Characterization of multilayer structures in fiber reinforced polymer employing synchrotron and laboratory X-ray CT

Oliver Wirjadi¹, Michael Godehardt, Katja Schladitz, Björn Wagner Fraunhofer ITWM, 67663 Kaiserslautern, Germany

Alexander Rack European Synchrotron Radiation Facility, 38043 Grenoble Cedex, France

Martin Gurka, Sebastian Nissle, Andreas Noll Institute for Composite Materials IVW, 67663 Kaiserslautern, Germany

Abstract

Specimens of carbon or glass fiber reinforced polymer (CFRP, GFRP) can be imaged using both conventional laboratory X-ray micro-computed tomography (μ XCT) equipment and synchrotron light sources (μ SCT). The image quality when using intense (partially) coherent synchrotron light is still superior, especially when applying phase-retrieval algorithms. In the resulting volume images, the fiber direction distribution and other mechanically relevant parameters can be determined. In this contribution, we will demonstrate how these fiber direction results can be used to detect regions with locally different fiber orientations in GFRP or CFRP which arise in the molding process of such samples. To this end, we evaluate the three-dimensional fiber orientation tensor locally across the thickness of different specimens. For each resulting individual layer, we can automatically detect the layer thickness and the preferred fiber direction. These methods have been successfully applied to various commercial specimens. We will demonstrate results on volume images of samples from both synchrotron and laboratory μ CT and discuss the specific advantages and disadvantages in this application.

¹ Corresponding author. E-Mail: oliver.wirjadi@itwm.fraunhofer.de, Phone: +49-631-31600-4396

1 Introduction

Due to their high weight-specific stiffness and strength, composite materials play an increasingly important role in lightweight construction. Especially glass and carbon fiber reinforced polymers are now used in a wide variety of applications. In general, parts from such fiber reinforced materials should be produced such that the fibers are aligned with the main loading direction in the final application to ensure high strength and stiffness. Yet, it is known that depending on various parameters of the used fibers and of the injection or molding procedures used to form a fiber-reinforced part, complex and inhomogeneous fiber direction distributions can result [16-18]. These effects seem to be insufficiently understood and simulation software is currently not yet capable to fully describe them. Three dimensional imaging using micro-computed tomography (μ CT) and image analysis may be used to describe the phenomenology and to give insight into the relations between production processes and fiber distribution in the final component.

The goals of the present paper are: First, to provide 3D-image analysis tools capable of extracting mechanically relevant parameters from μ CT-reconstructions of fiber-reinforced polymers; second, to compare laboratory (μ XCT) and synchrotron X-ray micro-tomography (μ SCT) regarding their usability for characterizing carbon fiber-reinforced polymers.

A quantitative correlation of microscopically determined structural parameters (orientation tensor, layer thickness, volume fraction) with mechanical properties needs exhaustive modelling and simulation studies, for which a qualitative starting point will be given with the mechanical analysis presented in this manuscript. The high complexity of such a simulation study follows from the fact that injection-molded GFRP and CFRP with fiber lengths in the mm-regime have a far more complex geometry than high-performance, unidirectional composites. This renders simple, one-parameter descriptions such as the direct relationship between a scalar angle and E-modulus presented in [23] infeasible for our data.

Similar algorithms to ours exist in the literature, e.g. [1-6], and structural studies of fiber reinforced polymers based on μ CT data have also appeared elsewhere, e.g. [23]. Yet, this paper presents to the [Type text]

best of our knowledge the first detailed quantitative comparison of the influence of injection rate on the resulting 3D-microstructure, and the first paper which is able to demonstrate reproducibility of image analysis results using different μ CT technologies for glass fiber-reinforced polymers. Furthermore, we present a novel method to identify and measure the thickness of regions with differently oriented fibers from local fiber orientation measurements.

