01-Instrumentation and Experimental Techniques (X-rays, Neutrons, Electrons)

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PS-01.02.10 MULTIPLE SMALL-ANGLE SCATTERING- A BLESSING IN DISGUISE? By S. Mazumder* and A. Sequeira, Solid State Physics Division, Bhabha Atomic Research Centre, Bombay-85,India.

In small-angle scattering (SAS) studies, multiple scattering is generally viewed as an unavoidable complication. The common practice, as it stands today, is to obtain correction factors to eliminate the effect of of multiple scattering in analysing the scattering data. A recent formalism on multiple SAS (Mazumder S. and Sequeira A., Pramana, 1992, 38, 95-159) indicates the following facts which we consider to be blessings in disguise if properly exploited:

By merely scaling down the wave-vector transfer, the Guinier law can be extended for multiple SAS. Porod law remains invariant under multiple scattering as far as the functional dependence of the scattered intensity on wave-vector transfer is concerned. For polydisperse systems, the extractable structural parameters are the moment ratios $\langle R^3 \rangle / \langle R^2 \rangle$ and $\langle R^2 \rangle / \langle R^4 \rangle$ where R_i is linear dimension of the i-th type of inhomogeneity. The averaging is over the population density.

In this paper, it is emphasized that the second moment of the single scattering cross-section manifests predominantly in the Guinier regime of the multiple scattering profile. Consequently, so as far as the curvature in the Guinier regime is concerned, multiple scattering profile does not bear the signature of the interparticle interference function to any significant level. The insignificance of the interparticle interference function in multiple scattering profile can be exploited to extract particle size distribution of inhomogeneities in dense systems with the help of variational technique like the maximum entropy. It is to be noted here that in a traditional SAS study, the extraction of particle size distribution from dense systems is not possible due to interparticle interference effect.

A methodology for extraction of integral and differential parameters from a multiple scattering profile, as also for the elimination of the effect of multiple scattering when it is a nuisance will be presented. The results of an experimental investigation on two bidisperse alumina samples demonstrating the validity of some of the above mentioned aspects will also be discussed.

PS-01.02.11 OMEGA ENERGY FILTERING TEM: APPLICATIONS IN QUANTITATIVE ELECTRON DIFFRACTION. C. Deininger and J. Mayer, Max-Planck-Institut für Metallforschung, Stuttgart, Germany.

With imaging energy filters becoming commercially available in transmission electron microscopy many of the limitations of conventional TEM instruments can be overcome. Energy filtered images or diffraction patterns can now be recorded without scanning using efficient parallel (2-dimensional) detection. We have evaluated a prototype of the Zeiss EM 912 Omega, the first commercially available electron microscope with integrated imaging Omega energy filter. In quantitative electron diffraction the filter is used to remove the inelastically scattered electrons (elastic or zero-loss filtering) and by this quantifiable intensity data without background can be obtained. In selected area diffraction (SAD) weak reflections can be recorded even in thick samples. Another example is the determination of amorphous structure factors of amorphous materials. The most accurately quantifiable data can be obtained by elastically filtered convergent beam electron diffraction (CBED). Structure factor amplitudes and phases can be determined very accurately. For phase measurements in non-centrosymmetric crystals, three beam patterns have to be used. Two dimensional energy filtered data are needed which (without scanning) can only be obtained with an imaging filter and a CCD camera as (linear) detector. From a whole set of such structure factors the charge density distribution in a crystal can be calculated. Furthermore, in the electron spectroscopic diffraction (ESD) mode of operation the angular distribution of inelastically scattered electrons can be imaged. This can be used in the ALCHEMI method to determine atom positions within a unit cell.

PS-01.02.12 DEVELOPMENT OF CCD-BASED AREA DETECTORS FOR MACROMOLECULAR CRYSTALLOGRAPHY USING SYNCHROTRON AND LABORATORY SOURCES, by Walter C. Phillips, Martin Stanton, Youli Li*, Daniel O'Mara, Juanhui Xie, Rosenstiel Basic Medical Sciences Research Center, Brandeis University, Waltham, MA 02254-9110, Edwin M. Westbrook, Istvan Naday, Steve Ross, Mary L. Westbrook, Miklos Kanyo, Argonne National Laboratory, Argonne, IL 60439, and James W. Pflugrath, Cold Spring Harbor Laboratory, Cold Spring Harbor, N.Y. 11724, U.S.A.

We are developing CCD-based x-ray area detectors for collecting macromolecular crystallographic data using synchrotron and laboratory sources. The basic unit of our detectors is a module which consists of a demagnifying fiberoptic taper with a phosphor x-ray converter bonded to the larger face of the taper and a CCD bonded to the smaller face. In order to achieve a large area, detectors are assembled from a number of identical modules. Currently, two detectors are being developed; (1) a high-performance detector for synchrotron applications, and (2) a detector in which a variable number modules can be assembled for a range of applications. The first detector is designed to have a large area, high spatial resolution, high DQE and fast readout. These goals are achieved by using a fixed 3x3 array of nine 5x5 cm square modules, forming a 15x15 cm area. Each module has a taper demagnification of 2:1, and uses a 1024x1024-pixel Tektronix CCD. As a result, this detector has 3072x3072 50um pixels and a high DQE. The second detector is designed so that a single module will form a useful instrument at a reasonable cost, while multiple modules can be used to increase the efficiency of data collection. Each module has a 10x10 cm front surface and a taper demagnification of 4:1, providing a pixel size of 100 μm with a 1024x1024 Tektronix CCD. Because of the larger taper demagnification used in this detector, the cost per unit area is lower, and the performance (as measured by the DQE) is decreased. In both detectors the CCD's are read out in parallel using two amplifiers on each CCD, with a total read time 1.7 s for the array of up to 9 modules. The pixels can be binned, reducing the readout time to 0.4 s.

