The ASAXS technique for analyzing structure and compositions, along with its applications in the field of catalysis

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The Small-Angle X-ray Scattering (SAXS) is a non-destructive method for the analysis of nano-structures in a wide variety of inorganic, organic, and biological materials. This method allows determining averaged structural parameters on a length scale from just above the atomic size up to about 100 nanometer in multicomponent systems and for amorphous materials also. Such structural parameters are the sizes, size distributions, volume fractions and inner surface sizes. SAXS has an increasing importance because many functional properties are getting nanosized dependent. Moreover, Anomalous Small-Angle X-ray Scattering (ASAXS) exploits the anomalous dispersion of the scattering amplitudes near the X-ray absorption edges of structural crucial elements that are contained in the sample [1, 2]. These element sensitive contrast variations are useable to analyze averaged composition fluctuations on the nm scale.
After an introduction into the methods of SAXS and ASAXS some technical and practical details of the ASAXS dedicated instrument at the HZB synchrotron BESSY II will be presented.
In the subsequent section, select material science and inorganic chemistry applications are chosen to exemplify various facets and advantages of SAXS and ASAXS.
The shift towards an economy reliant on renewable energy is accelerating as society endeavors to discover sustainable green energy alternatives to replace fossil fuels. Storing and transporting excess energy generated during periods of low demand as hydrogen is becoming increasingly attractive and essential [3].

Commercial applicability of proton exchange membrane fuel cells (PEMFC) is exclusively bound to the employment of expensive and rare platinum. However as, recently successfully demonstrated, selenium modified ruthenium-based catalysts also exhibit high catalytic activity for the oxygen reduction reaction (ORR) in fuel cells. An ASAXS experiment have been performed to clarify the structural and chemical features of these catalytically active Se-modified ruthenium nanoparticles. Taken all results into account (including the results from XRD, XPS, TEM and EXAFS) a structure model of the catalytically active metallic nanoparticles has been deduced, suggesting a nearly spherical ruthenium particles decorated with smaller selenium aggregates.

As an 2nd example, we will showcase the ASAXS analysis of the catalyst utilized in trials involving anion exchange membrane fuel cells (AEMFCs), wherein the main factor augmenting performance stemmed from the superior meso- and macroporosity established by the hard template [4].
This presentation will elaborate on the advantages of ASAXS in the analysis of catalyst nanomaterials for applications in energy technology.

References
[1] H. B. Stuhrmann, 1985, *Advances in Polymer Science*, 67, (123pp).
[2] A.Hoell, D. Tatchev, S. Haas, J. Haug, P. Boesecke, On the determination of partial structure functions in small-angle scattering exemplified by Al89Ni6La5 alloy, J. Appl. Cryst. (2009). 42, 0, doi:10.1107/S0021889808042453.
[3] I. Staffell, D. Scamman, A.V. Abad, P. Balcombe, P. E. Dodds, P. Ekins, N. Shah, K.R. Ward, The role of hydrogen and fuel cells in the global energy system, Energy Environ. Sci. 12 (2019) 463–491. doi.org/10.1039/C8EE01157E.
[4] P. Teppor, R.Jäger, M. Koppel, O. Volobujeva, R. Palm, M. Månsson, E. Härk, Z. Kochovski, J. Aruväli, K. Kooser, S. Granroth, T. Käämbre, J. Nerut, E. Lust, Unlocking the porosity of Fe–N–C catalysts using hydroxyapatite as a hard template en route to eco-friendly high-performance AEMFCs, Journal of Power Sources, 591, 2024, 233816, doi.org/10.1016/j.jpowsour.2023.233816.