Among the previously proposed algorithms for measuring fiber orientation in polymers, there are 2D methods [1] and some which do not allow for a quantification of local effects [4]. In contrast, morphological approaches such as [2,3], or filter-based methods [5] would also be applicable in our case. Yet, algorithms using partial gray value derivatives [6] are more closely related to our method, and we expect these could produce similar results as those presented here. Nevertheless, we will use the local estimation of fiber directions based on the three-dimensional (3D) Hessian matrix in this paper, which we have previously described in [7]. This method does not require segmentation of individual fibers and is therefore applicable also at medium resolutions and high fiber density. In the present context, a medium resolution means that the pixel distances in the reconstructed volume images are just below the fiber diameter (typically 10-15 μ m for glass fibers and below 10 μ m for carbon fibers).

Applying µCT to study fiber-structures in polymers does not only introduce challenges in terms of spatial resolving power: additionally, sensitive contrast modes are required in order to depict the only weakly varying density changes in the samples [24, 27]. Here, the usage of synchrotron light with its partial spatial coherence as well as intense photon flux is highly beneficial for µCT. It allows one to obtain images with high spatial resolution as well as high signal-to-noise ratio and, even more important, to exploit the full complex-valued refractive index of the specimen in terms of contrast. Especially the contrast related to the real part of the refractive index, frequently termed X-ray phase contrast, is of high interest as it is orders of magnitude larger for low-Z materials such as polymers then the imaginary part (absorption) [8]. X-ray phase contrast allows one to depict specimens with either negligible absorption contrast or where the absorption contrast between different constituents is

weak, e.g., as in the case of carbon fibers in polymers [7, 24, 25, 26] or even water-enriched regions in hardening cement [19].

References

- C. Eberhardt, A. Clarke. Fibre-orientation measurements in short-glass-fibre composites. Part I: automated, high-angular-resolution measurement by confocal microscopy. Composites Science and Technology, 61(10):1389-1400, 2001.
- [2] K. Sandau and J. Ohser. The chord length transform and the segmentation of crossing fibres. J. Microscopy, 226(1):1365-2818, 2007.
- [3] H. Altendorf, D. Jeulin. 3d directional mathematical morphology for analysis of fiber orientations. Image Analysis & Stereology, 28(3):143-153, 2009.
- [4] M. Kiderlen, A. Pfrang. Algorithms to estimate the rose of directions of a spatil fibre system. J. Microscopy, 219(2):50-60, 2005.
- [5] M. Axelsson. Estimating 3D fibre orientation in volume images. Proc. 19th International Conference on Pattern Recognition, 2008.
- [6] M. Krause, J.M. Hausherr, B. Burgeth, C. Herrmann, W. Krenkel. Determination of the fibre orientation in composites using the structure tensor and local X-ray transform. Journal of Materials Science, 45(4): 888-896, 2010.
- [7] C. Redenbach, A. Rack, K. Schladitz, O. Wirjadi, M. Godehardt. Beyond imaging: on the quantitative analysis of tomographic volume data. Int. J. Materials Research, 103(02): 217-227, 2012.

- [8] A. G. Peele and K. A. Nugent. Phase-contrast and holographic tomography. In: Advanced Tomographic Methods in Materials Resarch and Engineering, J. Banhart (ed.), Oxford University Press, 2008.
- [9] T. Weitkamp, P. Tafforeau, E. Boller, P. Cloetens, J.-P. Valade, P. Bernard, F. Peyrin, W. Ludwig, L. Helfen, J. Baruchel. Status and evolution of the ESRF beamline ID19. AIP Conf. Proc. 1221:33-38, 2010.
- [10] T. Weitkamp, D. Haas, D. Wegrzynek, A. Rack. ANKAphase: software for single-distance phase retrieval from inline X-ray phase-contrast radiographs. J. Synch. Rad. 18(4):617-629 (2011).
- [11] A. Mirone, E. Gouillart, E. Brun, P. Tafforeau, J. Kieffer. PyHST2: an hybrid distributed code for high speed tomographic reconstruction with iterative reconstruction and a priori knowledge capabilities. Nucl. Instrum. \& Meth. in Phys. Res. B (submitted), arXiv:1306.1392, 2013.
- [12] D. Paganin, S. C. Mayo, T. E. Gureyev, P. R. Miller, S. W. Wilkins. Simultaneous phase and amplitude extraction from a single defocused image of a homogeneous object. J. Microsc. 206(1), 33-40 (2002).
- [13] D. Eberly, R. Gardner, B. Morse, S. Pizer, C. Scharlach, Ridges for image analysis. Journal of Mathematical Imaging and Vision 4(4), 353-373, 1994.
- [14] Suresh G. Advani and Charles L. Tucker, The Use of Tensors to Describe and Predict Fiber Orientation in Short Fiber Composites. J. Rheol. 31(8), 751, 1987.
- [15] T. Kanit, S. Forest, I. Galliet, V. Mounoury, D. Jeulin, Determination of the size of the representative volume element for random composites: statistical and numerical approach. International Journal of Solids and Structures 40 (13–14), 3647-3679, 2003.

