Study of the structure of fibrous composite material using computational x-ray tomography

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Abstract: The article discusses the determination of the actual porosity and the actual volumetric content of the reinforcing material and binder in a sample of fibrous polymer composite material (FPCM) using the non-destructive method of computational x-ray tomography. It is shown that the linear attenuation coefficient (LAC) of X-ray radiation in the volume of the sample under study based on the set of integral shadow projections obtained by X-ray transmission in various directions and its root mean square deviation (RMSD) provide sufficient information about the parameters of the microstructure of the sample under study. To obtain quantitative characteristics of porosity and volumetric content of components, it is necessary to examine test samples made from similar components with previously known various parameters of the material structure in the required range of scatter of these characteristics. The proposed method can be used as a non-destructive control of the structure parameters of critically important products made of VPCM, both in general and in selected local areas of the product. Also, this method is recommended for determining the actual porosity and the actual volumetric content of the binder when carrying out research work on the influence of VPCM structure parameters on the durability and residual strength of samples when exposed to external factors during operation, in particular when carrying out certification work on elementary samples.

1 Introduction

Modern fibrous polymer composite materials (FPCM) have undeniable advantages over traditional structural materials, primarily due to higher specific elastic strength characteristics. However, the heterogeneous structure of VPCM presupposes the presence of porosity in the final volume of the manufactured part, associated with the peculiarities of the manufacturing technology, namely, during the implementation of the processes of combining the binder with the reinforcing material, forming the geometry and reinforcement scheme of the part, pressing and curing the composite package of the part with alternating curvature, as well as internal residual stresses in the part after its manufacture [1]. The latter lead to internal damage to the cured matrix, disruption of the...
fiber-matrix bond and rupture of individual fibers, which even theoretically does not allow obtaining an ideal heterogeneous structure. In addition, the actual ratio of reinforcing material and binder in different zones of the part may differ from the required one, especially when using methods of impregnation of the reinforcing frame of the part (preform) directly in the forming equipment (vacuum infusion or RTM). The porosity of the VPCM structure primarily leads to moisture absorption during operation, which is especially dangerous when the temperature passes through the freezing point of moisture [2]. As a result, internal changes in the structure are observed in parts made of VPCM, leading to a decrease in performance properties.

2 Materials and Methods

This article discusses the study of the structure of VPCM samples using computational X-ray tomography (XRT). The issue of determining the actual porosity and the ratio of components by non-destructive testing methods on the studied full-scale samples or manufactured parts is a complex technical problem, in particular in the industrial production of products from VPCM. Traditionally, the porosity of composites is determined quite accurately by the method of hydrostatic weighing, and the ratio of components is determined by the method of burning out the binder, but for this it is necessary to cut a control sample from the studied area of the part or make a witness sample [3]. Obviously, the first method is unacceptable for industrially manufactured composite parts, and witness samples reliably determine the necessary structural parameters outside the controlled zone of the composite of the part itself. The VRT method allows you to examine any selected zone of a manufactured part without violating its integrity.

When manufacturing parts from VPCM, the amount of reinforcing material (fibers) in any zone or section of the part is known in advance, constant and determined by a given reinforcement scheme [4]. The type of fibers is also clearly defined, in particular their true density and the textile form of the reinforcing semi-finished product, for example a unidirectional tape, an equilibrium fabric of a certain weave or a multi axial fabric. Thus, in any zone of the manufactured part, the amount of reinforcing material is known in advance. The cured binder (matrix) and the volume of voids (pores) in the section under study will occupy the remaining volume. This volume depends on the degree of compression of the reinforcing material during molding and the amount of pre-applied binder to the reinforcing material when used as a prepreg semi-finished product. Table 1 presents the measured values of the thickness of one layer of various types of carbon reinforcing materials (taking into account their textile form and laying pattern) for different crimping pressures and the calculated remaining “free” volume, which during the manufacture of the sample should be “occupied” by the binder, taking into account the pore volume [5]. As can be seen from the table, for the same textile form of reinforcing material at the same crimping pressure, the thickness of the monolayer of reinforcing material depends on the laying pattern of the layers.

