Karlsruher Institut für Technologie

Laboratorium für Elektronenmikroskopie

Master Thesis

4D-STEM analysis of beam-sensitive 3D printed glass precursor and its thermal conversion

Motivation

Combining two-photon polymerization (TPP) and post-annealing has opened a sinterless, low-temperature route to the 3D printing of free-form ceramic nanostructures[1]. This work uses polyhedral oligomeric silsesquioxane (POSS) resin as the precursor to the fused silica glass. However, the exact transformation mechanisms of the precursors to the final inorganic materials are still not well understood. Four-dimensional scanning transmission electron microscopy (4D-STEM) has emerged as an extremely powerful technique for the collection of a wealth of information about the nanoscale structure of a material. With many TPP-derived inorganic materials relying on similar organic cross-linkers, cryogenic 4D-STEM is highly recommended due to the electron beam sensitivity of the polymer compounds and significant improvement in signal-to-noise ratio [2].

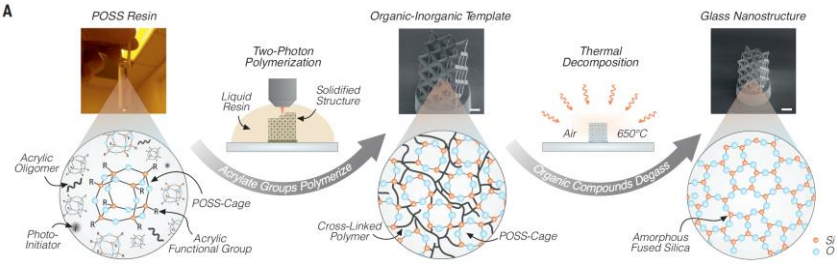

Figure 1: Schematic synthesis through TPP 3D printing and subsequent thermal treatment at 650°C [1].

Tasks

- 3D printing of precursor architectures for analytical TEM and *in situ* thermal conversion investigation.
- Characterization of the nanostructures of POSS-glass precursor by electron microscopy techniques, e.g. STEM-in-SEM and 4D-STEM.
- *In situ* thermal conversion of 3D printed nanoarchitectured glass.

Timeline

- **1. - 2. month**: Introduction to SEM, TEM as well as 3D printing operation, literature and book review to learn this cutting-edge topic, tailoring and preparation of 3D printed precursor architectures.
- **3. - 6. month**: Test of the 4D-STEM technique on 3D printed glass precursor samples with different imaging conditions and data evaluation
- **7. - 8. month**: *In situ* thermal conversion of glass precursor samples by ESEM and post TEM characterization.
- **9. - 12. month**: Preparation of figures and results, writing the thesis, presentation in LEM seminar.

Reference

[1] Bauer et al., Science 380, 960–966 (2023).
[2] Donohue et al., iScience 25, 103882 (2022).

Goals:

- Revealing nanostructures of POSS-glass precursor by 4D-STEM technique.
- Characterization of the organic-to-inorganic transformation by *in situ* environmental SEM study.
- Evaluation and interpretation of the results in collaboration with Prof. Dr. Jens Bauer's group INT.

What you will learn:

- 3D printing direct laser writing (DLW).
- Applied electron microscopy, e.g. SEM, HRTEM, STEM, 4D-STEM for microstructural analysis.
- Image and data evaluation
- Interpretation and presentation of the results

What you bring:

- Studies in physics, materials science, or similar disciplines
- Independent work (after initial guidance)
- Motivation to learn new topics and to work with Python and Jupyter notebooks

Starting date:

- as soon as possible

Contact:

- Qing Sun, qing.sun@kit.edu
- Dr. Martin Peterlechner, martin.peterlechner@kit.edu
- Prof. Dr. Yolita Eggeler, yolita.eggeler@kit.edu

http://www.lem.kit.edu/