Title: Preparation of polyimide aerogel beads using phase separation techniques and supercritical drying

Authors: Oleg Greyz; Bernhard Seifried

Presenter Organization: Deutsches Zentrum für Luft- und Raumfahrt e.V. (DLR)

Porous materials are used in a wide range of applications, such as membranes, catalysts, fuel cells, and thermal insulators, which all rely on tuned pore architectures to achieve the desired balance of permeability, selectivity, surface area, and mechanical performance.

Aerogels are a very special type of open-porous materials with very large specific surface areas ranging from tens to hundreds of m^2/gram, which are typically generated by sol-gel methods from inorganic materials, biopolymers or synthetic polymers followed by solvent exchange and supercritical drying. Several studies reported the fabrication of aerogels also via various phase separation techniques with comparable properties to conventional sol-gel approaches.

In the phase separation approach, a homogeneous polymer solution is driven out of equilibrium so that polymer-rich and polymer-lean phases separate, ultimately solidifying into a porous structure. The specific route by which phase separation is induced, for example either by contacting the solution with liquid non-solvent or vapor, strongly influences the resulting morphology, enabling a broad spectrum of pore sizes and distributions.

This study presents the preparation of polyimide (PI) aerogel beads based on the exposure of PI polymer solutions to a liquid non-solvent via the non-solvent induced phase separation method (NIPS). To fine tune the morphology and thus properties of the PI aerogels, such as the specific surface area, the PI solutions in either DMF or NMP were primed with various levels of an additional antisolvent, such as water, prior to dripping the PI solutions into a non-solvent coagulation bath containing anhydrous ethanol leading to the formation of polymer PI beads. The PI beads were then solvent exchanged and washed with ethanol and dried by either supercritical drying or atmospheric pressure oven drying. The Specific Surface Area (SSA) and Scanning Electron Microscope (SEM) images were generated and compared.

This study demonstrates that priming the PI solution and selecting the drying method are useful tuning knobs for fabricating PI aerogel beads with a wide range of morphologies and different surface areas.