

15.1-2 AN X-RAY POWDER DIFFRACTOMETER AT THE DARESBURY SYNCHROTRON RADIATION SOURCE. By P D Hatton<sup>1</sup>, P Thompson<sup>2</sup> and A M Glazer<sup>3</sup>.

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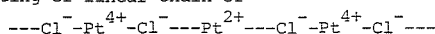
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A centralised data collection facility for powder diffraction is operational on the superconducting wiggler beam line. The wiggler radiation has a characteristic wavelength of 0.93Å, with an effective cut-off below 0.1Å. The diffractometer is a general purpose two circle goniometer which can be operated in a variety of configurations, including both energy- and angle-dispersive modes. For monochromatic work a double-crystal Si(220) monochromator is inserted and this can be scanned continuously from 0.2 to 2.5Å with a wavelength resolution of  $\Delta\lambda/\lambda \approx 1 \times 10^{-4}$ . The diffractometer is of modular construction allowing it to be assembled in a variety of ways, including either horizontal or vertical scattering geometry.

The inherent high intensity and collimation of the source can be effectively utilized to provide very high resolution. This makes powder diffraction on synchrotrons an important technique, not just for lattice-parameter measurements, but particularly for structural refinements either by the Rietveld method or by direct integrated intensity measurements. The apparatus is also particularly suitable for the examination of materials under non-ambient conditions; a variety of cryostats, furnaces and pressure cells are available. The latest results will be discussed and compared to previous measurements, particularly for both high resolution angle-dispersive (Thompson & Wood, *J. Appl. Cryst.* (1983) 16, 458 and Cox, Hastings, Thomlinson and Prewitt, *Nucl. Instr. Meth.* (1983) 208, 573) and energy-dispersive (Glazer, Hidaka and Bordas, *J. Appl. Cryst.* (1978) 11, 165) techniques.

15.2-1 EXAFS AND XANES STUDIES ON THE LOCAL STRUCTURE OF A QUASI-ONE-DIMENSIONAL MIXED VALENCE CRYSTAL: WOLFFRAM'S RED SALT. By H. Terauchi, S. Iida, K. Tanabe, K. Kikukawa, Y. Nishihata, Department of Physics, Kansai-Gakuin University, Nishinomiya 662, Japan, H. Maeda, Department of Chemistry, Okayama University, Okayama 700, Japan, M. Hida, School of Engineering, Okayama University, Okayama 700, Japan and N. Kamijo Government of Industrial Research Institute, Ikeda 536, Japan.

A number of insulating chain crystals have been investigated extensively because of interest in the fascinating phenomena due to a strong electron-phonon coupling. Here we report the results of the EXAFS and XANES studies on a quasi-one-dimensional crystal called Wolfram's red salt (WRS). WRS is considered to be a mixed valence crystal consisting of linear chain of



where each  $\text{Pt}^{4+}$  or  $\text{Pt}^{2+}$  ion is coordinated by four ethylamines. From the measurement of the diffuse X-ray scattering in WRS, we reported that the short range correlation shows the one-dimensional character (*J. Phys. Soc. Jpn.* 52 (1983) 2769). To clarify the local structure of the short range order, EXAFS and XANES measurements were carried out with synchrotron radiation of the 2.5GeV storage ring of Photon Factory in KEK (Tsukuba). From the Fourier transform of the EXAFS function observed near the Pt-L edges, the locally-distorted structure was determined. The mixed valencies of Pt ions were directly confirmed by measuring the XANES at Pt-L edges. In WRS the local distortion is stable in the ground state because of the strong electron-phonon interaction and the excited state is self-trapped with no potential barrier. The lattice relaxation of an optically excited state in WRS will be reported at the meeting. The preliminary results appeared in *J. Phys. Soc. Jpn.* 52 (1983) 3700.

15.2-2 EXAFS OF Tc IN BOROSILICATE GLASSES. By M. Antonini and F.R. Thornley, Department of Applied Physics, University of Strathclyde, Glasgow, U.K.; A. Merlini, Physics Division, Joint Research Centre, Ispra, Italy.

EXAFS measurements have been made of the K absorption edge of Tc for borosilicate glasses containing a nominal 2% wt concentration of Tc. There is interest in the valence state and coordination of Tc in these glasses, which have been proposed for the storage of high-activity wastes. Reference materials were Tc metal and an aqueous solution of  $\text{NH}_4\text{TcO}_4$ , for  $\text{Tc}^{7+}$ . A good match in phase and frequency could be obtained between the  $\chi(k)$  signals for the glass, and for the Tc metal at higher values of  $k$  ( $6.5 - 10 \text{ \AA}^{-1}$ ). However, the signal amplitude from the glass is about a factor of 3.5 smaller than that from the metal. The radial distribution function from the glass is dominated by a peak at the same position as that for the Tc - Tc nearest neighbour distance in Tc metal; there are subsidiary peaks at smaller values of  $R$ . This suggests that some of the Tc in the glass is present as the metal, but that there are also many Tc atoms with O as their nearest neighbours. The O scattering amplitude falls off with  $k$  much more rapidly than does the Tc amplitude, so at large  $k$  the Tc - Tc signal alone is seen. Attempts at extracting at  $\chi(k)$  signal for the glass representing just Tc - O, by subtracting a scaled Tc metal signal, have not been successful. A comparison with calculated signals suggests that one of the subsidiary peaks represents a Tc - O distance the same as in  $\text{TcO}_2$  - i.e. Tc present as  $\text{Tc}^{4+}$ .

15.2-3 Laboratory Fluorescent EXAFS Spectrometer for Thin Film Studies. By A. Nakano, Y. Hayashi and T. Edamura, 2nd Department, Production Engineering Research Laboratory, Hitachi Ltd., Totsuka-ku, Yokohama 244, Japan.

Thin film with amorphous structure is often found on the substrates by sputtering method using crystalline starting material which is an ordinary technique for the thin film formation. The extended X-ray absorption fine structure involves atom-atom distances and coordination numbers in the first several coordination shells surrounding the absorbing atom. The structure informations are of considerable utility when studying the electrical and thermal characteristics of the thin films. A laboratory fluorescent X-ray spectrometer has been constructed, which allows very high quality EXAFS data to be obtained. The spectrometer utilizes automatically changeable 4 Johansson cut bent crystal monochromators operated on the Rowland circle for the high X-ray flux. The spectrometer has an energy resolution of  $\Delta E/E < 10^{-3}$ . The scanning step width of the goniometer is determined on the basis of the constant wave vector increase of the ejected photoelectrons. The X-ray generator is specially designed for the low voltage and high current operation, 20kV-500mA, by a rotating anticathode and pierce electron gun. Incident and fluorescent X-ray photons are counted by a pair of flow proportional counters. The EXAFS data of the thin film with 1000Å thickness and composition of Cr-Si-O on a glass substrate was obtained within 2 hours by the spectrometer, which is almost a comparable time with the synchrotron X-ray source EXAFS spectrometer.