06.2–19 A CHARGE DENSITY STUDY OF VANADIUM METAL USING MULTI-WAVELENGTH γ-RAY DIFFRACTOMETRY. By H.R. Kretschmer and J.R. Schneider, Hahn-Meitner-Institut für Kernforschung, Berlin (FRG).

Within a project of the IUCr Commission on Charge, Spin & Momentum Densities on vanadium metal a full set of room temperature structure factors up to $\sin\theta/\lambda \leq 1.1\text{Å}^{-1}$ has been measured using 316.4, 411.8, 468.0 and 604.4 keV γ-radiation from radioactive Au-198 and Ir-192.

In a first step the temperature dependence of the structure factor anisotropy for the pairs of reflections 330/441 and 442/600 has been measured over the range from 70 to 800 K. The low temperature data agree well with earlier X-ray scattering results (Diana & Mazzone, Phil. Mag.(1975)32,1227) and confirm a deformation of the atomic 3d charge distribution in the solid which points towards the nearest neighbours in the bcc lattice. At higher temperatures the anharmonic motion towards the next-nearest neighbours first cancels the effect of the electronic anisotropy and finally dominates the measured structure factor anisotropy for the 442/600 pair of reflections. The temperature dependence of the lattice parameter was measured using the X-ray Bond method and no indication for a significant amount of impurities was found. The parameters α, β and δ of the one-particle potential which describes the thermal motion of the atoms, were determined by analysing the temperature dependence of the absolute 442 structure factor. All measured intensities were corrected for thermal diffuse scattering.

The low order structure factors were measured on two samples showing different amounts of extinction, but the wavelength extrapolation to the limit of zero extinction provided identical structure factor values for both samples. The experimental data are compared with the results of density-functional calculations within the local-density approximation to the exchange-correlation energy functional (e.g.: Laurent,Wang&Callaway,Phys.Rev.B(1978)17,455). The agreement between experiment and theory is reasonable, remaining differences are discussed in terms of difference charge density maps.

06.2–20 ELECTRON DENSITY DISTRIBUTIONS IN CRYSTALS OF ILMENITE-TYPE OXIDES, $MTiO_3$ (M:Mn,Fe,Co). By K. Kitō, K. Tanaka and F. Marumo, Research Laboratory of Engineering Materials, Tokyo Institute of Technology, Nagatsuta 4259, Midori, 227 Yokohama, Japan.

Electron density distributions in crystals of $MTiO_3$(M:Mn, Fe,Co) have been investigated with the single-crystal X-ray diffraction method. The metal ions occupy trigonally distorted octahedral sites. No indication of aspherical distribution of 3d electrons was detected around the Mn^{2+} ion. The Fe^{2+} ion gave deformation densities which are primarily explained with the high-spin electron configuration of a d^6 ion in a trigonally distorted octahedral field. Residual densities around the Co^{2+} ion after the spherical atom refinement are in accordance with the high-spin configuration of a d^7 ion in an octahedral field. The electron clouds around the Ti^{4+} ions are deformed to shield the positive charges of the M^{2+} ions in $MnTiO_3$ and $CoTiO_3$. The feature was not clear in $FeTiO_3$.

Refinements were carried out by utilizing the orbital scattering factors for 3d electrons with variable distribution of electrons over the 3d orbitals. When the scattering factors based on the wavefunctions derived on the assumption of a regular octahedral field, the values 1.47, 3.52 and 2.01 were obtained for the populations on a_g, $e_g(t_{2g})$ and $e_g(e_g)$ orbitals of the Co^{2+} ion, respectively. Refinement assuming the real symmetry (C_3) of the crystal field did not give significant difference. The populations obtained were 1.31, 2.33 and 2.36 for the respective orbitals of the Fe^{2+} ion.

In $MnTiO_3$ it is known that antiferromagnetic ordering of spin remains within the (0001) plane even above the Néel temperature. Examination of the residual density map revealed that neighbouring Mn^{2+} ions on (0001) are connected with a positive density region, suggesting existence of a direct interaction between the cations.

06.2–21 CHARGE DENSITY AND THERMAL VIBRATION ANALYSIS OF BIOTINE SANS CHAINE: THE BIOTIN BICYCLIC MOIETY.

George T. DeTitta, Robert H. Blessing and Grant R. Moss, Medical Foundation of Buffalo, 73 High street, Buffalo, New York 14203 USA

Biotin is a vitamin necessary for the biological uptake and transfer of carbon dioxide. We have measured diffraction data for biotine sans chaine (BSC), $C_5H_8O_2N_2S$, the bicyclic ring moiety of biotin, at 298K to $d^*=2.40\text{Å}^{-1}$, and at 120K to $d^*=2.64\text{Å}^{-1}$ and are currently measuring diffraction data at 85K. BSC is monoclinic, space group $P2_1/c$, with cell constants a=11.764, b=5.550, c=10.672Å and $\beta=111.68^\circ$ at 298K.

The data are being analyzed in terms of the deformation electron density, particularly in the ureido group where carbon dioxide bonds in biological transfer reactions, and in terms of a thermal vibration model. The systematic effects of variable integration limits in λ, as discussed by Mathieson (Acta Crystallographica, A38, 378-387; 1982), on the deformation density will be probed through a series of slice scan experiments at 85K.

This work supported by NIH Grant AM19856.