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Christian Franck · Karen Kasza · Jon Estrada · Rosa De Finis · Geir Ólafsson · Suhasini Gururaja · Jevan Furmanski · Aaron Forster · Pavan Kolluru · Mike Prime · Tom Berfield · Cahit Aydiner *Editors*

Challenges in Mechanics of Biological Systems and Materials, Thermomechanics and Infrared Imaging, Time Dependent Materials and Residual Stress, Volume 2

Proceedings of the 2023 Annual Conference & Exposition on Experimental and Applied Mechanics

SEM

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Conference Proceedings of the Society for Experimental Mechanics Series

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Society for Experimental Mechanics, Inc.,
Bethel, USA

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ISSN 2191-5644 ISSN 2191-5652 (electronic)
Conference Proceedings of the Society for Experimental Mechanics Series
ISBN 978-3-031-50469-3 ISBN 978-3-031-50470-9 (eBook)
https://doi.org/10.1007/978-3-031-50470-9

This Springer imprint is published by the registered company Springer Nature Switzerland AG
The registered company address is: Gewerbestrasse 11, 6330 Cham, Switzerland

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Preface

Challenges in Mechanics of Biological Systems and Materials, Thermomechanics and Infrared Imaging, Time Dependent Materials and Residual Stress represents one of five volumes of technical papers presented at the 2023 SEM Annual Conference & Exposition on Experimental and Applied Mechanics organized by the Society for Experimental Mechanics held on June 5–8, 2023. The complete proceedings also include volumes on: Additive and Advanced Manufacturing, Advancement of Optical Methods in Experimental Mechanics, Dynamic Behavior of Materials, Fracture and Fatigue, Inverse Problem Methodologies, Machine Learning and Data Science, and Mechanics of Composite and Multifunctional Materials.

Each collection presents early findings from experimental and computational investigations on an important area within Experimental Mechanics, the Mechanics of Biological Systems and Materials, Thermomechanics and Infrared Imaging, and Time-Dependent Materials being some of these areas.

The Biological Systems and Materials segment of this volume summarizes the exchange of ideas and information among scientists and engineers involved in the research and analysis of how mechanical loads interact with the structure, properties, and function of living organisms and their tissues. The scope includes experimental, imaging, numerical, and mathematical techniques and tools spanning various length and time scales. Establishing this symposium at the Annual Meeting of the Society for Experimental Mechanics provides a venue where state-of-the-art experimental methods can be leveraged in the study of biological and bio-inspired materials, traumatic brain injury, cell mechanics, and biomechanics in general. A major goal of the symposium was for participants to collaborate in the asking of fundamental questions and the development of new techniques to address bio-inspired problems in society, human health, and the natural world. The 2023 Symposium is the 13th International Symposium on the Mechanics of Biological Systems and Materials.

In recent years, the applications of infrared imaging techniques to the mechanics of materials and structures have grown considerably. The expansion is marked by the increased spatial and temporal resolution of the infrared detectors, faster processing times, much greater temperature resolution, and specific image processing. The improved sensitivity and more reliable temperature calibrations of the devices have meant that more accurate data can be obtained than were previously available.

The Time-Dependent Materials track was organized to address constitutive, time (or rate)-dependent constitutive and fracture/failure behavior of a broad range of materials systems, including prominent research in progress in both experimental and applied mechanics. Papers concentrating on both modeling and experimental aspects of Time-Dependent Materials are included.

The Residual Stress track addressed the state of knowledge in the area of experimental techniques for residual stress measurement and experimental characterization of residual stress effects on the performances of materials and engineering systems.

The track organizers thank the presenters, authors, and session chairs for their participation and contribution to these tracks. The support and assistance from the SEM staff are also greatly appreciated.

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Contents

Development of PEEK Matrix Polymer Composite and Additive Manufacturing by Pellet Extrusion Method

Merve Bagci Bilgen and Alaeddin Burak Irez

Abstract Within the context of this study, it is intended to develop new biocompatible polymer-based composite materials. For this purpose, PEEK (polyether ether ketone) was used as the matrix of the composites, and carbon nanotube (CNT) was used as the reinforcement. The PEEK matrix and 1 wt. % CNTs were melt mixed in a twin-screw extruder to obtain the compound in the form of pellets. Then, using these pellets, composites were manufactured using a pellet extruder type 3D printer. After manufacturing, microstructure of the specimens was observed using optical microscopy, and mechanical characterization was performed through three-point bending tests and Charpy impact tests. The most noteworthy result of the microscopy was the absence of any discontinuity between the layers of the specimen for pure PEEK specimens. Furthermore, the mechanical improvement was not apparent by the CNT incorporation for the tested composites. In conclusion, pellet extrusion is thought as a promising tool for the manufacturing of biocompatible materials for biomedical applications.

