NDT&E International 86 (2017) 65–72

Contents lists available at ScienceDirect

NDT&E International

journal homepage: www.elsevier.com/locate/ndteint

Nondestructive characterization of fiber orientation in short fiber reinforced polymer composites with X-ray vector radiography

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ARTICLE INFO

Keywords:
X-ray radiography
Injection molding
Fibers
Orientation
Microstructure

ABSTRACT

Short glass and carbon fiber reinforced polymer composites are used in many industrial fields such as in automotive and consumers industry. Their physical and mechanical properties are often superior to those of unfilled polymer components. One aspect being of utmost importance for these properties is the fiber orientation distribution. Here, we present X-ray vector radiography as a method to characterize fiber orientation in short fiber reinforced polymer components. The method is based on X-ray grating interferometry and takes advantage of X-ray scattering caused by the sample's microstructure. Therefore, micro-structural properties can be probed nondestructively without the need for high spatial resolution. Compared to standard X-ray imaging techniques, currently applied for fiber orientation studies, the presented method does not restrict the size of the sample under investigation and allows for much shorter measurement times. In contrast to existing methods, X-ray vector radiography allows the characterization of carbon fiber reinforced polymers despite the weak attenuation contrast between the fibers and the polymer matrix. As this method is also extendable into three dimensions it is a very attractive tool for complex component geometries and carries potential to be applied to materials other than short fiber reinforced polymers.

1. Introduction

The microstructure of a material is a crucial characteristic as it determines the macroscopic behavior of the material such as its mechanical properties. Thus, the need for experimental methods that provide access to this microstructure consistently increases with the development of new materials as well as the advancement of already established materials. Current methods for material and component testing of carbon or glass fiber reinforced polymers (CFRPs or GFRPs) are mainly materialographic specimen preparation, visual examination, and tap testing. Wet chemical analysis can be used to destructively determine porosity and fiber content. Besides visual inspection ultrasonic testing and active thermography are established nondestructive methods [1,2]. In general, the state of the art testing methods, e.g. in the automotive and aerospace industry are ultrasonic testing [3] and acid digestion [2], which are not able to deliver a three-dimensional representation of the components internal structure. As X-rays can penetrate matter they are a valuable tool for analysis of the internal geometry and structure. X-ray radiography and computed tomography (CT) are the two most common techniques applied in order to obtain information on the internal structure of a material in two or three dimensions respectively [4]. Fiber reinforced materials, such as CFRPs or GFRPs, are one class of materials where CT is extensively applied in order to analyze fiber orientation and fiber length distribution [5–7]. Here, the fiber orientation is extracted after the measurement by applying processing algorithms. This requires CT measurements to resolve single fibers with sufficient contrast to the matrix. Because the fiber diameters are in a range between approximately 5–10 μm for CFRPs and 10–20 μm for GFRPs, the necessity for a high resolution results in the following limitations: First of all, the sample's physical dimensions limit the spatial resolution of the CT scan. Therefore single fibers might not be resolvable for large samples. A sample of several centimeters in size has to be divided into several pieces with smaller dimensions, usually below 5 mm. Secondly, the attenuation contrast

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http://dx.doi.org/10.1016/j.ndteint.2016.11.013
Received 13 August 2016; Received in revised form 25 November 2016; Accepted 28 November 2016
Available online 29 November 2016
0963-8695/

between fibers and the surrounding matrix has to be strong enough in order to ensure the robustness of the applied processing algorithm. This is not the case for carbon fibers (1.8 g/cm^3) embedded in a polymer matrix, e.g. polyamide (1.15 g/cm^3). Even though the contrast is higher for carbon fibers and a polypropylene matrix (0.94 g/cm^3) it is still not as high as between glass fibers (2.5 g/cm^3) and the polymer matrix. Hence, for large samples and materials suffering from small attenuation contrast, the CT based fiber extraction techniques are prone to fail. Furthermore, high resolution CT measurements are time consuming because they take up to several hours of scanning time.

Here, we report on a recently developed X-ray imaging technique providing fiber orientation as a physical measure, which does not suffer from the mentioned limitations. It relies on interference effects from grating structures which are placed into the X-ray beam enabling the detection of scattered X-rays [8,9]. These scattered X-rays are represented as the so-called dark-field image. This signal carries information of the sample's microstructure without the necessity to spatially resolve single microstructural features [10,11]. Due to the nature of X-ray scattering, the detected signal is also much more sensitive to differences in the material's electron density as compared to the attenuation contrast [9]. Thus, X-ray dark-field imaging is an effective tool to study microstructures that appear with low contrast in attenuation based CT. Grating-based X-ray dark-field imaging is easily applicable at laboratory X-ray systems and therefore has recently been used in studies of various materials. Besides water-transport and hydration processes in cementitious materials [12,13] the structure of bio-materials such as bone and teeth has been studied based on this method [14,15]. Due to the unidirectional sensitivity of the interferometer for scattered X-rays, the relative orientation between the gratings and the scattering structures influences the detected dark-field signal [16–18]. This property has recently been used to obtain information on fiber orientation in CFRPs and GFRPs [19,20]. Especially for short fiber reinforced polymer (SFRP) components, fiber orientation is a very important characteristic as it is not known prior to the manufacturing process. During the manufacturing process, such as injection molding, the fibers may align according to the flow of the matrix polymer [21]. This behavior results in anisotropic fiber orientations or in the formation of features such as weld lines. Both characteristics strongly influence the mechanical properties of these components. For the latter the mechanical strength is decreased compared to the rest of the injection-molded part [22]. In our study we measured the fiber orientation of various SFRP components made of different sizes, geometries and fiber materials. Our results show that X-ray vector radiography (XVR), which is an extension of grating-based X-ray dark-field imaging (see Materials and methods section), does not suffer from the mentioned limitations of a CT measurement [23,24,14]. It is much faster but still provides results which are qualitatively comparable to CT data. Furthermore, the fiber orientation comes as physical measure and is not extracted via data processing algorithms. We show that weld lines can be easily studied in flat components with this technique while neither the fiber material nor the component size complicates the measurements.

