Crossing borders of material science – a new approach of aerogel preparation for electron microscopy

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Project Overview

Aerogels are innovative materials exhibiting a porosity of up to 99.98% demonstrating a wide range of possible applications. To get a better understanding of the morphology of these materials, electron microscopy proves to be a valuable analytical method. Since classic metallographic embedding techniques are not suited for organic aerogels, new approaches are necessary to enable more efficiency and possibilities of image and data acquisition. With regard to electron microscopy, organic aerogels show a behavior similar to biological samples. Therefore, a possible approach is to adopt biological preparation techniques to material science samples where conventional techniques find their limits to image and measure the aerogels' pore sizes, inter-connection and morphology. Resins used to embed of biological samples are comparable to organic aerogels constituents with regard to mechanical properties, they don't shrink during hardening and infiltrate the sample effectively.

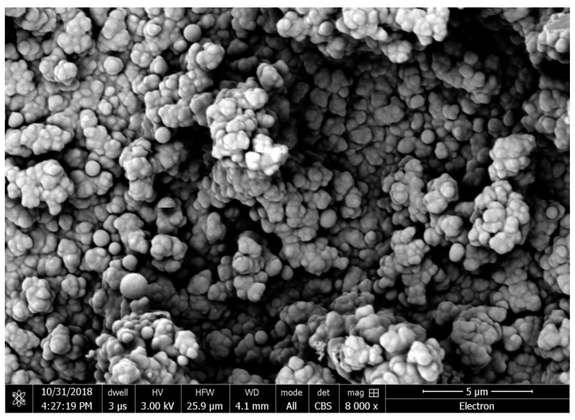

Fig. 1: SEM image of a RF-Aerogel

Preparation and Embedding

The EM preparation of the aerogel starts with the choice of an appropriate resin to fill the pores and to avoid deformations of the aerogel-resin block while drying. Additionally, the hardness of the resin needs to be similar to the one of the aerogel to prevent tensions.

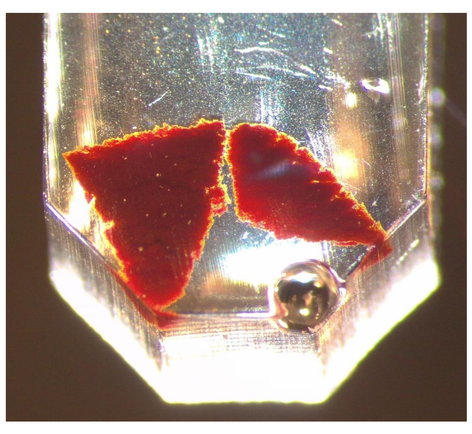

Fig. 2: RF-Aerogel embedded with Embed-812

For further processing and analysis by SEM, FIB or TEM, the sample is trimmed with an ultra microtome to expose the aerogel to the surface.

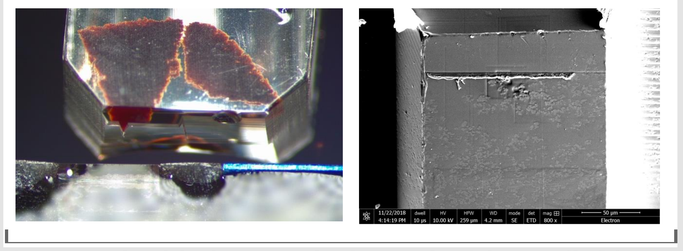

Fig. 3: Embedded aerogel trimmed with UM (left) and SEM image of the trimmed surface (right)

SEM Results

By the conventional preparation dispersing particles on a SEM stub, pore-sizes between particles are just estimations because of the missing depth information in the two-dimensional projection of the SEM image. With a sample embedded and prepared, the distance between particles can be measured

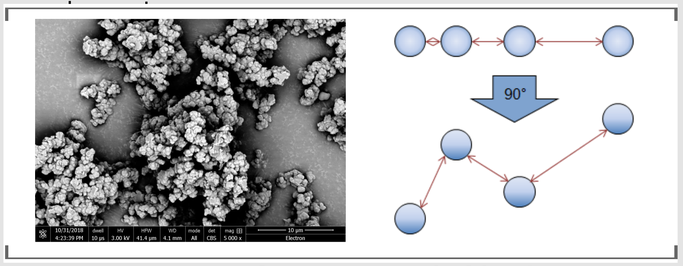

Fig. 4: SEM view of an aerogel (left). The particles seems to be next to each other, but lie in different heights like in the schematic drawing (right).

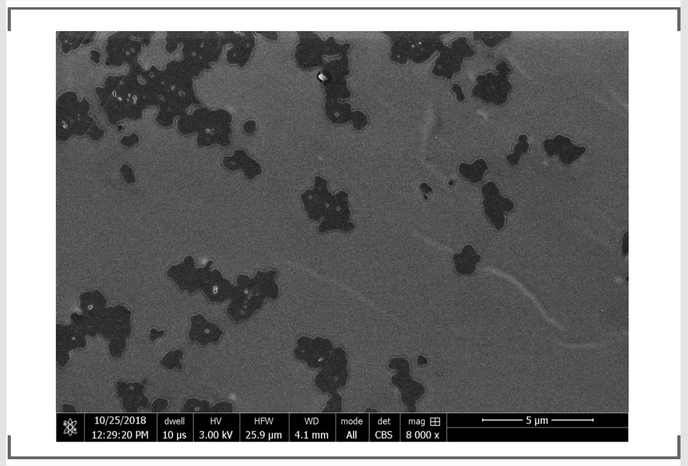

Fig. 5: SEM image of an embedded aerogel section in within a sample

FIB Tomography

By creating a slice-and-view tomogram, it is even possible to analyze the structure of a whole volume, acquiring up to 2000 SEM images in one measurement forming one dataset, thus enabling the reconstruction of the pore -size, -morphology as well as structure and the volume of the aerogel. Additionally, a 3-dimensional reconstruction of these images can be visualized.

