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

peak-to-background ratios and an almost constant resolution of 2.5 $X10^{-3}$ down to $\sin\theta/\lambda = 0.04$ $Å^{-1}$ (25 Å neutrons). The contributions to the resolution are given. The performance has enabled us to determine the incommensurate magnetic propagation vector in the triclinic antiferromagnet $FeVO_4$ and to study its temperature dependence in the range from 4 K to its Néel point at 21 K. Other examples include the magnetic scattering from the two commensurate antiferromagnetic phases of Mn_5Si_3 and the pressure dependence of their magnetic structures. The design of a purpose-built cold neutron diffractometer is described.

PS–01.04.07 CIRCULAR MAGNETIC X-RAY DICHROISM AT FE K-EDGE AND GD $L_{2,3}$-EDGES IN Fe/Gd MULTILAYERED FILMS
F.Itoh*,+, M.Nakamura+, H.Sakurai+ and H. Kawata++
+ Department of Electronic Engineering, Gunma University, Japan
++ Photon Factory, KEK, Tsukuba, Ibaraki 305, Japan

Fe/Gd multilayered film is known to have interesting properties such as spin flop[1] and temperature compensation[2]phenomena which sensitively depend upon artifacial period of the multilayer. In this paper, we report measurements of circular magnetic x-ray dichroism(CMXD) at Fe K-edge and Gd L_2- and L_3-egdes of Fe/Gd multilayetred films as a function of artifacial period of the film, using circular polarized X-rays at AR NE-1 of KEK.

It is shown that the CMXD spectra of Fe K-egde in samples with longer period than 10 Å is similar to that in pure Fe while the CMXD spectra of Gd L_2- and L_3-edges are opposite in sign to that in pure Gd. In samples with shorter period than 5 Å, on the other hand, spectra of Fe K-egde and Gd L_2 and L_3-egdes are com@pletely reversed compared to those in samples with longer period. This means that Fe moments are dominant in sapmles with longer period than 10Å, while Gd moments become dominant in samples with shorter period than 5Å, keeping both Fe and Gd moments anti-ferromagnetic. A L-S separation of Gd moment was tried based on the sum rule[3], showing clear change of both components against the artifacial period of the multilayered film.

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PS–01.04.08 MAGNETIC X-RAY DIFFRACTION FROM FERROMAGNETIC MATERIALS
S P Collins and D Laundy and R J Cernik*, SERC Daresbury Laboratory, Warrington, UK

This poster describes the simple 'White-beam' technique, developed at the SRS to measure non-resonant magnetic X-ray diffraction from ferromagnetic crystals with synchrotron radiation. The results of several experiments are presented.

Early work on iron [1, 2] has demonstrated the feasibility of the X-ray technique, and produced data which are in excellent agreement with, and of similar quality to, the first polarized neutron measurements.

More recent data have highlighted the complementarity between X-ray and neutron diffraction in two important respects. First, X-ray diffraction has been adopted to determine the *ratio* of spin to orbital magnetization in a ferrimagnetic Rare Earth compound [3] - a measurement which cannot be made directly with neutron diffraction.

The second aspect of complementarity brought to light by the synchrotron X-ray measurements concerns the accessible range of momentum transfers. We show that high-energy X-ray data can extend far beyond the maximum wavevectors which are practicable with thermal neutrons, and that the corresponding data quality is surprisingly good This is in contrast to situation with low momentum transfers where neutron data are currently of far superior quality.

References
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PS–01.04.09 EXCITATIONS OF CONDENSED MATTER STUDIED BY INELASTIC X-RAY SCATTERING WITH HIGH ENERGY RESOLUTION By E. Burkel, Sektion Physik, University of Munich, Munich, Germany

Very high energy resolution measurements using X-rays can be achieved by extreme backreflection (Bragg angle close to 90^0) from perfect crystals. This technique allowed the development of the instrument INELAX for inelastic scattering experiments at the HARWI wiggler at DORIS, DESY Hamburg. At present, an energy resolution of 9 meV is achieved and the instrument proves to be an excellent tool to investigate collective excitations in condensed matter. Energy transfers from 10 meV to 5 eV and wavevectors up to 13 $Å^{-1}$ are accessible.

Longitudinal and transverse dispersion curves of beryllium and diamond were extracted from measurements of phonons in single crystals of these materials. The method was also applied to single crystals of He and to superconductors.

Furtheron, collective excitations of liquid lithium were studied and the dispersion of these excitations could be detected.

An important application of inelastic X-ray scattering is the study of electronic excitations in solids. Measurements of such excitations in single crystals of lithium were performed up to energy transfers of 5 eV with an energy resolution of 38 meV. They provided information on the dispersion of excitations which can be described as zone boundary collective states. The measurements revealed a fine structure which was not observed before.

01.05 – X–ray and Neutron Powder Diffraction

MS–01.05.01 MODELING AS A COMPLEMENT TO POWDER DIFFRACTION EXPERIMENTS IN STUDYING INORGANIC AND ORGANIC SOLIDS. By C. M. Freeman and J. M. Newsam*, BIOSYM Technologies Inc, 9685 Scranton Road, San Diego CA 92130, USA

Dramatic improvements in analytical instrumentation have been paralleled by equally impressive advances in computer hardware and in modeling and theoretical methods. Computer modeling has in fact become established as a key complement to diffraction experiments, aiding in the evaluation of experimental results and in the interpretation of analytical data in terms of atomic-level behavior. A suite of modeling methods appropriate for

developing viable initial models of solid state inorganic systems is outlined, with an emphasis on their application to topical materials problems. In addition to direct analogs of classical modeling-building methods, techniques for automated model-building, from polyhedra, cages or sheets, and for the automatic determination of unit cells and/or space group symmetries from such models are described. Simulated annealing, using simple geometric, potential, or diffraction-pattern matching functions is also proving effective as a direct space route to structure determinations from powder diffraction data. Recent results for aluminosilicate frameworks, mixed metal oxides, and molecular system are described.

