STRUCTURAL ANALYSIS AND PHASE FORMATION OF PbO - MnO_2 SOLID SOLUTIONS (Pb_2MnO_4 STRUCTURE)

Pwint Hlaing Ni[1], Win Win Yee[2], Zin Oo Hlaing[3],
May Aye Khine[4] and Pwint Yi Thein[5]

Abstract

PbO - MnO_2 solid solutions (Pb_2MnO_4 structure) are prepared by using starting materials, PbO and MnO_2 with equal molar ratio and solid state reaction method. The mixed samples are heat treated at 600 ^oC, 650 ^oC, 700 ^oC, 750 ^oC and 800 ^oC for 3 hrs, respectively.

After heat treatment schedule, structural properties and phase formation of the samples are characterized by using XRD. From the XRD investigation, structural properties, such as, lattice parameters, lattice micro strain and crystallite size are evaluated. Furthermore, variation of sintering temperature with structural properties are also examined.

Keywords : XRD, solid solutions, structural properties.

Introduction

The A_2BO_4 spinel oxides forms a family of ~120 compounds spanning a significant range of properties including ferro ~ and antiferromagnetism, coexistance of transparency and conductivity, superconductivity and ferroelectricity. In spinels, A and B can be divalent, trivalent, or tetravalent cations. In the normal atom pattern, the oxygen forms a cubic closed packed (face centered) array A(II) and B (III) in the tetrahedral (1/8 occupied) and octahedral (1/2 occupied) sites in the lattice. Few works are reported concerning antiferromagnetic Pb_2MnO_4 structures.

In this study, PbO - MnO_2 solid solutions (Pb_2MnO_4 structure) is investigated by using solid state reaction method and conventional funance annealing process. Structural properties and phase formation of the samples are characterized by using XRD. From the XRD investigation, variation of sintering temperature with structural properties are examined.

Experimental

PbO - MnO_2 solid solutions (Pb_2MnO_4 structures) were prepared solid state reaction method. Locally available, analar grade starting materials PbO and MnO_2 were weighted in equal molar ratio using digital balance. The two oxides powder were mixed in agate mortar for 2 hrs.

After mixing the powders, the mixture was pre heat treated at 500^oC (the melting point of MnO_2 is 535^oC) for 3 hrs. After that, the mixture was grinded with ball milling for 6 hrs to get the homogeneous mixture. Then, the mixture was heat treated again at 600^oC, 650^oC, 700^oC, 750^oC and 800^oC for 3 hrs respectively. X - rays diffraction studies were carried to determine the structural properties and phase formation of the samples by means of Rigaku Multiflux 2000 using Cu/K_α monochromatic radiation. The wavelength, voltage and current were respectively,

[1] Lecturer, Department of Physics, University of Information Technology, Yangon.
[2] Department of Physics, University of Computer Science, Yangon.
[3] Department of Physics, University of Maubin.
[4] Department of Physics, University of Kyaing Ton.
[5] Department of Physics, Nationality Youth Resources Development Degree College Yangon.

1.5418 Å, 40 kV and 50 mA. From the XRD spectra, structural properties were evaluated. Furthermore, variations of sintering temperature with structural properties were examined.

Results and Discussion

Specimen were scanned from 10^o to 70^o in diffraction angle, 2Θ, with a step size of 0.01 ^{o}/sec, as depicted in figure (1). Peak search algorithm, known as Jade software was used to identify the peaks in this study. It was obvious that, at sintering temperature at 600^{o}C, only PbO structure was found. Both PbO and Pb_2MnO_4 structures were coexisted together at sintering temperature 650^{o}C.At the sintering temperatures 700^{o}C, 750^{o}C and 800^{o}C, single phase spinel Pb_2MnO_4 polycrystalline structures were obtained and (131) peak was peak maximum, known as Bragg peak in those temperatures. Lattice parameters “a” and “c” were evaluated and listed in table (1). Crystallite size and micro strain were examined by using Debye - Sherrer equations and also listed in table (1).

According to the table (1), it is noted that, structural properties, especially, lattice parameters "a " and " c " values are nearly the same as standard values (a = 12.7854 Å and c = 5.1228 Å).It is found that, there are very small variations of lattice parameters with sintering temperature. It may be due to, solid- state- reaction mixtures become more crystalline on heating process. During the sintering temperature range from 650^{o}C to 750^{o}C, crystallite size decreases with increasing full width at half maximum of (131) peak, it is possible due to lower crystal quality. At sintering temperature 800^{o}C, crystallite size abruptly increases and the better crystal quality is obtained. This result leads to the sintering temperature 800^{o}C is the optimum condition for preparation of Pb_2MnO_4 structure in this study.

Figure (2) shows the variation of lattice parameters "a" and "c" with process temperature. Effect of process temperature on lattice distortion is depicted in figure(3). The minimum lattice distortion is occurred at temperature 800^{o}C, as seen in figure (3). Influence of process temperature on (tetragonal type) cell volume is illustrated in figure (4). The minimum cell volume is found at temperature 800^{o}C, as seen in figure (3).These results indicate, variation of process temperature with lattice distortion (c/a) and cell volume of the Pb_2MnO_4 ceramics are nearly the same, in the range of temperature 700^{o}C - 800^{o}C.

Louis Neel literally defined antiferromagnetic materials as" interesting but useless " during his Nobel lecture in 1970.Antiferromagnetic materials concentrate numerous features that encourage the research on its integration in devices for both information processing and storage. Works based on spintronic device applications on antiferromagnets have grown exponentially during the last decad.