Keywords Biocompatibility · Pellet extrusion · PEEK · CNT · 3D printing

Introduction

Technological advancements have a significant impact in all areas. In particular, in the manufacturing industry, modern methods are increasingly being used instead of conventional techniques. In this respect, additive manufacturing, also known as 3D printing, is a growing field. A three-dimensional printer is a machine that produces three-dimensional solid objects from a three-dimensional CAD (computer-aided design) file prepared in the digital environment. With three-dimensional printers, models designed in a digital environment can become objects that can be manipulated and examined in a short time [1]. Three-dimensional printer technologies work with the technique of stacking layers on top of each other. However, the methods of creating these layers may differ. For polymers, the most widely known of these methods are the ones that form solid objects by melting the plastic material [2]. The advantages of three-dimensional printers are as follows: the design is easily transferable/shared as it consists of digital data, the ability to make changes and corrections quickly, the ability to easily produce customized products, the efficiency in terms of investment and production, the price of the product can be calculated prior to production, and the use of recyclable materials, which is equivalent to producing a minimum of material waste [3]. There are many types of additive manufacturing methods, and the FDM (fused deposition modeling) additive manufacturing type is one of the most common ones because this technique is simple, cost-effective, and widely available for many different polymers [4]. Studies have been carried out on the FDM technique for a long time, and this method continues to be developed. Despite the advantages mentioned, the FDM technique has the following disadvantages: low extrusion speed and lack of enclosed workspaces to apply this technique to create a large prototype and processing thermoplastic materials such as PEEK (polyether ether ketone) through FDM is a difficult process due to the high melt viscosity. To overcome these challenges, the pellet extrusion technique was used in this project [5]. In pellet extrusion, instead of using the polymer in the filament form with a specific diameter and stiffness, the polymer pellets are fed directly into the extruder of the 3D printer. This offers freedom in the material composition, and it is possible to achieve customized material properties. In this study, PEEK matrix composites were developed to meet the requirements of a wide variety of applications. PEEK is a semi-crystalline thermoplastic used in high-performance engineering applications. Compared to

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C. Franck et al. (eds.), *Challenges in Mechanics of Biological Systems and Materials, Thermomechanics and Infrared Imaging, Time Dependent Materials and Residual Stress, Volume 2*, Conference Proceedings of the Society for Experimental Mechanics Series,
https://doi.org/10.1007/978-3-031-50470-9_1

other thermoplastics, PEEK has very good thermal stability and excellent resistance to chemicals, solvents, and hot water and can be used continuously for very long periods of time up to 250 °C in air. PEEK also has an important deformation capacity and excellent impact resistance. Besides, the crystalline nature of PEEK provides its high temperature resistance [6]. Similar to many other thermoplastics, the mechanical properties of PEEK can be enhanced by nano-reinforcements such as carbon nanotubes (CNTs) [7]. CNTs exhibit a very high elastic modulus of more than 1 TPa and a strength 10–100 times that of the strongest steel. CNTs also have excellent thermal and electrical properties [8].

Within the context of this study, it is intended to develop new biocompatible polymer-based composite materials that can be utilized in biomedical applications. To this end, PEEK was used as the matrix of the composites, and carbon nanotubes (CNT) were used as the reinforcement. The PEEK matrix and 1 wt% CNT were melt mixed in a twin-screw extruder to obtain the compound in the form of pellets. Then, from these pellets, the composites were fabricated using a pellet extruder type 3D printer. After fabrication, the microstructure of the specimens was observed by optical microscopy, and mechanical characterization was performed by three-point bending tests and Charpy impact tests.

Materials and Methods

In this study, the materials to manufacture the composites were provided by different companies. First, PEEK was supplied from Evonik® under the commercial name VESTAKEEP 2000 G in the form of pellets. It has a density of 1.3 (g/cm^3) and a melting temperature of 340 °C. Next, 0.7 wt% COOH-activated carbon nanotubes (CNT) were provided by Nanografi®, Turkey. The CNT has a density of 2.4 (g/cm^3), a surface area of 65 m^2/g, an outer diameter of 28–48 nm, and a length of 10–25 μm.

After the materials were procured, the composites were manufactured using the additive manufacturing technique of pellet extrusion. Due to the nature of this technique, the composite pellets must be obtained before the specimens are printed. Therefore, the composite pellets were obtained through an extrusion process. Prior to the extrusion process, the PEEK pellets were dried for 3 h in a vacuum oven at 150 °C to remove moisture that may have been trapped due to storage conditions. Then, the composite pellets were fabricated using a Kökbir Makina® co-rotating twin-screw extruder with an L/D ratio of 22, a screw diameter of 12 mm, and a screw speed adjusted to 60 rpm. The twin-screw extruder consists of six zones, and in order to reach the melting temperature of the materials used, the temperature values were set to 47.5 °C for the feed zone, 325 °C for the first zone, 330 °C for the second zone, 340 °C for the third and fourth zones, and 360 °C for the extruder exit. At the exit of the extruder, after the compound has cooled down, the composite is obtained in the form of a filament, and, with the help of a rotary cutter, it is transformed into granules. In the next step, using the pellets from the extrusion process, composites are manufactured using the Yizumi® SpaceA-900-500-S 3D printer. In this 3D printer, the screw diameter was 16 mm, and the screw speed was set to 200 rpm. Regarding the operational parameters of the 3D printer, the extruder temperature was 400 °C, the nozzle diameter was 2.5 mm, the layer thickness was 1.6 mm, and the printed material width was 3 mm. After the composites were fabricated, experimental characterizations were performed.

In the experimental characterizations, the effect of CNT reinforcement as well as the effect of printing direction was examined. The fundamental mechanical properties were identified by three-point bending (3PB) tests. The 3PB tests were performed using a Testometric testing machine according to ASTM D790 test standard, and the crosshead speed was imposed at 10 mm/min. Next, the impact strength of the composites was investigated using a Zwick Roell HIT5P Charpy Universal Tester according to ASTM 6110-10. Each characterization was done using at least five specimens and the standard deviations are given in the results. In addition, specimens from two different composite groups were sectioned and cold mounting was performed. Then, the mounted composites were polished for optical microscopy to observe the microstructure. In addition, scanning electron microscopy (using Tescan Vega 3 SEM) was conducted to observe the damage mechanisms on the fracture surfaces of the failed specimens.

The specimens for the bending tests are given in Fig. 1. The light-colored specimens are pure PEEK specimens, while the black specimens show the CNT-incorporated PEEK matrix composites. In addition, the printing direction can be seen in Fig. 1; Fig. 1b, d shows the longitudinal printing direction, while the others show the transversal printing direction.