The Alternative Procedures of Fiber Volume Ratio Determination of Long-Fiber Carbon – Epoxy Composites

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The need to determine actual fiber volume ratio of the real composite part manufactured in the semi-closed mold led to application of the alternative procedures than the widely used Burn-off method. The low cost one, which is required preferably by any manufacturer, is based on Archimedes law (AL) and the most accurate determination of volume of displaced water. The more complex method which is based on the reverse engineering approach where the 3D scan technique is used to determine the volume of the part. Both are nondestructive methods. And finally the last method mentioned in this study is a sophisticated optical visualization technique and Figure post-processing by mathematical software (it was used NIS Elements and Wolfram Mathematica). Figures for mathematical post-processing were taken using light optical microscope (LOM) and scanning electron microscope (SEM). While better contrast between matrix and fibers is achieved in case of composites filled by transparent fibers, SEM figures provide better image contrast for non-transparent fibers.
The AL and 3D measurement methods are very precise and suitable for orientative control during production, but they give only an average values of the fiber volume ratio in whole product. The mathematical post-processing of image is suitable for laboratory designing of composite part. It is destructive investigation method and image thresholding is time consuming, but on the other hand image processing gives the resultant fiber volume ratio in a particular cross section of the part. That makes the technique a good tool to find the weakest place of the part in meaning of the mechanical properties.

Keywords: Fiber volume ratio, 3D scan, binary image analysis.

1 Introduction

The composites are very promising and useful materials of today and the future. They are characterized by the fact that they consist of two or more components. The main advantage of composites is their high strength at low component's weight. Composites are also characterized by high level of fatigue, they do not deform due to very similar elasticity and ultimate strength.

There are three different type of composites depending on type of used matrix – composites with polymer, metal or ceramic matrix. Polymer matrix composites are the easiest to produce. The reason is that polymer matrix need relatively low processing temperature (20 – 200 °C for epoxy resin). Epoxy resin is very widespread and has long been used as a polymer matrix for carbon fibre composites. [1] Disadvantage of fibre composites could be their anisotropy. [2] Also the delamination of fibre/epoxy composites may appear. [3]

The manufacturing of composites is very complex process with many parameters which have essential influence on the final properties of the composite part. The reinforcement content expressed by fiber volume ratio is one of the main factors influencing the mechanical properties of the composite as the reinforcement is the load carrying constituent. [4, 5] The reinforcement distribution is also important. [6, 7] Based on the manufacturing process the real part value of fiber volume ratio can differ from the value used in design procedure [8]. That is the issue of the compression molding process in semi-closed mold. When the pressure is applied, the extra resin is displaced out of the mold, but the fibers come out sometimes as well. That is not an acceptable degradation effect of the mechanical properties of the part. [9] To prevent such a quality reduction of the part properties, the check of the fiber volume ratio must be done. The purpose of this paper is to describe selected methods used for determination of fiber volume ratio and compare them with each other.

There exists a standard describing so called "burn-off" test. [10] The idea is to let the resin burn off and then to weight the remaining reinforcement. The need given by the circumstances to find another possibility of determination of fiber volume ratio brought us to three respectively four other ways.

The least demanding way how to determine the fiber volume ratio is using the Archimedes law (AL) and scales. Such a "semi-experimental" method is based on weight measurement of the water displaced by the solid body. The weight [in g] and the volume [in cm^3] are measured, the density [in $g{\cdot}cm^{-3}$] of fibers and resin is given in material datasheets. After the evaluation of composite part density, the values of density are inserted into the Eq. 1 [10]

$$v_{fiber} = \frac{\rho_{composite} - \rho_{resin}}{\rho_{fiber} - \rho_{resin}}, \qquad (1)$$

Where:
v_{fiber}... Fiber volume ratio [-],
ρ_{fiber}... Density of carbon fibers [$g{\cdot}cm^{-3}$],
ρ_{resin}... Density of epoxy resin [$g{\cdot}cm^{-3}$],
$\rho_{composite}$... Density of carbon – epoxy composite [$g{\cdot}cm^{-3}$],

The obtained result is the average value of the fiber volume ratio of the composite part. [11]

The problem of the previous method could be the shape complexity or the quality of the part surface. Air bubbles caught in cavities or in surface defects rise the volume of the immersed part. That negatively influences the accuracy of the resultant fiber content of composite. To eliminate such a behaviour the decision to use 3D scanning (3D) is done. The main objective is to determine the part volume more accurately than by method using water container and scales.

