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CT STUDY OF PRODUCTS FROM NON-METALLIC COMPOSITES REINFORCED BY CARBON FIBERS

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Computerized tomography is used to study products from non-metallic composites reinforced by carbon fibers. It is demonstrated that samples do not rotate during the analysis process and can have diameter not exceeding the gantry hole (75 cm) of the unit. The weight of samples can be up to 200 kg, and the length during one-time testing does not exceed 150 cm. The results of spatial calibration of the technology are shown using a set of copper wires with diameters from 0.56 to 0.05 mm. The paper presents the results of defects detecting in products in the form of gas pores and metal inclusions.

INTRODUCTION

In critical industries, which include, first of all, nuclear power engineering, aeropace technology, military industry, medicine and etc., traditionally, increased requirements are imposed on analysis methods. One of the concepts is production of reference samples of products with subsequent determination of the obtained characteristics on them and attribution of the obtained results to the entire batch of products. This concept uses destructive research methods, which, on the one hand, lead to cost overspending for the production of reference samples, and, on the other hand, do not provide absolute quality guarantee for the remaining products in the batch.

Use of non-destructive research methods constitutes an alternative concept to solve the quality analysis problem, production support and final product certification.

Fig. 1 shows analytical technologies which can be used as non-destructive methods to study surface and amount of materials, as well as capabilities of each method in terms of spatial resolution.

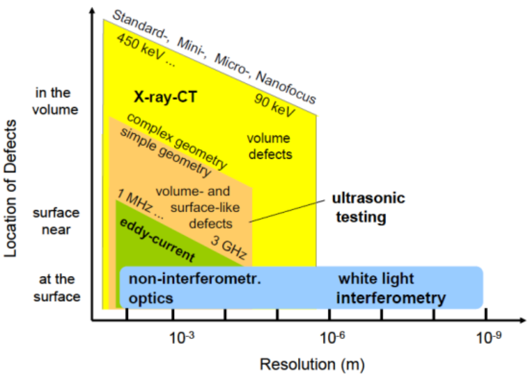


Fig. 1. Non-destructive analysis methods [1]

The most commonly used methods in production are ultrasonic diagnostics and X-ray analysis methods. However, ultrasonic diagnostics method has disadvantages which include difficulties with insufficient thickness of analysis, spatial localization of defects, their visualization and classification.

One of the most advanced and effective methods of non-destructive analysis is X-ray computerized tomography (CT) which allows to effectively solve the problems described above, in particular, allows identification and spatial localization of inner defects, assess variation in material density and geometric conformity of the object. Use of computerized tomography has been steadily expanding in mane industrious sectors.

As can be seen from Fig. 1, computerized tomography, depending on radiation energy of the source, allows to determine material defects in the sample amount with rather good spatial distribution, for large objects as well.

In the world scientific community, more and more attention is paid to research related to capabilities and application of computerized tomography in the study of advanced materials. Large literary reviews are published on this method of materials research [2, 3].

Non-metallic composites reinforced with carbon fibers belong to the class of materials that are intensively studied in the scientific field and are increasingly used in industry. Use of carbon fibers is associated with their unique properties, primarily high strength characteristics, high elasticity modulus, excellent corrosion properties, high temperature, radiation and chemical resistance. For example, publications [4, 5] present the microCT method in the study of polymer reinforced with carbon fibers. This examines the structure of the weave and determines the distribution and size of the carbon fibers.

However, the published materials practically do not study defects in the form of voids, cracks in the material, or metal inclusions in the production of products. At the same time, presence of metal and gas inclusions in materials, especially when they are used in critical nodes, can have a significant negative effect on the performance of products as a whole, their integrity, strength, and corrosion. radiation and erosion resistance.

Fig. 2. Tomograph SOMATOM Definition AS 64

The main purpose of the article using CT method with non-rotating object is to demonstrate capabilities of this method to visualize spatial distribution of defects in preform (product), including voids (gas pores) in material and possible metallic *inclusions*.

METHOD AND EQUIPMENT

The concept of CT method is to irradiate the object under study by X-ray beam in different directions on the object and determine the decrease of their intensity linear trajectories. This decrease is determined by the Lambert-Baer law. In the case of the intensity of the monochromatic X-ray radiation from the source I_0 and the recorded radiation I, as well as the object under study, consisting of several materials, this law is written in the following way:

$$I = I_0 \cdot exp\left[\sum_i \left(-\mu_i \, x_i\right)\right],$$

where μ_i – attenuation factor of irradiation for each and material; x_i – path of irradiation transport.

Based on the measured values of X-ray radiation, it is necessary to restore the decay function of parts of the irradiated sample.

CT main elements are: radiation source and detector, as well as their spatial arrangement. X-ray tube is a source of radiation in most cases of tomography; however, it is possible to use synchrotron radiation or particle beam [6, 7].

