

## 21. STRUCTURAL RESULTS FROM METHODS OTHER THAN DIFFRACTION

21.1-3 SITE SYMMETRY INFORMATION IN ELECTRON ENERGY LOSS NEAR EDGE FINE STRUCTURE\* By M.M. Disko, J.C.H. Spence & O.F. Sankey, Arizona State University, Tempe, Arizona, 85287 U.S.A.

Electron energy loss near edge fine structure (ELNES) data show features characteristic of the excited atomic species (J. Taftø and J. Zhu, Ultramic. 9, 349 (1982)). The data of Taftø and Zhu consists of K edges in olivine (Mg-oct, Si-tet), spinel (Al-oct, Mg-tet), and orthoclase (Al-tet). Here "oct" specifies that the atom is at an octahedral site with inversion symmetry, and "tet" refers similarly to tetrahedral sites without inversion symmetry. In each case the ELNES data (0 to 20 eV above threshold) for inversion centers show a single, broad (~10eV wide), almost Gaussian feature, while the ELNES data for tetrahedral sites contains several sharp features.

It has been demonstrated that the K edge ELNES can be interpreted as features related to the density of states (DOS) in the presence of the core hole above the Fermi level, superposed on a smooth background that is essentially atomic (R.D. Leapman, L.A. Grunes, & P.L. Fejes, Phys. Rev. B26, 614 (1982)). The ELNES experiments are conducted under conditions where dipole selection rules apply, and hence a 1s core electron makes transitions to final states with p character. The present work extends the work of Taftø and Zhu to materials having less complex geometries. Electronic structure calculations are performed and compared with the new ELNES data to gain an understanding of the correlation between edge shape and site symmetry.

A preliminary study of Be<sub>2</sub>C, which is an antiferroite structure, (C atoms at inversion centers, Be-tet) gives results that follow the trend of Taftø and Zhu. A simple tight binding calculation of the Be<sub>2</sub>C DOS shows certain peaks present in the Be p-DOS are absent in the C p-DOS above the Fermi level, explaining the extra features at the Be K edge. Efforts are underway to study Mg<sub>2</sub>Si, and improve the Be<sub>2</sub>C calculation.

Extension of this work to a number of systems should provide a new structural tool that takes advantage of the high spatial resolution available in STEM, and may prove valuable in the study of disordered materials.

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21.1-4 SITE SYMMETRIES IN OLIVINES OBTAINED FROM STRUCTURAL AND SPECTROSCOPIC DATA By H. Rager, FB Geowissenschaften, Universität Marburg, Lahnberge, D-3550 Marburg (FRG) and S. Hosoya, Research Institute for Iron, Steel and Other Metals, Tohoku University, Sendai, Japan.

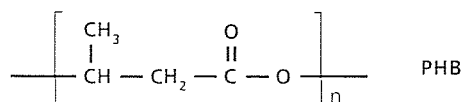
Various attempts have been made to quantify the distortion of the octahedral positions in olivines with varying cations. Usually the distortion is expressed in terms of the cation-oxygen distances and of the angle variance within the octahedra. These terms are obtained from data for refined olivine structures. Measures of the distortion for the M1 and M2 sites have been proposed by several authors. Essentially, they give the same result i.e. the distortion at M1 is approximated by the point symmetry D<sub>4h</sub> with the main axis along O3-M1-O3 and at M2 by C<sub>3v</sub> with the main axis along a or b (space group Pbnm).

In the case of paramagnetic cations, present at M1 and M2 in low concentration, the local symmetry can also be obtained by measuring the electron paramagnetic resonance spectra in single crystals. The results obtained in that way are: The local symmetries of Fe<sup>3+</sup>, Cr<sup>3+</sup> and Ni<sup>2+</sup> at M1 are D<sub>4h</sub> (main axis along O2-M1-O2), D<sub>2h</sub> (no correspondence of the main axis with any cation-oxygen direction), and C<sub>3i</sub> (main axis along a), respectively. The local symmetry of both Mn<sup>2+</sup> and Fe<sup>3+</sup> at M2 is C<sub>2v</sub> (main axis nearly along M2-O2) and of Cr<sup>3+</sup> at M2 is D<sub>2</sub> (main axis nearly along a). Thus, the local symmetries at M1 and M2 derived from structural and spectroscopic data differ significantly either in the direction of the main axis or in the local symmetry. This will be discussed.

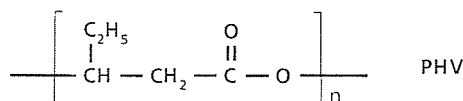
21.1-5 Analysis of Poly(β-hydroxybutyrate-co-β-hydroxyvalerate) Using X-ray Diffraction, NMR Spectroscopy and Differential Scanning Calorimetry

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Poly-β-hydroxybutyrate (PHB) is an important storage material in a variety of bacteria. This optically active polymer is found as crystalline granules (1) in the native bacteria. When isolated from the bacteria and cast into films PHB has mechanical properties similar to those of polypropylene.



Poly-β-hydroxyvalerate (PHV) is also a bacterial storage material but less ubiquitous than PHB.



The crystal structures of these optically active polymers have been previously determined (2,3,4). PHB crystallizes in an orthorhombic unit cell with a = 5.76 Å, b = 13.20 Å and c (fiber axis) = 5.96 Å which contains two antiparallel chains. PHV also crystallizes in an orthorhombic unit cell with a = 9.32 Å, b = 10.02 Å and c (fiber axis) = 5.56 Å again containing two antiparallel chains.