The image processing (IP) is more demanding activity. A special software and a quality cross-section figure of investigated material is needed. A good image contrast is necessary for a correct image thresholding and the following area fraction computation.

Two similar methods are used for determination of fiber volume ratio of composite. The first using the NIS Elements software (IP-NIS) for computation, the second using mathematical software Wolfram Mathematica (IP-WM). The basic idea of the first method is to transform original image into binary image and then calculate the area of volume with the binary code inside selected gray-scale interval.

2 Materials and experimental methods

The sample is a composite part made of HR UD carbon (unidirectional high strength carbon fibres) and epoxy resin (C_2H_4O). Fibers are stacked in several layers with 0°, 45° and -45° orientation. The manufacturing process is compression molding at room temperature.

The different methods of metallographic sample preparation are tested for IP. As the most appropriate seems to be wet grinding (paper grid #1200) without polishing. The matrix is blurred during polishing and the fibers are covered by matrix residuum. The visualization of the matrix and composite's reinforcement are performed on the specimen's cross-section using light optical microscope (LOM) Zeiss Axio Imager and using scanning electron microscope (SEM) Tescan Vega SB. The samples for SEM were prepared by the same way as those for LOM. SEM figures were taken using BSE detector at accelerating voltage 30 kV and beam intensity of 14 to achieve a good contrast between phases.

In AL method, the water container is filled with solution of water and wetting agent. The agent helps to better wet the surface of the part. The density of the agent is neglected as the mixing ratio is 200:1. The container is connected by a hose with tank which is placed on the reset scales. The volume of the solid body immersed into the water displaces the water through hose into the tank. The mass of displaced water is known now, as the tank is placed on the scales. As the water density is well known 0.998 g·cm^{-3} at standard temperature (20° C), the volume can be calculated from the measured weight.

The 3D scanning is done by laser scanner ROMER F-41800, type RA-7525SI-2. This device is used for a very accurate scanning in reverse engineering process. The scales used are very sensitive with an accuracy to 2 decimal places. The laser is fastened on the manually operated arm for a slight movement. The surface of the composite part must be covered by a special powder (usually white chalk powder in a spray) for a better scanning of the transparent surfaces which resin is.

IP-NIS is performed using NIS – Elements software. Thresholding is performed manually from binary image and the fiber area ratio is computed. Another approach for finding the fiber volume ratio value is based on detection of fibers and getting the fibers radii in software Wolfram Mathematica – IP-WM. Multiplication of fiber area and number of fibers gives the total area of fibers in the image. This method presumes constant radius of all the fibers in the composite. This condition is accomplished by the manufacturing technology of carbon fibers which produces fibers of equal radii. Similar method is used for computation of thermal conductivity of pultruded profile [12].

3 Results and discussion

3.1 The method using Archimedes law (AL)

The Fig. 1 and 2 show the statistical data processing results of AL method. Measured values of composite's part volume are shown in Fig. 1. The Gaussian distribution is established as the best fitted distribution on the data set (Fig. 2). The mean value of the distribution is 195.6 cm^3 and standard deviation is 0.84. So the probability density function of the distribution is given in Eq. 2

$$f(x) = 0.474513 \cdot e^{-0.707368 \cdot (-195.595 + x)^2} \quad . \tag{2}$$

The part displaced 195.59 cm^3 of water. HR carbon fibers density is 1.81 g·cm^{-3} and epoxy resin density is 1.19 g·cm^{-3}. According formula (1) the resultant fiber volume ratio of composite by AL is 38 %.

The first method used to determine the fiber volume ratio by scales and calculation. Even more simple way would be to use the graduated cylinder. However, the problem is the cylinder scale accuracy affected by water capillarity. Depending on the size of the part, the decision to use scales turned out to be right and more precise.