Currently, most firms use solid-state detectors [8] These are various scintillators and detectors with direct conversion of electromagnetic radiation into electric charge.

For each generation of tomographs, detector and X-ray tube are located in different spatial positions. We selected tomograph with spiral CT technology to study non-metallic composites reinforced with carbon fibers.

Study is focused on the use of carbon-carbon composite fiber material with orthogonal 3D reinforcement structure made using original thermo-gradient gas-phase methods of NSC KIPT [9–11]. Material was made on the basis of PAN (polyacrylonitrile) carbon fiber frame. The resulting composite had an apparent density of 1.75 g/cm^3 with open porosity of 4…6% and netting unit cell size of ~ 2.5 mm.

Samples made of carbon materials were tested on SOMATOM Definition AS 64 multi-detector computer tomograph (Siemens) (Fig. 2). Measurement was performed using spiral X-ray tomography. UFC (Ultra Fast Ceramics) detector of CT allows to get 64 cuts in one rotation. STRATON X-ray tube was used. Frequency of position change between points is 4608 Hz. Tube provides direct oil cooling of anode at rate 7.3 MHU/min with maximum rotation rate of anode 0.33 s. z-Sharp technology was used during the image reconstruction; this technology allows to get minimum isotropic voxel with size of 0.24 mm regardless of scanning field. Gantry aperture of tomograph was 78 cm. Computerized tomograph allows scanning along Z axis up to 200 cm with rate up to 98 mm/s with time separation 165 ms. Main data on scanning of products made from carbon materials were obtained at voltage of X-ray tube of tomograph 140 keV.

Tomograph table was moved in horizontal direction according to displacement equation for spiral tomography while the layers were close to each other.

Sensitivity of tomograph is related to the physical basis of the method and possible presence of artifacts in image reconstruction determined by presence of possible metal inclusions in the object.

For each of the monitored objects, its image was reconstructed using the obtained X-ray spectra. Standard tomograph software based on the Algebraic Reconstruction Method (ART) was used.

RESULTS

Previously, the state standard for radiographic testing was used in the study and testing of products made of carbon-carbon materials. Its use allowed to detect defects of the inspected sample in flat version using X-ray film. Further, at NSC KIPT, after a series of experiments, the enterprise standard STP NSC KIPT 076:2017 was established, according to which the study of carbon samples was performed on standard tomograph by CT method. This considered the need to simultaneously and without destroying the object to test and study carbon products with sufficiently large weight, reaching hundreds of kilograms; large sizes – up to 70 centimeters in diameter and various specific gravity. Tomographic studies were performed both at the stage of quality control of the weaving of the reinforcing frame (preform) with a specific gravity of 0.35…0.5 g/cm^3, and at the stage of testing blanks after pyrocompaction, having a density of 1.65…1.85 g/cm^3. At the same time, in all cases, CT testing was performed with a fairly good spatial resolution – hundreds of microns.

Study of real objects made of carbon-carbon materials was preceded by the calibration of the tomograph using measured samples with pre-introduced calibrated defects, which showed the possibility of using CT method to solve the indicated problems.

Maximum possible X-ray energy for the specified tomograph was selected to prevent intensive background and achieve the best spatial resolution during scanning of products and standard samples. It was 140 keV with current more than 200 mAc.

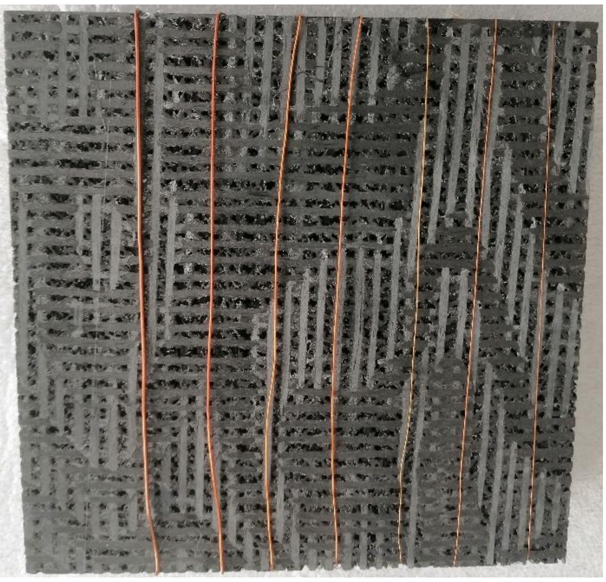
Study of spatial resolution method and the tomography unit was performed before testing real products, which clearly demonstrates possibilities of CT method in use.

Fig. 3 shows an object in the form of parallelepiped made of carbon material, into which copper wires of different diameters are introduced. Wires are intended for real determination of spatial resolution of tomograph which combines its physical equipment and measurement method with the applied software.

