MLF Experimental Report	提出日 Date of Report 2011/6/30
課題番号 Project No. 2010A0058 実験課題名 Title of experiment Crystal structure analysis of Li_2MO_3 (M=Co,Ni,Mn) reduced by metal hydride - New cathode material for lithium batteries 実験責任者名 Name of principal investigator Ryoji Kanno 所属 Affiliation Tokyo Institute of Technology	装置責任者 Name of responsible person Touru Ishigaki 装置名 Name of Instrument/（BL No.） iMateria (BL No.20) 実施日 Date of Experiment 2011/1/31 - 2/1

試料、実験方法、利用の結果得られた主なデータ、考察、結論等を、記述して下さい。（適宜、図表添付のこと）
Please report your samples, experimental method and results, discussion and conclusions. Please add figures and tables for better explanation.

1. 試料 Name of sample(s) and chemical formula, or compositions including physical form.

Powdered $^7Li_2MnO_{3-d}$, was synthesized by reduction of $^7Li_2MnO_3$ using LiH. From an X-ray diffraction analysis, Li_2MnO_{3-d} has a layered structure with a space group of $C2/m$, which structure is the same as that of $R3m$ layered rocksalt structures. The lattice constants are a=4.939 Å, b=8.540 Å, c=9.619 Å, β=99.98˚. The detailed structure such as occupancy of oxygen defects and positions of a part of lithium has been not determined.

2. 実験方法及び結果（実験がうまくいかなかった場合、その理由を記述してください。）
Experimental method and results. If you failed to conduct experiment as planned, please describe reasons.

Neutron diffraction data of $^7Li_2MnO_{3-d}$ was taken at room temperature on a time-of-flight (TOF) neutron powder diffractometer at iMATERIA (BL20) using the BS (Back Scattering) bank. The specimen of ca. 1.5 cc is contained in a cylindrical vanadium cell of dimensious 10 mm inradius, 20 mm in height. Silicon power (NIST 640c) was mixed with $^7Li_2MnO_{3-d}$ powder to evaluate the accurate occupancies of lithium and oxygen. This data was analyzed by the Rietveld method using the Z-Rietveld program.

Fig. 1 shows a preliminary Rietveld analysis result using neutron diffraction data of $^7Li_2MnO_{3-d}$. Structural parameters are summarized in Table 1. The structure was refined based on a structure model investigated by the X-ray Rietveld analysis. The detailed structure is under analyzing to clarify (i) position of the oxygen vacancy, (ii) extent of vacancy ordering, (iii) extent of disordering at the lithium layer and (iv) stacking disorder perpendicular to the two-dimensional

2. 実験方法及び結果（つづき） Experimental method and results (continued)

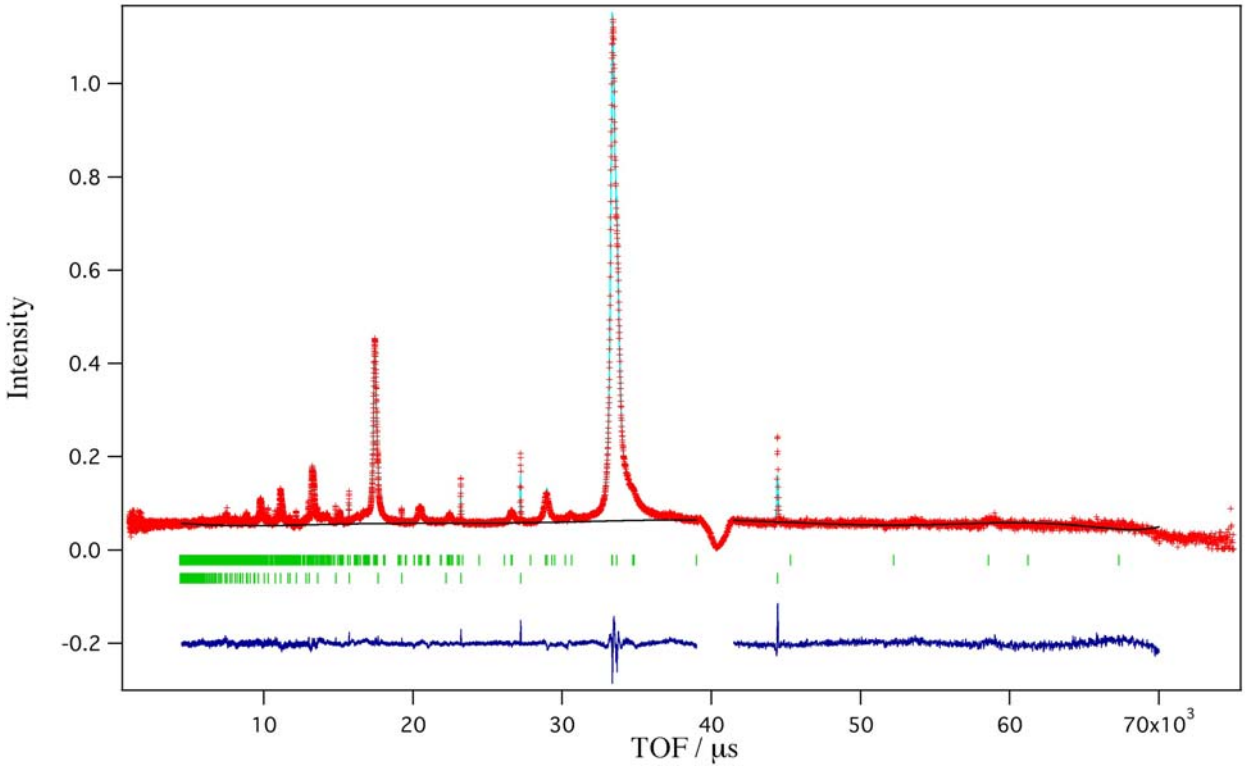

Fig. 1 Observed (plus marks), calculated (solid line), and difference (bottom) patterns for the Rietveld refinement from neutron diffraction data of $^{7}Li_2MnO_{3-d}$. The short vertical lines below the profiles mark the peak positions of all the possible Bragg reflections. Unit cell: *C*2/*m*, *a*=4.9809711), *b*=8.64234(14), *c*=5.02416(9), *β*=109.042(2), R_{wp}=0.049, R_p=0.045, R_e=0.028, R_B=0.052, R_F=0.088, S^2=2.99.

Table 1 Rietveld refinement results for $^{7}Li_2MnO_{3-d}$.

Atom	Site	*g*	*x*	*y*	*z*	*B* / Å^2
Li(1)	2*b*	0.1548	0.0	0.5	0.0	0.5
Mn(1)	2*b*	= 1 – *g*(Li(1))	0.0	0.5	0.0	0.5
Li(2)	2*c*	0.950(18)	0.0	0.0	0.5	1.0
Li(3)	4*h*	0.914(11)	0.0	0.6422(7)	0.5	1.0
Mn(1)	4*g*	0.4226	0.0	0.1643(5)	0.0	0.5
Li(4)	4*g*	= 1 – *g*(Mn(1))	0.0	= *y*(Li(4))	0.0	0.5
O(1)	4*i*	1.000(5)	0.2225(5)	0.0	0.2315(6)	0.8
O(2)	8*j*	0.836(3)	0.2495(5)	0.323742)	0.2248(4)	0.8