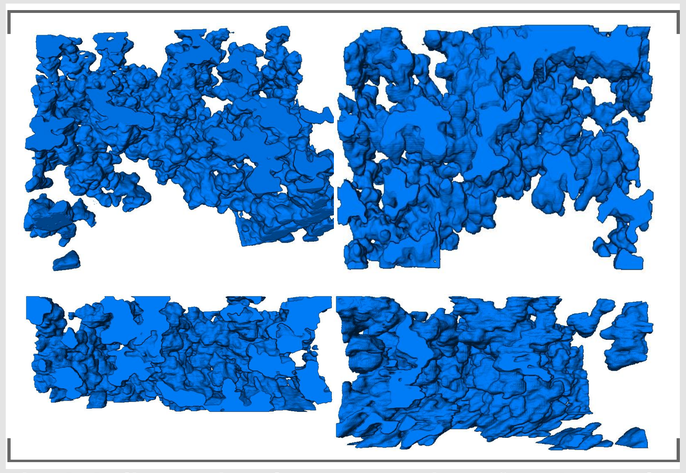

Fig. 6: 3-dimsensional reconstruction of an Aerogel based on a FIB tomogram from different viewing angles

Furthermore, each image can automatically be analyzed for its ratio between micro-pores (literally resin) and aerogel particles. These ratios can be plotted as a graph (fig.7) to characterize the distribution between pores and aerogel particles. Furthermore, the calculated mean value of this graph gives total volume ratio of aerogel and micro-pores into the complete volume measured .

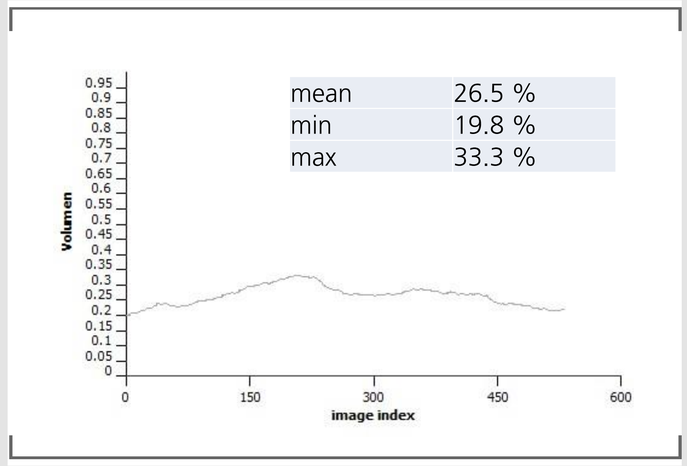

Fig. 7: Ratio between aerogel and micro-pores from an RF-aerogel from 550 images.

TEM results

Some aerogels, like carbon aerogels, exhibit nano-pores that are too small for SEM analysis. Therefore, TEM imaging is an important tool for the investigation of these aerogels, especially for morphology characterization and analysis. With a sample prepared conventionally, it is difficult to separate the individual particles (fig. 8).

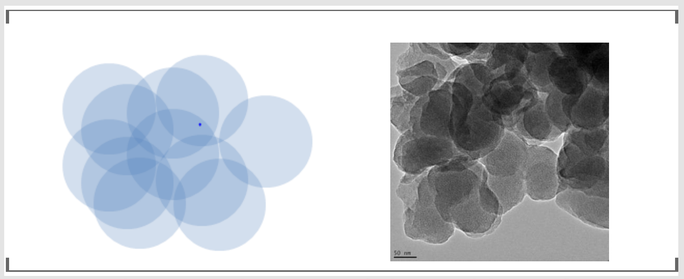

Fig. 8: Schematic drawing of the problem of overlapping particles (left) and a TEM image of an C-aerogel (right).

With an embedded sample, it is possible to prepare thin slices with an UM or to lift out lamella with FIB, thin enough to analyze them in TEM. Therefore, it possible to analyze the morphology and the nano-pores of each particle (fig. 9) without the shortcoming of overlapping information.

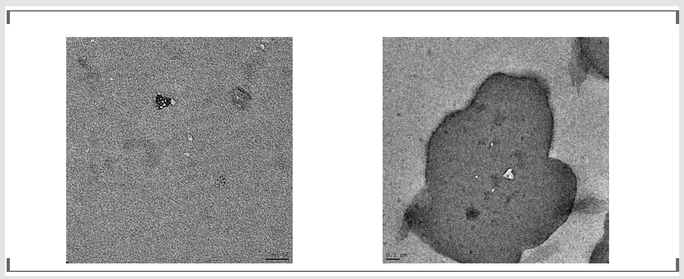

Fig. 9: morphology and nano-pores of an RF-aerogel from and UM-prepared section.

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