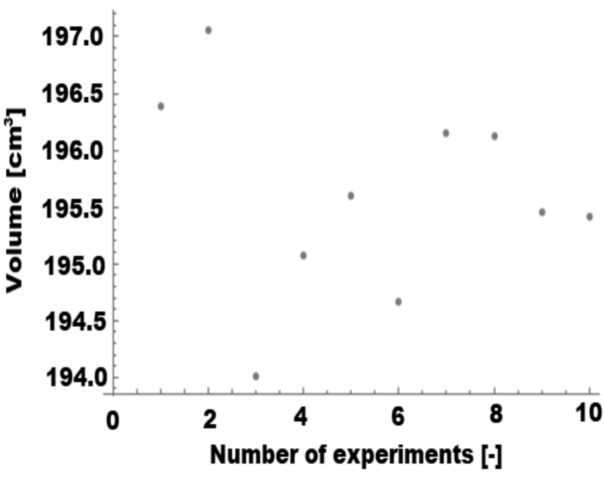

Fig. 1 *Experimental data of measured volume of the composite part*

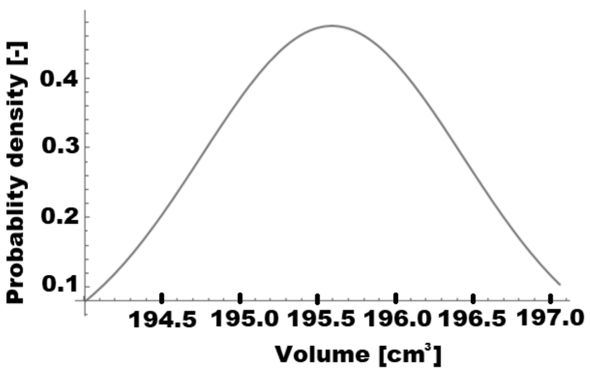

Fig. 2 Probability density of the gauss distribution

3.2 The method using 3D scanning (3D)

There is a geometric deviation map of the scanned part on the Fig. 3. Only the most deviated area of the part is shown. Red values represent positive surface deviation and blue values are for negative deviation. This volume calculation method is not just about the exact value of the volume, but it has additional benefit in determining the location of the undesirable geometric deviation. Such an information is a useful feedback in the "improvement of manufacturing process" procedure.

The volume of the scanned part is 196.5 cm^3. That is about 0.5 % bigger volume than the one set by previous method. As the rest of the values for weight and density are the same, the fiber volume ratio is 36 %. The difference in results of fiber volume of composite acquired by scan and by scales is so small that can be neglected. That makes this "reverse engineering" method applicable in practice as well.

The issue is the surface reconstruction of the scanned part during the reverse engineering process. As the scanned surface is not perfect, the proper creation of the NURBS is the basis for the surface generation and the volume definition of the part. And that is the potential source of the geometrical inaccuracies which can have the same effect on the value of the fiber volume fraction as the bubbles trapped in the surface defects in the AL. The quality of the scan is the issue especially in the case of the shape complexity.

