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



**OCM-01.03.02** X-RAY TV DETECTOR FOR STRUCTURAL STUDIES WITH SYNCHROTRON RADIATION SOURCE. by

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X-ray TV detectors have been developed for structural studies with synchrotron radiation, especially for exeriments in which real-time measurements and dynamic observations of X-ray diffraction patterns are required. A prototype of the X-ray TV detector (the first generation) consists of four major components;i)phosphor screen,ii) visible-light image intensifier,iii) optical-lens coupling, and iv) a cooled CCD (charge coupled device). In the system, incident X-rays are converted to visible light by a phosphor screen ($Y_2O_2S$:Tb or CsI:Na) which is evaporated on a fiberoptic plate. The fiberoptic plate is attached to the input surface of a large-aperture (100mmf) visible-light image intensifier. The image intensifier multiplies the number of visible light photons by about 70 times and the image size is de-magnified to one fourth. The intensified image (25mmϕ) on the output phosphor of the image intensifier is viewed through a 1:1 optical lens coupling by the cooled CCD (Thomson: THX31156). The CCD has 1024x1024 pixels (pixel size : 19μm x 19μm), a pixel well-depth of 290,000 electrons, and a readout noise of 20 electrons rms.

The prototype has the following characteristics: The active area is 78mm x 78mm.The spatial resolution is 150μm x 150μm (fwhm). The detective quantum efficiency is about 60% for 8 kev, and the dynamic range is more than 104. The readout time is 4 sec. As to the non-uniformity of response, the intensity response at the peripheral is about 65% of that at the central region. This is due mainly to time-invariant vignetting of the image intensifier. The non-uniformity of response can be corrected by using a shading pattern which is obtained by uniform irradiance from a radioactive isotope source. The CCD is cooled to -35C to -45C to reduce the dark current (-5 electron/pixel/sec @ -45C) during measurements. The rms noise of the TV detector (-20electron/pixel) is comparable to the signal produced by one X-ray photon per pixel. The prototype was applied to measure small-angle X-ray diffraction patterns of collagen and of muscle.

The second generation of the X-ray TV detector is developed mainly for time-resolved measurements. In this detector is, a Hi-selvicon camera (avalanched Saticon camera) which has the capability of intensifying the signal amplitude is used in place of the cooled CCD. The output signals of the Hi-selvicon camera are in the standard TV scan mode called NTSC, and are digitized by a 8-bit image-digitizer system which can record successive 64 images of 512x512 pixels in a memory at a speed of 30 images per second. Time-resolved X-ray diffraction patterns from a contracting frog muscle can be recorded of up to a 5.9nm actin layer line with a 33ms time resolution by only one repetition of muscle contraction cycle .The experiments were carried out at BL-15A at Photon Factory, where a mirror-monochromator doubly-focusing X-ray optical system is installed. The camera length was short (675 mm) enough to record a diffraction pattern of up to 5.1 nm actin reflection in the area size of 51.2 mm x 38.4 mm. Dynamic observation of X-ray diffraction patterns during twitch and tetanus muscle contraction will be shown by a video tape.

In order to realize a large area size of the detector, an X-ray image intensifier with a beryllium-window (150mmf) has been recently developed on the basis of a technology for aluminum-windowed medical X-ray image intensifiers. With the Be-window X-ray image intensifier, the photon gain is also improved by more than 10 times compared with the visible-light image intensifier. This improvement has enabled the use of the standard CCD in place of the Hi-selvicon to perform time-resolved measurements at a rate of 30 frames per second (the third generation). The detailed performance of the third generation X-ray TV detector with the Be-window X-ray image intensifier will be described as well as preliminary applications to small-angle X-ray scattering and protein crystallography.

**OCM-01.03.03** GAS DETECTORS AND IMAGE PLATES. THE PROS, THE CONS, THE FUTURE.

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Modern x-ray diffraction experiments, particularly those using synchrotron x-ray sources, often impose extremely stringent requirements on detection systems. Different detector systems have varying strengths and weaknesses, and in order to perform competitive science, it is important that the detector is well matched to the type of experiment. As a result it is often necessary to develop detectors aimed at specific areas of work. At the Daresbury SRS, detector development for x-ray diffraction has concentrated on the production of high speed gas proportional counters and the assessment and operation of commercial image plate phosphor systems.