2. Materials and methods

2.1. Sample description

In this work, five pieces of four different injection molded parts, shown in Fig. 1, were investigated. All samples have a fiber content (glass or carbon fiber, respectively) of about 30 wt%. The fiber diameter is about 18 μm for glass and 7 μm for carbon fibers. The samples *S*1 and *S*2 are GFRPs produced by injection molding with a thickness of 2 mm. Density of glass was about 2.52 g/cm^3. The mean fiber length in the granulates before injection molding was 400 μm. During molding however fibers can break and we therefore assume a fiber length of less than 400 μm in the final sample. The true fiber length distribution

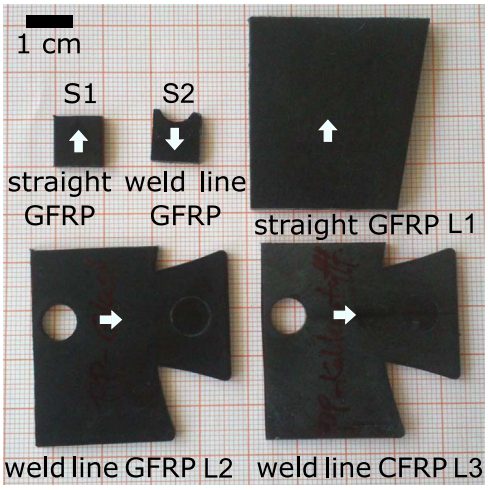

Fig. 1. Photograph showing all samples measured for this study on graph paper. The arrows indicate the flow of the polymer melt during injection molding.

can be verified by CT measurements as described in Ref. [7]. The matrix material was polypropylene with a density of 0.9 g/cm^3. In *S*1, the fibers are expected to be mainly aligned to the direction of injection. *S*2 was cut out around a hole, where the melt front splits in front of the hole and then flows together creating a weld line. The large samples were a straight segment (*L*1, thickness of 4 mm) containing glass fibers and two samples (thickness of 2 mm) with a hole and a weld line containing glass (*L*2) fibers and carbon fibers (*L*3). The samples *S*1 and *S*2 have already been studied by CT and dark-field CT in a previous study [20]. Fig. 1 shows a photography of all samples presented in this study with the arrows indicating the direction of injection.

2.2. CT measurement and parameters

The specimens were scanned with a Nanotom 180NF (GE phoenix | X-ray). This system consists of a nano focus tube and a 2304 × 2304 pixel Hamamatsu detector (Hamamatsu City, Japan) . Molybdenum was used as the target material. The scanning parameters for investigating the samples *S*1 and *S*2 were 80 kV tube voltage, 180 μA tube current, 1800 projections and (6.5 μm)^3 voxel size. Six single images were averaged resulting in one projection. The integration time for each single image was 750 ms. Datos | x was used as the reconstruction software. It works with a filtered back-projection and a beam-hardening correction was applied.

2.3. Experimental setup and measurement parameters for the dark-field measurements

The X-ray vector radiography measurements were performed on a laboratory based symmetric Talbot–Lau interferometer. Fig. 2 shows a schematic of the setup and the measurement geometry. Detailed information on the used gratings, detector and X-ray tube can be found in Ref. [11]. Only the gratings G_1 and G_2 were slightly modified. A new G_2 with a gold lamellae height of 200 μm was used and G_1 was replaced by a grating with 10 μm period and gold as a phase shifting material with a height of 8.3 μm. The acceleration voltage of the tube was set to 60 kVp while the tube power was set to 100 W. In the first experiment all samples shown in Fig. 1 were placed between the phase and analyzer grating, G_1 and G_2, with a distance of 33 cm from the phase grating. This resulted in an image pixel size of 86 μm taking into account the physical detector pixel size of 127 μm. Additionally, the samples *S*1 and *S*2 were placed between the source and the phase grating, G_0 and G_1, for a second experiment. The tube power was set to 20 W and the distance to the phase grating was 55 cm, in this case resulting in an image pixel size of 29 μm. For all the measurements the