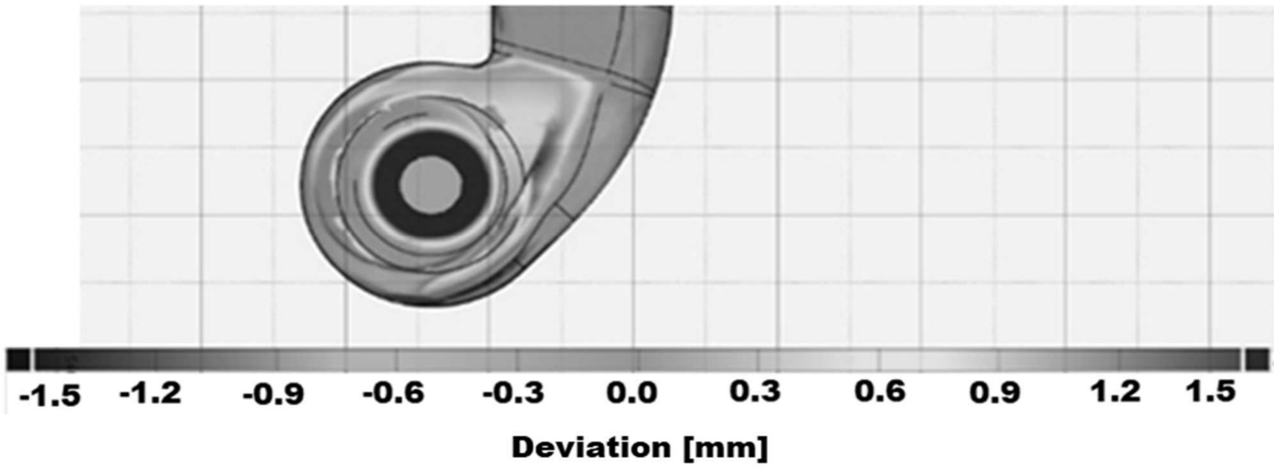

Fig. 3 Deviation map of scanned part

3.3 The method using image processing (IP-NIS and IP-WM)

The visualization of non-transparent carbon fibers in epoxy resin is more complicated in comparison to the visualization of phase layout in composites reinforced by transparent glass fibers. It is very complicated to obtain a sufficient image contrast between the carbon fibers and resin matrix for image analysis and volume fraction computation on the LOM due to a similar reflection's properties of both phases (see Fig. 4). The image contrast is not sufficient enough for software computation due to high level of the background noise.

However, the sufficient contrast is obtained using SEM (see Fig. 5). Epoxy resin (C_2H_4O) and pure carbon fibers (C) have slightly different average atomic number (Z) – C has Z = 6, C_2H_4O has Z = $(2{\cdot}6 + 4{\cdot}1 + 8){\cdot}7^{-1}$ = 3.4. Therefore, the carbon fibers are lighter in imaging using backscatter electron (BSE). The most contrast visualization of carbon fibers in epoxy matrix is obtained using higher current and higher accelerating voltage (HV = 30 kV).

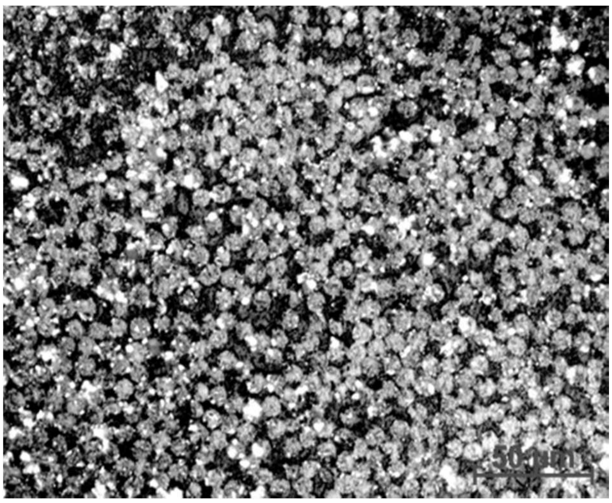

Fig. 4 The visualization of carbon fibres in epoxy resin using LOM – sample ER+C (polarized light)

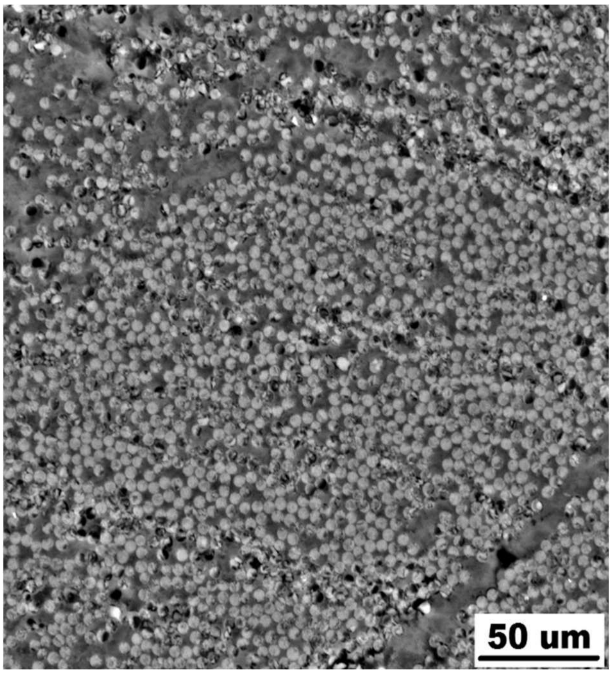

Fig. 5 *The visualization of carbon fibres in epoxy resin using SEM – sample ER+C (BSE, 30 kV)*

The detection of fibers in software Mathematica also includes the binary operation as the first step after that many other Mathematica´s functions follow. It concerns detection of bright areas, gradient filters, watershed transform and others. These functions enhance the binary image so finally only the areas with specific interval of number of pixels are selected. The circle which represents the fibers is inserted into every calculated area center. Some manual work is necessary because some fibers are not detected properly and some duplicated circles had to be deleted. The detail part of the final image with highlighted circles is in the Fig. 6.

