

NOVEL RHEOLOGICAL MEASUREMENTS TO UNDERSTAND STRUCTURAL STABILITY OF DIW-PRINTED EPOXY COMPOSITES DURING THERMAL CURING

Stian Romberg, National Institute of Standards and Technology (NIST), Gaithersburg, MD, USA
stian.romberg@nist.gov

Paul Roberts, NIST, Gaithersburg, MD, USA

Sarah Lehrman, NIST, Gaithersburg, MD, USA & University of Maryland, Baltimore County, Baltimore, MD, USA

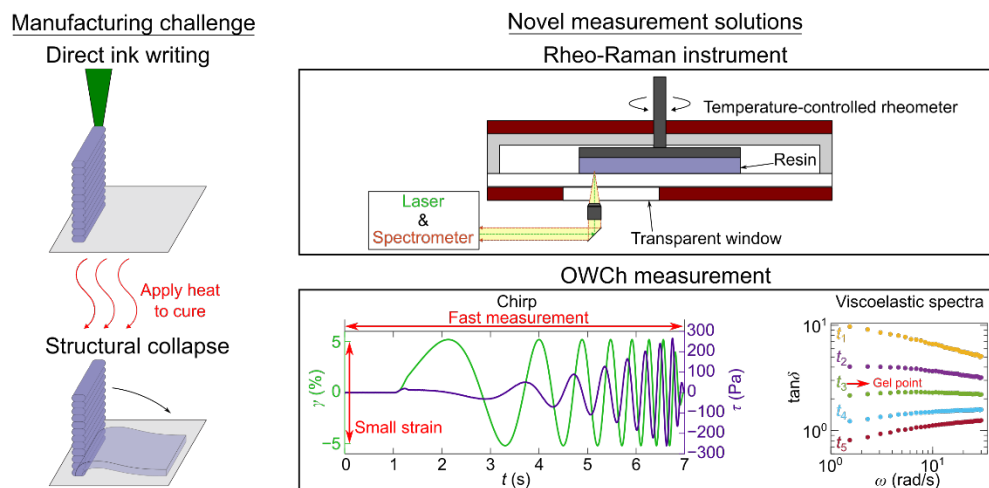
Chad Snyder, NIST, Gaithersburg, MD, USA

Jonathan Seppala, NIST, Gaithersburg, MD, USA

Anthony Kotula, NIST, Gaithersburg, MD, USA

Key Words: Direct ink write, thermoset composite, rheology, Raman spectroscopy, curing

Current literature frequently presents geometrically complex thermoset composites with excellent chemical, mechanical, and thermal stability produced via material extrusion. However, non-empirical strategies to avoid structural collapse during curing are rarely presented, because traditional measurement techniques are not equipped to directly relate temperature and conversion to the rheological properties responsible for structural stability. Further, identifying the moment at which the printed material is no longer susceptible to self-weight structural collapse (i.e., the chemical gel point) is not straightforward. Traditional definitions of the chemical gel point are difficult to apply to these physically gelled composites due to their unique rheological profile and rapid curing rate. This gap in literature may be addressed with two recently developed measurement technologies – the rheo-Raman instrument and Optimally Windowed Chirp (OWCh) measurements. In 2016, Kotula et al. developed a rheo-Raman instrument [1] to simultaneously capture rheological measurements and Raman spectra, which can be used to measure conversion. This technique provides direct relationships between rheological properties, conversion, and temperature without introducing ambiguity created by taking measurements on separate samples in different instruments. These measurements can be used to determine the conversion at which key rheological transitions occur, like the chemical gel point, provided that we can accurately identify where the gel point occurs for printable thermoset composites. In 2018, Geri et al. presented a way to find the gel point using OWCh measurements, which impart a frequency-varying sinusoidal strain to quickly obtain the viscoelastic spectrum without applying high strains [2]. Printable thermoset composites often have modest linear viscoelastic regimes and cure rapidly, making OWCh measurements ideal for tracking the evolution of the viscoelastic spectrum during curing. The resulting time-resolved viscoelastic spectra help to understand the solidification process of these materials that do not conform to the traditional definitions of the chemical gel point.



[1] A.P. Kotula, M.W. Meyer, F.D. Vito, J. Plog, A.R.H. Walker, K.B. Migler, 2016. Rev. Sci. Instrum. 87(10), 105105. <https://doi.org/10.1063/1.4963746>.

[2] M. Geri, B. Keshavarz, T. Divoux, C. Clasen, D.J. Curtis, G.H. McKinley, 2018. Phys. Rev. X 8(4), 041042. <https://doi.org/10.1103/PhysRevX.8.041042>.