<table>
<thead>
<tr>
<th>Fiber Type</th>
<th>Surface Density (warp/weft, weave)</th>
<th>Fiber Type</th>
<th>Fiber Density (g/cm³)</th>
<th>Styling</th>
<th>Layers</th>
<th>Layer Thickness (mm)</th>
<th>Free Volume (%)</th>
<th>Layer Thickness (mm)</th>
<th>Free Volume (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>INUMiT 2</td>
<td>200; (2:2, twill)</td>
<td>HTA40</td>
<td>1.76</td>
<td>0/90</td>
<td>0.194</td>
<td>41.4</td>
<td>0.180</td>
<td>36.9</td>
<td>0.168</td>
</tr>
</tbody>
</table>

Table 1. The values of the thickness of one layer of various types of carbon reinforcing materials
The apparent density of a composite sample, taking into account the presence of voids and pores in its structure, can be calculated based on the following relationship (additivity rule):

\[ \frac{m_v}{\rho_v} + \frac{m_m}{\rho_m} + \frac{V_{por}}{V_{km}} = 1; \]  

(1)

The calculated value of the relative porosity of the composite sample is determined from the following dependence:

\[ V_{por} = \left(1 - \frac{p_{km}}{m_{km}} \right) \left(\frac{m_m}{\rho_m} + \frac{m_v}{\rho_v}\right); \]

(2)

where \( V_{km} \) and \( m_{km} \) - actual volume and mass of the composite sample; \( m_m \); \( \rho_m \) and \( m_v \); \( \rho_v \) - the actual mass and true density of the matrix and fiber in the composite specimen. The actual density of the fibers of the reinforcing material is taken from the passport data for the material, the actual mass of the composite sample is determined by weighing, the density of the composite sample and the cured matrix is determined by hydrostatic weighing.

Thus, for a constant volumetric content of reinforcing fibers and applied binder, depending on the manufacturing technology of the part or sample, different relative voids or pores are obtained, which leads to a scatter in the apparent density of the composite.

In Fig. 1 shows the calculated dependence of the apparent density of carbon fiber reinforced plastic on porosity for different volumetric contents of reinforcing fibers. It is clear from the graphs that the value of the apparent density of carbon fiber plastic depends on the ratio of the relative volume content of the fibers of the reinforcing material and the pore volume. Thus, the same value of apparent density can correspond to a composite more “filled” with reinforcing fibers with a significant volume of voids and pores, or less “filled”, but with low porosity.
It is known that when materials with different densities are illuminated with X-rays, the attenuation of radiation increases in proportion to the increase in the density of the material, which makes it possible to quantify the apparent density of the sample under study.

In the general case, VRT solves the problem of reconstructing the three-dimensional distribution of the linear X-ray attenuation coefficient (LCO) in the volume of the sample under study. Reconstruction of the LCO distribution is carried out using integral shadow projections obtained by X-ray transmission in various directions [7]. In this case, a three-dimensional problem, as a rule, is reduced to a two-dimensional one, when the research process is reduced to the reconstruction and study of two-dimensional tomograms, which represent the distribution of LCR and its standard deviation (RMSD) over the section under study. Figure 2 shows the resulting LCR distribution and visualization of the reconstructed cross-section of several carbon fiber samples with different volumetric content of reinforcing fibers and porosity [8-10]. Since the LCO distribution at a constant composition of the composite is proportional to the density distribution \( p(x,y) \), then from the reconstructed tomogram it is possible to make a quantitative estimate of the apparent density in the sample section under study. Having obtained the average LCR value for the studied cross-section of a sample with a given volumetric content of reinforcing fiber, one can generally estimate the different combination of the amount of binder and pore volume that affects the apparent density of the composite, which is clearly presented in Fig. 1.

To analyze the structure of the material based on processing the averaged scanning parameters, a correlation criterion between the reconstructed scanning results is adopted—the standard deviation of the value (RMS) for the LCO in the scanned section. The RMSD parameter characterizes the spread of the absolute value of the deviation of the LCO from the most probable value characteristic of different porosities of a composite made from a selected reinforcing material with its fixed volumetric content. With an increase in porosity, the average value of the LCO in the section under study decreases, and the absolute value of the MSD increases [11-13]. This “scenario” corresponds to a change in the apparent density of the composite depending on the volume “free” of fibers occupied by the binder and pores in their different ratios.
3 Research and results

To experimentally determine the actual volumetric content of the binder and the real porosity, test samples were made from the same reinforcing material (fiber type and textile form) with a given volumetric content of fibers and different porosities, a series of such samples were studied and the dependences of the LCO and RMSD were constructed. The actual porosity of the samples was determined in advance by hydrostatic weighing. Table 2 presents a summary table of studies of a series of samples made from unidirectional tape 10425 “INUMiT” based on HTS40 carbon fiber in the form of a thread with a linear density of 12K (800 tex), as well as the average values of LKO and MSD in scanned sections. Samples 1.1….1.5 (see Table 2) were made of prepreg. Different porosities for samples made of prepreg with constant binder application were achieved by using a release film with different open perforation areas. The use of separating films with different perforations during evacuation in an autoclave provided different “output” of the binder into the drainage layer of the vacuum bag [14-17]. Minimum porosity for prepreg samples was achieved through the use of a microperforated release film that allows air and volatiles to pass through, but serves as a barrier to binder melt. Samples 2.1 ... 2.4 were produced by infusion under a double vacuum bag; different porosity in the sample was ensured by reducing the vacuum depth under the inner bag during the impregnation of the preform with a binder.