The circles don´t align exactly with fibers due to different brightness levels of the objects. Each fiber has lighter and darker areas and the algorithm assigns the center of circle to the brightest area of fiber. Hence in some cases the circles can intersect. Generally, it can be said that the distribution of circles corresponds well to the origin image, the distribution in plane is good and those intersections don´t represent a problem for the following computation.

The threshold value significantly influences the value of volume fraction. The same influence has radius of fibers measured from the image, so finding of a precise value is necessary. The Fig. 7 shows detail of a fiber with the circle on its borders. In this case the chosen circle radius is 8.5 pixels which is 2.14 μm. The fiber volume ratio is then computed to 41.4 %.

The computed value of fiber volume ratio using the software NIS Elements is 45.4 %. It corresponds well to the computed value of the fiber volume ratio of 41.4 % obtained by the image processing using the software Mathematica.

It is obvious that the image processing results are sensitive to computational parameters (threshold value, fiber radius) and the outputs depend on subjective opinion and that is why using of visualization techniques requires well experienced and skillful operator. The problem is a big number of very sensitive parameters which must be set. And a specimen preparation must be done also very precisely.

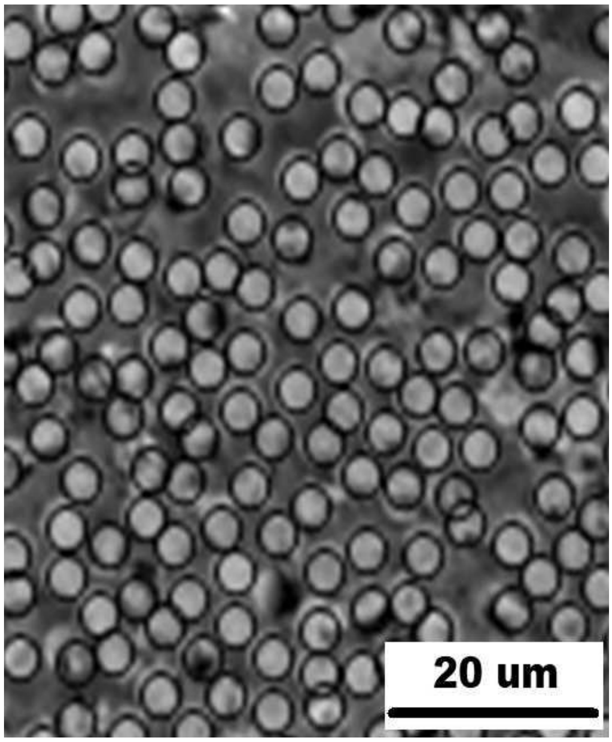

Fig. 6 *The detail of composite with highlighted circles*

4 Conclusion

The fiber volume ratio determined by Archimedes law method is 38 %. The value of 36 % is the result of 3D scan method. Using NIS – Elements SEM image processing gives 45.4 % of fiber content in composite. And finally the second image processing method result is 41.4 %. Theoretical maximum for fiber volume ratio for compression molding process is stated to 40 % [8]. It depends on the manufacturing conditions such as mold shape, type of fabrics and the stacking sequence, etc.

The resultant values of fiber volume ratio for Archimedes law method and 3D scan method are average values for the whole composite part. On the other hand, the image processing allows to set the values of the fiber volume ratio in one individual cross section. So the fiber volume ratio can differ with the place of the art where the cross section is situated, especially in discussed semi-closed mold compression molding process. The local fiber volume ratio is very useful in case one needs to find the weakest place of the part and focus on the structural optimization to get better mechanical behavior. Image processing is also method which may show the places where delamination occurs [13].

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