

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

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 scattering (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., 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.