Table 2. Samples 1.1….1.5

<table>
<thead>
<tr>
<th>Sample No</th>
<th>2.1</th>
<th>2.2</th>
<th>2.3</th>
<th>2.4</th>
<th>1.1</th>
<th>1.2</th>
<th>1.3</th>
<th>1.4</th>
<th>1.5</th>
</tr>
</thead>
<tbody>
<tr>
<td>reinforcing material</td>
<td>unidirectional tape 10425 “INUMiT”</td>
<td>(reinforcing fiber HTS40 F13 12K)</td>
<td>infusion</td>
<td>prepreg, vacuum ± autoclave</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>molding method</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>relative volume content</td>
<td>58%</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Arm. m.la</td>
<td>62%</td>
<td></td>
<td></td>
<td></td>
<td></td>
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<td></td>
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</tbody>
</table>
It should be noted that samples prepared by infusion under a double vacuum bag (when impregnated with full vacuum) have porosity that is less than the threshold sensitivity of the method for determining it. It was not possible to obtain porosity less than 1.1% on prepreg samples [18, 19].

In Fig. Figure 3 shows graphical dependences of the average value of LCO and RMSD in the studied sections of carbon fiber samples on the specific porosity of the composite. The averaged values of LKO and MSD were obtained as a result of several scans of sections in mutually perpendicular directions (see Table 2).
thickness of the sample. The actual porosity of the obtained samples is determined by an independent method. All samples are examined on a tomograph and, based on the results of the averaged LCO value and the corresponding RMSD, dependencies similar to those presented in Fig. 3 are constructed.

2. Acceptable values for the relative volumetric content of fibers of the reinforcing material VB min and VB max and porosity are assigned, based on the requirements for the material of the composite product. Based on the results of the study of test samples, the range of permissible values of LCR and RMSD is determined as a function of porosity for parametric values of the relative content of reinforcing fibers (see Fig. 4).

Fig. 4. Illustration of a method for determining permissible porosity in a composite with a given spread of values of the relative content of reinforcing material using averaged scanning results using the HRT method.

In Fig. 4 the following notations are used:

- $V_{\text{por}}^{\text{min}}$ – minimum detectable porosity,
- $V_{\text{por}}^{\text{max}}$ - maximum permissible porosity,
- $P_{\text{LCO}}^{\text{min}}$ and $P_{\text{LCO}}^{\text{max}}$ – minimum and maximum LCO value for the restrictions specified above.

The selected section in the controlled part is examined and the LCO values are recorded – $P_{\text{LCO}}^{\text{fact}}$ and $P_{\text{SCO}}^{\text{fact}}$, the actual thickness in the selected section is measured $\delta_{\text{fact}}$, from equation (3) the actual volumetric content of reinforcing fibers is determined $V_{v}^{\text{fact}}$, its value must be in the range $V_{v}^{\text{min}}$ and $V_{v}^{\text{max}}$.

From the obtained dependences of LCO and RMSD on porosity for the actual fiber content $V_{v}^{\text{fact}}$ the corresponding dependencies are highlighted, which must be in the specified range of parametric dependencies $V_{v}^{\text{min}}$ and $V_{v}^{\text{max}}$, it determines the porosity corresponding $P_{\text{LCO}}^{\text{fact}}$ (see fig. 4).

Depending on the “porosity – RMS”, for the parameter $P_{\text{SCO}}^{\text{dop}}$, in Fig. 4 is indicated by a green segment. Actual value $P_{\text{SCO}}^{\text{fact}}$, obtained as a result of studying the selected area of the part must be in this range.

4 Conclusion

The proposed method can be used as a non-destructive control of the structure parameters of mass-produced critical products made of VPCM in selected local areas of the product. To do this, it is necessary to manufacture and study the structure of the composite on a tomograph of reference samples with a given volumetric content of reinforcing fibers and different porosities determined by other reliable methods. Reference samples with different
ratios of components must be made of the same reinforcing material and binder as the part being tested. As shown above, this will make it possible to quantify the actual binder content and real porosity in different sections of the part, using the resulting parametric “grid” of associated values of LKO and MSD. The actual volumetric content of the reinforcing material in any section of the part, regardless of the ratio of the amount of binder and pores, can be determined as the ratio of the volume of fibers of the reinforcing material to the measured thickness of the part in the section under study (see Table 1).

Also, this method is recommended for determining the actual porosity and the actual volumetric content of the binder when carrying out research work on the influence of VPCM structure parameters on the durability and residual strength of samples when exposed to external factors during operation, in particular when conducting certification tests on elementary samples.

References