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MS–01.05.02 ORIENTATIONAL ORDER-DISORDER TRANSITIONS IN THE VAN-DER-WAALS COMPLEX C_6H_6:C_6F_6 – A CASE FOR COMBINING POWDER NEUTRON AND X-RAY DATA. By Jeremy K. Cockcroft[1]*, Andrew N. Fitch[2], and Jeffrey H. Williams[3], [1]Department of Crystallography, Birkbeck College, Malet Street, London WC1E 7HX, United Kingdom; [2]ESRF, BP220, F-38043 Grenoble Cedex, France; and [3]Institut Laue Langevin, BP156, F-38042 Grenoble Cedex, France.

Recent developments in powder diffraction during the last few years indicate that in future *ab initio* crystal structure solution will no longer be the sole domain of single crystal methods. In particular, the development of high-resolution powder diffractometers with sample environments routinely covering the temperature range 2 K to 1500 K at both X-ray synchrotron radiation and neutron reactor and spallation sources opens up a field of chemical crystallography of interest to both the synthetic and physical chemist. The developments in hardware are being mirrored by the production of user-friendly data treatment packages suitable for the non-specialist crystallographer. There is growing interest in the refinement of structures using more than one source of data, for example combining the information from neutron and X-ray data sets or combining X-ray data with molecular simulations.

This paper will discuss several systems showing the advances recently made in powder diffraction with respect to *ab initio* structure determination with emphasis on systems that needed both neutron and X-ray data for their solution and refinement. The phase transitions in the 1:1 complex formed by benzene and hexafluorobenzene will be used as one example of a system whose chemical crystallography can now easily be studied using high-quality powder data and modern software. The desirable attributes of modern Rietveld programs will be mentioned.

MS–01.05.03 COMPLEMENTARITY OR COMPETITIVITY OF SYCHROTRON AND NEUTRON POWDER DIFFRACTION. A.W. Hewat, Diffraction Group, ILL, 156X Grenoble, Cedex, France.

The European Sychrotron Radiation Facility (ESRF) is becoming operational in Grenoble France, on the same site as the European High Flux Reactor (ILL). In the USA too, a new Synchrotron source is being constructed at the Argonne National laboratory, not far from the existing pulsed neutron source. It is hoped that the combination of the best synchrotron and neutron scattering facilities on the same site will stimulate complementarity between the two techniques. Certainly in times of budget constraints it will produce a certain competition.

Crystallography using neutrons will be among the areas most challenged by the new synchrotron sources. With such high X-ray intensities it becomes in principle possible to work with 'single crystals' of inorganic materials the size of the crystalline grains of powders. Alternatively, the high synchrotron intensity can be traded for very high resolution powder diffraction, while for neutrons, resolution remains limited in many cases by the limited intensity from even the best sources.

In this paper we will examine some of the recent successes of both neutron and synchrotron powder diffraction, and show that the two techniques are more complementary than competitive, and are likely to remain so in the immediate future. Neutron powder diffraction has evolved considerably over the years, and must continue to evolve to retain its place as an essential crystallographic tool for chemists. Synchrotron powder diffraction must also evolve to establish a rôle for itself, distinct from both conventional X-ray powder diffraction and neutron powder diffraction.

MS–01.05.04 A MULTI-PURPOSE VACUUM DIFFRACTOMETER FOR OPERATION AT THE PHOTON FACTORY. By S.W. Wilkins,*+ Z. Barnea,! D.C. Creagh,@ T.J. Davis,+ R. Garrett,# S. Janky+ and A.W. Stevenson+
* CSIRO, Division of Materials Science and Technology, Clayton, Vic 3168, Australia. ! School of Physics, University of Melbourne, Parkville, Vic 3052, Australia. @ Physics Department, University College, University of NSW Campbell, ACT 2600, Australia. # ANSTO, Lucas Heights Laboratories, New Illawarra Rd., Lucas Heights, NSW 2234 Australia.

A versatile high-resolution two-axis diffractometer has been constructed for operation at the Photon Factory as an Australian National Facility [Barnea, Z. et al (1989) Rev. Sci. Instrum. **60**, 2537-40] . The instrument is capable of operation in various modes including:

i) high-resolution powder diffraction with single-counter and crystal analyzer (Fig.1a),
ii) high-resolution powder diffraction in Debye-Scherrer mode with imaging plates as recording medium, either stationary or translating for time-dependent studies (Fig.1c),
iii) small-angle x-ray scattering (Fig.1b),
iv) protein crystallography in screenless Weissenberg mode (Fig 1c),
v) two-axis single crystal diffractometry using mono-detector or imaging plates (Fig.1a or 1c).

Some important features of the instrument are the capability for operation with all main components in vacuum and also the use of a double-crystal sagittal focusing monochromator as primary monochromator together with the optional use of a condensing-collimating channel-cut (CCCC) monochromator or other channel-cut as secondary monochromator. The use of a CCCC monochromator enables fine tuning of beam position on the sample, harmonic suppression, beam condensation and variation of wavelength band-pass [Wilkins, S.W. & Stevenson, A.W. (1988) Nucl. Instrum. & Meths. **A269,** 321-8. Wilkins, S.W. (1986) Aust. and Int. Pat. PCT/AU87/00262.] Further features include the use of high-precision absolute encoders on both shafts and the use of a large camera radius (570 mm) for the imaging plate cassette.