STRUKTURA 2013

Češkovice, 9. 9. - 12. 9. 2013

Session I, Monday, September 9

L1

INTRODUCTION TO SAXS (SMALL-ANGLE X-RAY SCATTERING), INSTRUMENTATION, APPLICATIONS

J. Ilavský

Advanced Photon Source, Argonne National Laboratory, Argonne, IL 60439, USA
ilavsky@aps.anl.gov

Small-angle scattering of X-rays or neutrons is time tested technique suitable for characterization of microstructural features from few Angstroems to tens of microns and larger. While well known by name and principle, it is, unluckily, often misunderstood or even abused by current generations of scientists. And that at time when, at least from point of view of SAXS beamline scientist, the field of applications broadens up tremendously. Clearly, the education of the principals and methods in current university curriculum on SAXS and SANS is lacking. This is pity as the advancement in X-ray and neutron sources, instrumentation, and detectors - combined with the latest developments in computer modeling - provide tremendous opportunities and capabilities. Current systems can collect broad range of data (up to five decades in sizes), can measure in very short times (nano seconds and shorter), have sensitivities unheard of just few years ago etc. Current experiments can be performed at wide range of wavelengths and with tremendous flux - both on synchrotrons as well as high flux neutron sources. And latest developments in desktop X-ray optics have also resulted in major improvements of desktop SAXS capabilities. The lecture will attempt to summarize the basics of small-angle scattering, current capabilities available on new desktops and at advanced facilities, and present examples of applications from speakers experience. We will discuss methodology, including instrument and radiation selection, absolute intensity calibration, and sample preparation. Software for data reduction and analysis will be briefly listed and summarized. While presentation in the allocated time cannot substitute proper college level course or specialized courses offered routinely by large facilities, it should be a good start for novices and, hopefully, enjoyable and entertaining event for present SAXS/SANS experts.

USAXS/SAXS/WAXS INSTRUMENT FOR MATERIALS RESEARCH

J. Ilavský[1], A. J. Allen[2], F. Zhang[2], G. G. Long[2], P. R. Jemian[1], L. E. Levine[2]

[1]*Advanced Photon Source, Argonne National Laboratory, Argonne, IL 60439, USA*
[2]*National Institute of Standards and Technology, Gaithersburg, MD 20899, USA*
ilavsky@aps.anl.gov

Practical engineering materials typically exhibit complex microstructures spanning many decades in size. These are often responsible for the in-service properties. Advances and developments for new or improved materials require a detailed understanding of these microstructures over their entire operative size range. Complete characterization over the size range is usually complicated by the need to combine multiple techniques such as SAXS, SANS, electron or optical microscopy, tomography, etc. In this context, the Bonse-Hart type USAXS instrument at APS [1] with its wide range of length scales characterized during one measurement was already a unique tool for the quantitative, statistically representative, characterization of material microstructures relevant to a wide range of scientific applications. To address the needs of the user community, we have recently upgraded the USAXS instrument through increases in the measurable ranges of both scattering vector (q) and scattering intensity [2]. At large q values, the combination with He-filled pinhole SAXS (pinSAXS) and in-air WAXS cameras improves the data quality by increasing sampling statistics and signal-to-noise sensitivity. More importantly, it extends the contiguous scattering vector q range coverage up to 6 $Å^{-1}$ (energy dependent). The need for environmental chambers to simulate *in-operando* measurements requires higher energy X-rays that minimize window absorption and reduce the exit angles. To reach X-ray energies between 20 keV and 30 keV, the USAXS instrument uses higher order Si(440) instead of Si(220) optics. The narrower Si(440) rocking curves also reduce q_{min} to 3 x10^{-5} A^{-1}. Currently, the combined USAXS/SAXS/WAXS instrument at the ChemMatCARS facility at APS, comprising USAXS with Si(440) crystals, pinSAXS, and WAXS, can span about five decades in q, characterizing structures from 30 m down to 1 Å during one measurement of typically less than 8 minutes. The talk will present the technical design and capabilities of this instrument, as well as scientific examples taking advantage of this unique instrumentation.

ChemMatCARS Sector 15 is principally supported by the National Science Foundation/Department of Energy under grant number NSF/CHE-0822838. Use of the Advanced Photon Source was supported by the U. S. Department of Energy, Office of Science, Office of Basic Energy Sciences, under Contract No. DE-AC02-06CH11357.

1. Ilavsky, J., Jemian, P.R., Allen, A.J., Zhang, F., Levine, L.E. and Long, G.G. Journal of Applied Crystallography, 42, 2009, 469-479.
2. Ilavsky, J., Zhang, F., Allen, A.J., Levine, L.E. Jemian, P.R., and Long, G.G. Metallurgical and Materials Transactions A, Metallurgical and Materials Transactions A, 44, 2013,68-76.

Session II, Tuesday, September 10

L4

GISAXS – THEORY, EXPERIMENTAL REALIZATION AND SOME RESULTS

V. Holý

Department of Condensed Matter Physics, Faculty of Mathematics and Physics, Charles University in Prague, Ke Karlovu 5, 121 16 Praha, Czech Republic
holy@mag.mff.cuni.cz

In the last decades, various types of nanostructures have been intensively studied by physical, chemical and biological methods. Since the properties of nanostructures are substantially influenced by their structure, structural research of nanostructures is extremely important for the understanding of their performance. Under the structure of the nanoobjects we understand not only the atomic arrangement (the crystal structure in the case of crystalline objects), but more importantly the shapes, sizes and arrangement of individual nano-objects. Methods based on scattering (X-ray-, electron- or neutron scattering) consist in mapping of a part of reciprocal space, the size of which depends on the size of the nanoobjects. Small-angle X-ray scattering (SAXS) usually probes the reciprocal space up to distances of few reciprocal nm, i.e. the method can study particles of sizes from several nm up to approx. one micrometer. For nanoobjects dispersed on a surface or in a thin layer or multilayer, small-angle scattering method is usually performed in grazing incidence geometry (grazing-incidence small-angle x-ray scattering – GISAXS), in which specular scattering from the surface or from the interfaces in the host layer system plays important role.

The theoretical part of the talk will summarize the basic theoretical approaches describing the scattering process (kinematical approximation, distorted-wave Born approximation – DWBA), as well as several theoretical approaches describing self-organized systems on nanoparticles. Several software packages of GISAXS simulations available mostly free of charge will be introduced as well. The theory of GISAXS can be found in [1, 2].

In the second part of the talk the attention will be paid to several experimental realizations of a GISAXS measurement, including laboratory and synchrotron set-ups. The third part of the talk will focus to several experimental examples including mainly semiconductor quantum dots in epitaxial layered systems (Ge/Si, InAs/GaAs, PbSe/PbEuTe) and semiconductor and metallic nanocrystals in amorphous matrix (Ge/SiO_2, Ge/Al_2O_3, Co/SiO_2, among others).

1. U., Pietsch, V. Holý and T. Baumbach T., *High-Resolution X-Ray Scattering From Thin Films to Lateral Nanostructures*, Advanced Texts in Physics, Springer-Verlag Berlin, Heidelberg, New York 2004.
2. G. Renaud, R. Lazzari and F. Leroy, Surface Science Reports **64**, 255-380 (2009).