MS10.08.08 DETERMINATION OF POLYMER ELECTRO-LYTE STRUCTURES BY X-RAY POWDER DIFFRACTION.

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Polymer electrolytes consist of inorganic salts dissolved in coordinating polymers such as poly(ethylene oxide) $[(CH_2CH_2O)_n]$. They can exhibit conductivities approaching that of many liquid electrolytes and their materials properties make them very attractive for use in the next generation of solid state devices. Since single crystals cannot be grown we have recently pursued *ab initio* powder methods to determine some of these structures. Several structures of the form MX:poly(ethylene oxide)$_n$ (M = Li+, Na+, K+, Rb+, NH$_4$+; X = ClO$_4$-, CF$_3$SO$_3$-, or SCN-; n = 3 or 4) will be discussed. More recently a new Monte Carlo based method[1] has been developed and applied to the determination of the structure of LiN(CF$_3$SO$_2$)$_2$:PEO$_3$, a problem which we were unable to solve by "conventional" powder methods. The use of constrained Rietveld refinement will be emphasised in tackling these structures. The effect of cation and anion size in determining the structures adopted will also be discussed.

1. Y. Andreev et al. This meeting.

PS10.08.09 FOUR TERNARY STRUCTURES IN THE Al-Cu-Ru SYSTEM. C. Freiburg, U. Lemmerz, W. Reichert Zentralabteilung für Chemische Analysen, Forschungszentrum Juelich, D-52425 Juelich

The structures of (Al,Cu)$_{13}$Ru$_4$ (C2/m), Al$_7$Cu$_2$Ru (P4/mnc), Al$_{58}$Cu$_{30}$Ru$_{12}$ (Pm3), and Al Ru(Cu) (Pm3m) have been determined from 2-phase powder mixtures by X-ray diffraction and Rietveld refinements. The Al-Cu-Ru system also has an icosahedral phase Al$_{65}$Cu$_{20}$Ru$_{15}$ (FCI) [1], so it is important to investigate its neighbourhood. Many alloys with different nominal compositions were prepared by inductive melting in a water cooled copper crucible under Ar-atmosphere and annealed for different times and temperatures. Two powdered alloys each with two of the above thermodynamically stable phases, were chosen for the final structure determinations with a suitable Rietveld program [2].

The first alloy had a nominal composition Al$_{70.3}$Cu$_{19}$Ru$_{10.3}$, it was annealed for 1630h at 650°C, and it consisted of 13 vol% of the 1st above mentioned structure and 87% of the 2nd. The second alloy had a nominal composition of Al$_{56}$Cu$_{31}$Ru$_{13}$, it was annealed for 600h at 800°C, and it consisted of 73 vol% of the 3rd structure and 27% of the 4th. The first two structures are homeotypic to (Al, Cu)$_{13}$Fe$_4$ [3] and Al$_7$Cu$_2$Fe [4]. The 3rd structure is also adjacent to the FCI-phase and homeotypic to α-Al Mn Si [5], the 4th is of the CsCl-type and has an unexpected concentration range.

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PS10.08.10 STRUCTURE REFINEMENT OF GAMMA ALUMINA - REVISITED. Bee K. Gan, Brian H. O'Connor, Department of Applied Physics, Curtin University of Technology, GPO Box U1987, Perth6001 ,Western Australia

The extensive literature on gamma alumina has established that the structure approximates the spinel, AB$_2$O$_4$ form, with either a cubic or tetragonally distorted cell. Various authors have proposed different models for the locations and site-occupancies of the aluminium ions. From the literature, it is clear that the structure of gamma alumina have not been established unequivocally. It was therefore seen to be of interest in this study to re-examine the gamma alumina structure.

Deuterated gibbsite specimens were used as starting materials to study the transformation from gibbsite to boehmite, which then transformed to gamma alumina. Gibbsite was selected because of its availibility as one of the products produced from bauxite by the Bayer process by Alcoa of Australia Limited, Western Australia.

The present neutron diffraction data and Rietveld refinement results have shown that two different polymorphs of gamma alumina have been found to co-exist, which has resolved the long-standing dispute over the nature of the structure, with either the cubic or tetragonal models. In this study, two conclusions have been note: (i) the boehmite to gamma alumina transformation occurs at approximately 500 C, resulting in the formation of a dominant tetragonal phase and a smaller fraction of the cubic phase and (ii) the proportion of the tetragonal phase increases at the expense of the tetragonal phase over the range 500-900 C, before transforming to higher temperature transition aluminas.

PS10.08.11 NEUTRON POWDER DIFFRACTION STUDY OF THE NUCLEAR AND MAGNETIC STRUCTURES OF La$_{1-x}$M$_x$MnO$_3$ (M=Ca and Sr). Q. Huang[1,2] A. Santoro[1], J.W. Lynn[1,2], R. Erwin[1], J. Borchers[1], J. Peng[2] & Z.Y. Li[2], Reactor Radiation Division, NIST, Gaithersburg, MD 20899[1], University of Maryland, College Park, MD 20742[2]

A study of the nuclear and magnetic structures of the Ca and Sr doped perovskitetype compounds La$_{1-x}$Ca$_x$MnO$_3$ (x=0, 0.15, 0.175, and 0.33) and La$_{0.67}$Sr$_{0.33}$MnO$_3$ has been made using neutron powder diffraction. For x=0, the Rietveld refinements indicate that two phases coexist. One phase is orthorhombic (Pnma), and the fraction of the Pnma phase increases smoothly from 14wt% at 450 K to 85wt% at 100 K, and remains constant below 100 K. The other phase is rhombohedral (R$\bar{3}$c) at room temperature and above, while below 300 K a monoclinic model of P112$_1$/a provides a significantly better fit. For x=0.15, 0.175, and 0.33 the structure is orthorhombic in the temperature regime investigated (10-300 K). Finally, for La$_{0.67}$Sr$_{0.33}$MnO$_3$ two structural models having R$\bar{3}$c and P112$_1$/a symmetry were assumed in the refinements, and the results indicate that the monoclinic model yields significantly smaller Rfactors and better fits.

A ferromagnetic ordering occurs for all the compounds studied. For the Ca-doped system the Curie temperature T$_c$ increases from ~150 K for x=0 to ~250 K at x=1/3. For the x=0.33 material where the magnetoresistance anomalies are largest a strong structural anomaly is observed at Tc, while at smaller x the variation with T is smooth through Tc. For the magnetic structures, a model with orthorhombic symmetry Pn'ma' and the moments parallel to the c-axis was used for the phases with the Pnma nuclear structure. For the P112$_1$/a nuclear structure a magnetic P112$_1$/a' model and moments parallel to c-axis was found to be appropriate, while for the R$\bar{3}$c nuclear structure a triclinic P1 magnetic structure was used, where the moments can point along either the a- or b- axis.