- [16] A.N. Oumer and O. Mamat, A Review of Effects of Molding Methods, Mold Thickness and Other Processing Parameters on Fiber Orientation in Polymer Composites. Asian Journal of Scientific Research 6(3), 401-410, 2013.
- [17] E. Lafrache, P. Krawczak, J.P. Ciolczyk and J. Maugey, Injection Mouldoing of Long Glass Fibre Reinforced Polyamide 6-6: Guideline to Improve Flexural Properties. eXPRESS Polymer Letters 1(7), 456-466, 2007.
- [18] H. Bijsterbosch and R.J. Gaymans, Polyamide 6 Long Glass Fiber Injection Modings. Polymer Composites 16(5), 363-369, 1995.
- [19] G. Artioli, M.C. Dalconi, M. Parisatto, L. Valentini, M. Voltolini, G. Ferrari, 3D imaging of complex materials: the case of cement: Int. J. Mater. Res. 103 (2012) 145.
- [20] A. Papadimitropoulos, S. Friess, F. Beckmann, P. Salmon, S. Riboldi, D. Hutmacher, I. Martin, B. Müller, Comparative study of desktop- and synchrotron radiation-based micro computed tomography analyzing cell-seeded scaffolds in tissue engineering of bone: Proc. SPIE 7078 (2008), 70780T.
- [21] O. Brunke, K. Brockdorf, S. Drews, B. Müller, T. Donath, J. Herzen, F. Beckmann, Comparison between X-ray tube based and synchrotron radiation based μCT: Proc. SPIE 7078 (2008), 70780U.
- [22] R. Bernhardt, D. Scharnweber, B. Müller, P. Thurner, H. Schliephake, P. Wyss, F. Beckmann, J. Goebbels, H. Worch, COMPARISON OF MICROFOCUS- AND SYNCHROTRON X-RAY TOMOGRAPHY FOR THE ANALYSIS OF OSTEOINTEGRATION AROUND TI6AL4V-IMPLANTS: Eur Cell Mat 7 (2004): 42.

- [23] G. Requena, G. Fiedler, B. Seiser, P. Degischer, M. di Michiel and T. Buslaps, 3D-Quantification of the distribution of continuous fibres in unidirectionally reinforced composites. Composites: Part A 40, 152-163, 2009.
- [24] J. Martin-Herrero and Ch. Germain, Microstructure reconstruction of fibrous C/C composites from X-ray microtomography, Carbon 45 (2007): 1242.
- [25] O. Coindreau, C. Mulat, C. Germain, J. Lachaud, G. L. Vignoles, Benefits of X-Ray CMT for the Modeling of C/C Composites, Adv. Engin. Mater. 13 (2011): 178
- [26] T.-H. Le, P.J.J. Dumont, L. Orgeas, D. Favier, L. Salvo, E. Boller, X-ray phase contrast microtomography for the analysis of the fibrous microstructure of SMC composites, Composites A 39 (2008): 91.
- [27] O. Wirjadi, K. Schladitz, A. Rack, T. Breuel, Applications of anisotropic image filters for computing 2D and 3D-fiber orientations, Stereology and Image Analysis (Proc. 10th European Congress of ISS), eds.: V. Capasso and G. Aletti and A. Micheletti, p. 107-112 (2009)