Gas detectors have certain characteristics that make them well suited to experiments requiring good time resolution and excellent dynamic range. They do however, suffer from counting rate limitations, parallax problems, and have often been considered to be unreliable and difficult to maintain. The gas detector development program at Daresbury has led to the production of a variety of gas detectors that have been in continuous routine operation on small angle scattering beamlines for over six years. The performance of these and other gas detector systems and technologies will be reviewed including;

1) Existing delay line area, linear and quadrant detectors,
2) The recently completed ultra high speed multi-channel microgap linear detector,
3) Results from the prototype high speed multi-channel area detector,
4) Gas microstrip and microgap technology.

For those experiments where fast time resolution is not required, image plate phosphors are rapidly replacing x-ray film. For many experiments they offer much higher dynamic range and lower noise levels. Experience with various types of image plate scanners will be detailed with particular emphasis on the strengths and weaknesses of the various systems.

In conclusion, likely future improvements to detector systems technology will be discussed in the light of x-ray diffraction requirements.

**OCM-01.03.04** DAFS AND DANES: TWO NEW X-RAY DIFFRACTION TECHNIQUES USING REAL PHOTONS AND VIRTUAL PHOTOELECTRONS. by

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This talk will describe two new anomalous x-ray diffraction techniques that use the fine structure versus photon energy in the elastic diffraction intensities to provide spatial, site and valence selective atomic structural information about condensed matter systems. DAFS (Diffraction Anomalous Fine Structure) combines the sensitivities of conventional x-ray diffraction and the sensitivities of XAFS into a single technique [H.Stragier et.al.,Phys.Rev.Lett.69,3064(1992)]. DANES (Diffraction Anomalous Near Edge Structure) combines the sensitivities of diffraction and XANES. The diffraction sensitivities of DAFS and DANES add wavevector(spatial) and crystallographic site selectivities to XAFS and the valence and bonding sensitivities of XANES add these sensitivities to diffraction. The emphasis of this talk will be the simple physics behind these two new techniques. The interesting possibility of using DAFS and DANES to do completely new forms of "locally-sensitive" and "valence-sensitive" crystallography will also be described.

**OCM-01.03.05** RARE EARTH MÖSSBAUER SPECTROSCOPY AND MAPON AS MICROSCOPIC PROBES OF THE LOCAL CRYSTAL FIELD. By Glen A. Stewart, Department of Physics, University College, UNSW, ADFA, Canberra 2600, Australia.

The electric quadrupole interaction (EQI) acting at a rare earth atom's nucleus is a useful microscopic probe for the strength and symmetry of the local crystal field (CF). In the ideal case of a perfectly ionic crystal, the electric field gradient (EFG) tensor is comprised of a constant lattice contribution proportional to the rank 2 CF tensor component and a temperature-dependent contribution associated with the CF-induced distortion of the 4f shell (Stewart, Hyp. Int., 1985, 23, 1-16). The latter contribution is dependent on all three ranks (ranks 2, 4 and 6) of the CF hamiltonian but vanishes for the trivalent, S-state ions, $La^{3+}$, $Gd^{3+}$ and $Lu^{3+}$.

As early as 1964 (Barnes et al, Phys. Rev. A, 1964, 136, 175), it was recognised that the $^{169}$Tm Mössbauer resonance is particularly well suited to the characterisation of the local CF in nonmagnetic materials. The approach is to monitor the EQI at the $^{169}$Tm nucleus as a function of temperature (typically 4.2 K - 300 K) and then to interpret these data in terms of the full CF hamiltonian appropriate for the local site symmetry. In this sense, it constitutes a viable alternative to inelastic neutron sacattering (requiring on-line access to a reactor) and optical spectroscopy (requiring an optically transparent specimen). An important advantage over bulk measurements (such as specific heat or susceptibility) is that individual rare earth sites and impurity phases are able to be resolved. For materials with sufficiently high local symmetry, but not cubic, the CF parameters are determined unambiguously from the data. However, this becomes more difficult to accomplish as the symmetry decreases and the number of CF parameters increases. In order to restrict the number of free parameters, it is sometimes argued that only the rank 2 CF component is important and that the ranks 4 and 6 can be ignored. However, this is not always justifiable. A semi-empirical approach is to use theoretical model computations to fix CF parameter ratios within each of the three ranks. For example, useful CF characterisations for the high-$T_c$ "123" ceramics and their related phases have been achieved via simple point charge model computations of within-rank CF ratios. $^{155}$Gd Mössbauer spectroscopy of the isostructural Gd compound is often employed to provide an independent determination of the rank 2 CF parameters alone.