Study of elemental composition of material from which the sample is made demonstrated that it is carbon with with possible additives of organic matter and impurity elements (B, Si, S, Ca < 0.01%, methods PIXE, PIGE). And this does not change the absorption factor of the applied X-ray radiation in the objects of the specified size.

Wires were closed with another scrap of material to simulate more correctly the location of metal objects in the body of the carbon material after being installed and glued on the lateral surface of the sample. Fig. 3b shows sample for CT studies with dimensions 100 x 100 m x 40 mm.

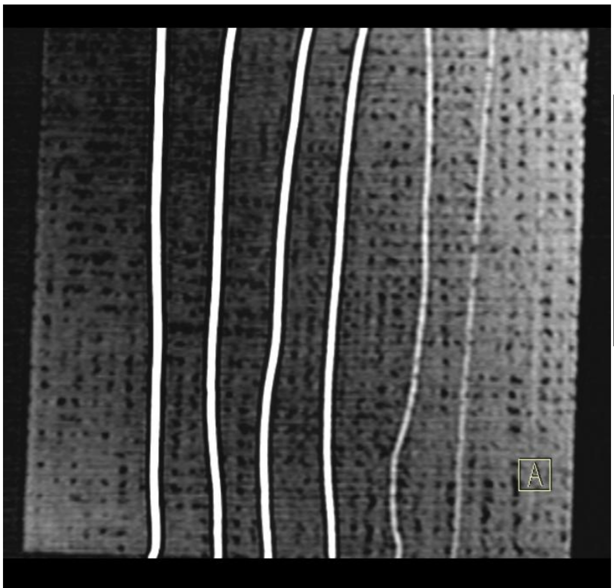
Size of wires was selected so as to cover the range of possible spatial resolution of the tomograph. Wires are varnished and, therefore, their actual diameter should be reduced by 6…15% from indicated value. It was taken into account that when obtaining a multi-cut image, the thickness of one cut was equal to 600 μm, which is practically the minimum for medical X-ray tomographs.



a

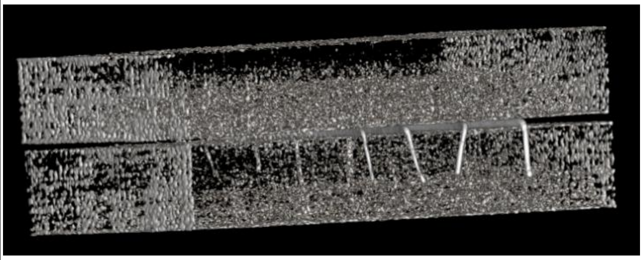
b

Fig. 3. Real object from carbon material with added copper wires: a – calibrated wires with the changed diameter (left-to-right) 0.56, 0.4, 0.36, 0.29, 0.18, 0.1, 0.05 mm; b – assembled sample



0.56	0.4	0.36	0.29	0.18	0.1	0.05

a



b

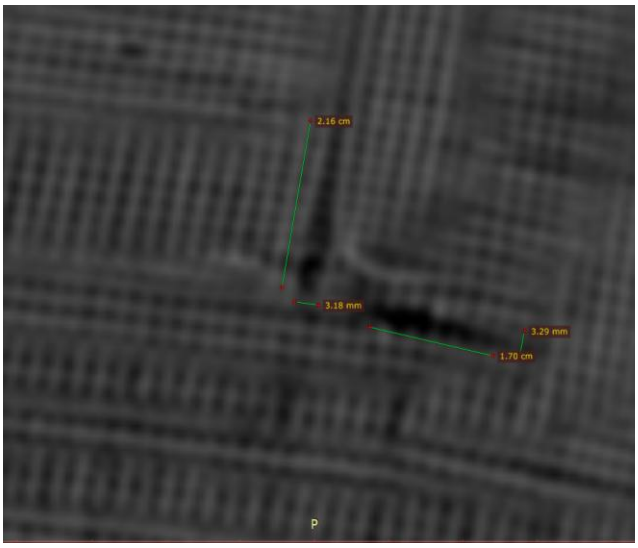
Fig. 4. X-ray image of the sample from carbon material with wires:
a – axial projection with the set of wires with diameter (left-to-right) 0.56, 0.4, 0.36, 0.29, 0.18, 0.1, 0.05 mm;
b – 3D image

Objects with copper wires made of carbon material were examined by method CT. In this case, the object under study did not rotate, but only shifted in the horizontal plane, together with the table tomograph. The experimental conditions were the same as already indicated. The study time for such small objects and the subsequent computer processing of the images was equal to several tens of seconds. Fig. 4 shows X-ray images of the object with wires after reconstruction. Fig. 4,a – axial projection of sample, Fig. 4,b – reconstruction of 3D structure of the object.

During CT it was found that with different projections of the test object the sensitivity of the method and tomograph can change. Thus, when studying the reference sample with wires it was found that maximum sensitivity occurs in the axial projection. Noteworthy, that influence of the tomograph table material on the results of the reconstruction of the image of the sample can be ignored.

