity properties most suitable for use in electronic technology. The wide use of diamonds as heatsinks in microelectronics requires development of a rapid and reliable method for testing the thermal conductivity χ of crystals. The traditional methods of thermophysical experiment (see, for example, [1]) are cumbersome, require much time, and are unsuitable for studying crystals of small size. Spectral measurements [2] provide indirect information on χ and do not consider the presence of various defects, for example, dislocations, which affect the thermophysical properties of the diamond. There are available a few studies [3] using Rayleigh scattering of light within a diamond, on the basis of which it is in principle possible to determine the thermal diffusivity coefficient κ. However the extremely low level of the desired signal and noise produced by elastic scattering on crystal inhomogeneities require use of expensive optical and electronic instrumentation and lengthy processing of the measurement results.

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