Structural investigations on resorcinol-formaldehyde aerogels and carbon aerogels by means of holotomography

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EXPERIMENTAL

Preparation of aerogel samples for holotomography:

2. Carbonization yielded monolithic stiff and flexible carbon aerogels.
3. Characterization revealed particle size (scanning electron microscopy), porosity (picrometry) and surface area (nitrogen sorption isotherms).
4. Suitable milling/abrasive grinding yielded small particles that retained the properties of the monoliths (as indicated by scanning electron microscopy and nitrogen sorption).
5. The aerogel powder was transferred to thin glass capillaries (100µm diameter), sealed with paraffin wax, and glued onto sample holders for nanotomography.

Flexible carbon aerogels could be visualized for the 10 nm voxel size experiments. The computed porosity accounts for approximately half of the porosity and the surface area as determined by picrometry and nitrogen sorption (BET surface area).

For flexible RF aerogels, experiments with 60 nm voxel size were computed to account for a large fraction of the total pore volume (72 % porosity), but only approx. 20 % of the surface area. So, although mesopores seem to account for only a quarter of the total pore volume, the surface area is still dominated by mesopores (80 % mesopores and small macropores).

Additionally, flexible RF aerogels could be evaluated in terms of pore size distribution. Our processing resulted in B317 macropores with average diameter >60µm within a box of 15 µm edge length. This size distribution of these macropores was then calculating a multidimensional distribution of pores in the sub-micrometer level. Since many pores appear at the size of one voxel (here: 60 nm), a higher-resolved structure would be needed in order to discriminate the size of the smallest macropores further.

RESULTS & DISCUSSION

CONCLUSION

Holotomography could successfully be applied to resorcinol-formaldehyde and carbon aerogels yielding 3D-structural data in the higher nanometer range. Therefore, macropores could be visualized, and the porosity and surface area were computed. In the case of flexible resorcinol-formaldehyde, the (macro)pore size distribution could be calculated and pointed towards a multidimensional size distribution at the macropore level.

The pore size distribution of carbon aerogels is subject to current investigations. Further studies will be devoted to extracting further data from experiments with 10 nm voxel size.

REFERENCES

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