In the described mode of the tomograph, a number of carbon materials were investigated. Some of the most obvious defects are shown in the figure. Fig. 5 shows some defects in products from carbon-carbon composite materials which can be identified using CT method. Left Fig. (see Fig. 5,a) shows possible formation in the form of void due to separation of fibers by metal rod inserted into the reinforcing preform. The second projection shows possible defect caused by presence of metal inclusion (see Fig. 5,b).

As can be seen from the Figure, both defects are registered and visualized quite well using the CT method. Moreover, software capabilities make it easy to perform 3D reconstruction of the object and perform spatial visualization and geometric localization of the defect in volume. This test method allows to identify defects and assess their effect on functional properties of the final product.



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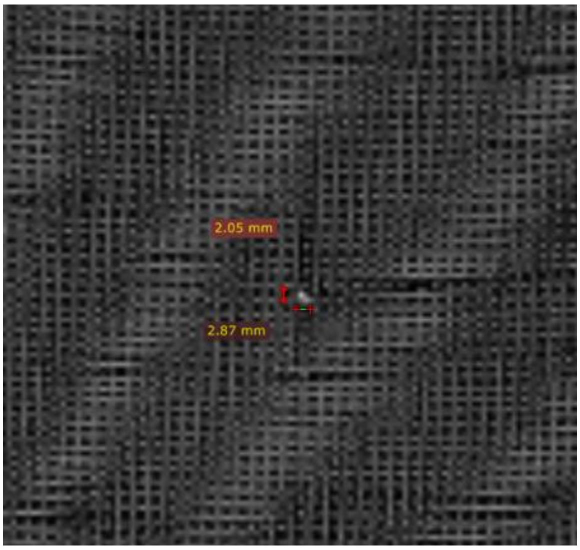

b

Fig. 5. Defects in preform registered using CT method: a – void formation when weaving from carbon filaments, b – metal inclusion

The developed method of using CT for non-destructive analysis was successfully tested on carbon-carbon composite materials, composites with polymer matrix, including the objects that are part of the final product.

CONCLUSIONS

CT method for non-destructive analysis on product made from carbon-carbon materials demonstrated its capabilities in identifying different defects – in the form of gas pores and metal inclusions.

Existing spatial resolution of the method and the tomograph was determined. It was shown that it is no worse than 100 μm. Optimal test conditions were determined on series of samples –optimal combination of scanning parameters, X-ray tube energy value and filters for image reconstruction were selected.

It was demonstrated that samples are not rotated during testing and can have diameter not exceeding the diameter of gantry hole (75 cm) of the unit. Weight of samples can be up to 200 kg, and length during one-time test does not exceed 150 cm. These restrictions are imposed by the physical capabilities of a particular tomograph.

The developed CT method for non-destructive analysis was successfully tested on carbon-carbon composite materials, composites with polymer matrix, including the objects that are part of the final product.

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КТ-ДОСЛІДЖЕННЯ ВИРОБІВ З НЕМЕТАЛЕВИХ КОМПОЗИТІВ, АРМОВАНИХ ВУГЛЕЦЕВИМИ ВОЛОКНАМИ

В.В. Левенець, І.В. Гурін, М.А. Овчинникова, Є.В. Гуріна

Комп'ютерна томографія використовується для дослідження виробів з неметалевих композитів, армованих вуглецевими волокнами. Показано, що в процесі аналізу зразки не обертаються і можуть мати діаметр, що не перевищує портальний отвір (75 см) установки. Вага зразків може бути до 200 кг, а довжина при одноразовому випробуванні не перевищує 150 см. Наведено результати просторового калібрування технології за допомогою набору мідних дротів діаметром від 0,56 до 0,05 мм. У роботі представлено результати виявлення дефектів у виробах у вигляді газових пір та металевих включень.

КТ-ИССЛЕДОВАНИЕ ИЗДЕЛИЙ ИЗ НЕМЕТАЛЛИЧЕСКИХ КОМПОЗИТОВ, АРМИРОВАННЫХ УГЛЕРОДНЫМИ ВОЛОКНАМИ

В.В. Левенец, И.В. Гурин, М.А. Овчинникова, Е.В. Гурина

Компьютерная томография применяется для исследования изделий из неметаллических композитов, армированных углеродными волокнами. Показано, что образцы не вращаются в процессе анализа и могут иметь диаметр, не превышающий портального отверстия (75 см) установки. Вес образцов может быть до 200 кг, а длина при разовых испытаниях не превышает 150 см. Показаны результаты пространственной калибровки технологии с использованием набора медных проводов диаметром от 0,56 до 0,05 мм. В работе представлены результаты обнаружения в изделиях дефектов в виде газовых пор и металлических включений.