Application of rare earth Mössbauer spectroscopy to the characterisation of the CF for magnetic materials is more complex and tends to be restricted to cases of high site symmetry. However, for "hard" magnets in which the 4f magnetic moment can be considered to be fully stretched, several of the commonly employed Mössbauer resonances permit reasonable estimation of the axial rank 2 CF parameter which is primarily responsible for the direction and extent of the magnetic anisotropy.

$^{169}$Tm Mössbauer spectroscopy has been employed to investigate axial distortions (both static and dynamic) of cubic sites in nonmagnetic materials and, in principle, it is possible to employ the $^{155}$Gd Mössbauer resonance to monitor small cubic site distortions in magnetic materials. However, the minimum resolvable lattice EFG contribution is limited by the Mössbauer resonance's line width and, in magnetic specimens, the small EQI is often buried within an inhomogeneous magnetic line broadening. In such cases, the low temperature MAPON (modulated adiabatic passage on oriented nuclei) technique developed by the Canberra group should prove useful. The approach would be to substitute radioactive S-state probes ($^{177}$Lu say) at the rare earth sites of a single crystal specimen. At dilution refrigerator temperatures (<100 mk), the nuclear sublevel populations are unequal (the nuclei are oriented) and the radiation distribution is aspherical. The radiation distribution is then used to monitor the sublevel populations as an amplitude-modulated rf field is swept through the sequence of EQI-split subresonances. When the modulation frequency is small compared with the EQI, the sublevel populations are left unchanged by the sweep. When the modulation frequency is large compared with the EQI, the populations are cyclically permuted. Thus by repeating the sweep procedure for different modulation frequencies, the EQI distribution can be mapped out. The technique has already enjoyed considerable success in dilute transition metal alloy investigations (Chaplin and Hutchison, Hyp. Int., 1992, 75, 209-228).

**OCM-01.03.06** RESONANT NUCLEAR X-RAY SCATTERING AS A CRYSTALLOGRAPHIC TOOL. By J. Arthur*, Stanford Synchrotron Radiation Laboratory, Stanford, CA, USA.

Modern synchrotron x-ray sources are bright enough to efficiently excite low-lying nuclear resonances. Excitation followed by elastic emission, a process closely related to the Mössbauer effect, gives rise to coherent resonant nuclear scattering. The extremely narrow energy widths of the nuclear resonances and the multipole nature of the resonance transitions make the scattering very sensitive to the hyperfine environment of the nuclei, and thus make this technique potentially very useful as a crystallographic tool for studying magnetic materials. Current research is directed principally at understanding the coherent resonant scattering process itself, which involves many subtle multiple-scattering effects, and on developing techniques for expanding the range of sample types that can be studied. Single crystals, artificial layered structures, and polycrystalline foils are now routinely examined. Three isotopes (57-Fe, 169-Tm, and 119-Sn) have been used so far, and several others should soon follow.

**OCM-01.03.07** MICRO-REGION CRYSTALLOGRAPHY BY THE LAUE METHOD USING WHITE SR. By K.Ohsumi*(1),K.Hagiya(2), T.Takase(3),S.Yasuami(4),M.Miyamoto(3) and M.Ohmasa(2)
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In order to analyze micro-textures in complicated crystal aggregates, and to refine structures of submicrometer-sized crystal particles or twinned domains, an equipment and software system were developed using the Laue method combined with synchrotron radiation(SR). This work was carried out at beamline 4B of the Photon Factory(PF), KEK and were successfully applied to some inorganic specimens (K.Ohsumi et al.,1991,J.Appl.Cryst. Cryst.,24,340-348:K.Ohsumi et al.,1992,Rev.Sci.Instrum. ,63(3),1181-1184).
The micrometer region of the specimen becomes a target to be analyzed in such cases as mentioned above. Due to limited space around beamline 4B of PF, a micro-pinhole is used for making very fine incident SR.