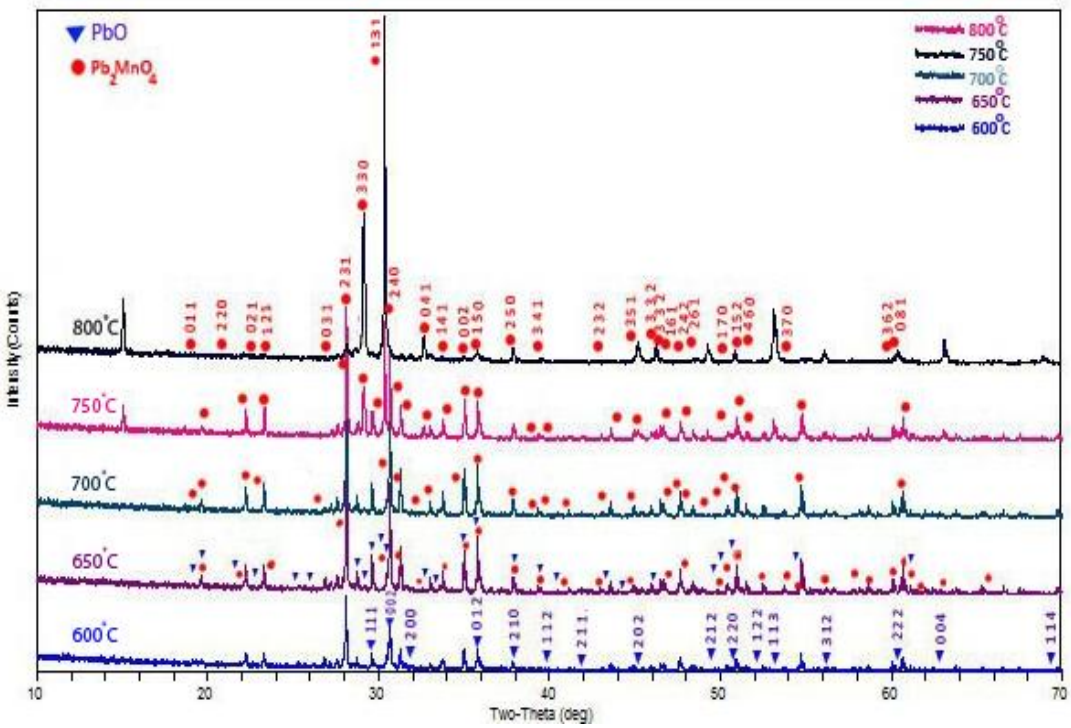

Figure 1 XRD spectra of Pb_2MnO_4 ceramics at different sintering temperatures.

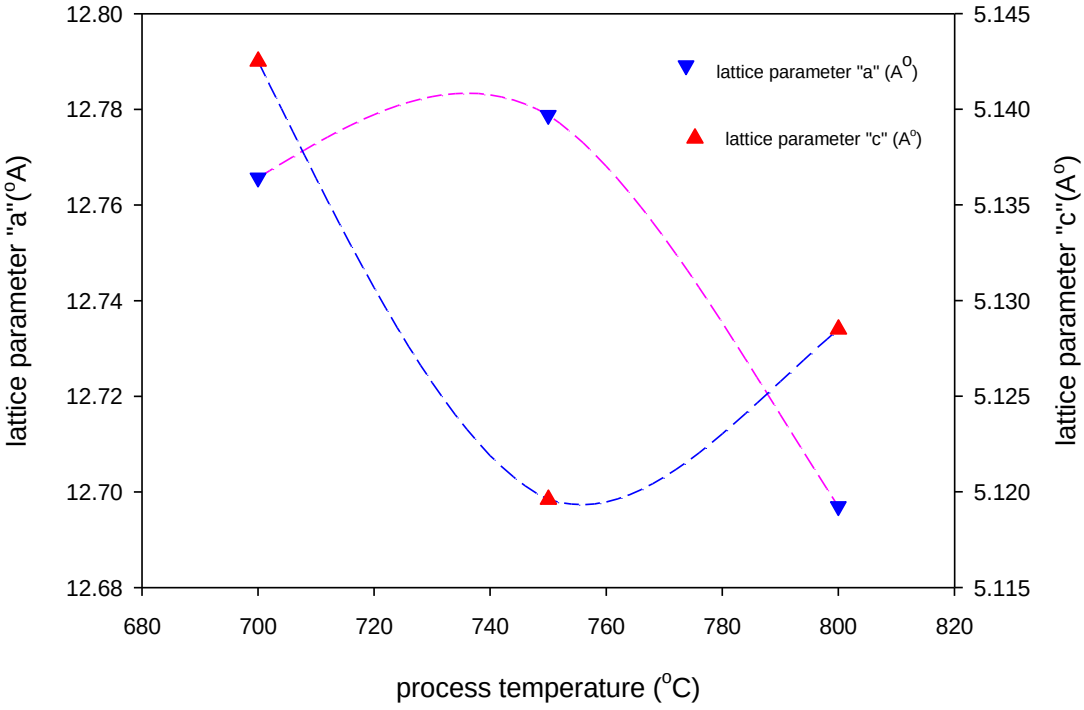

Figure 2 The variation of lattice parameters " a " and " c " with process temperature of the Pb_2MnO_4 ceramics

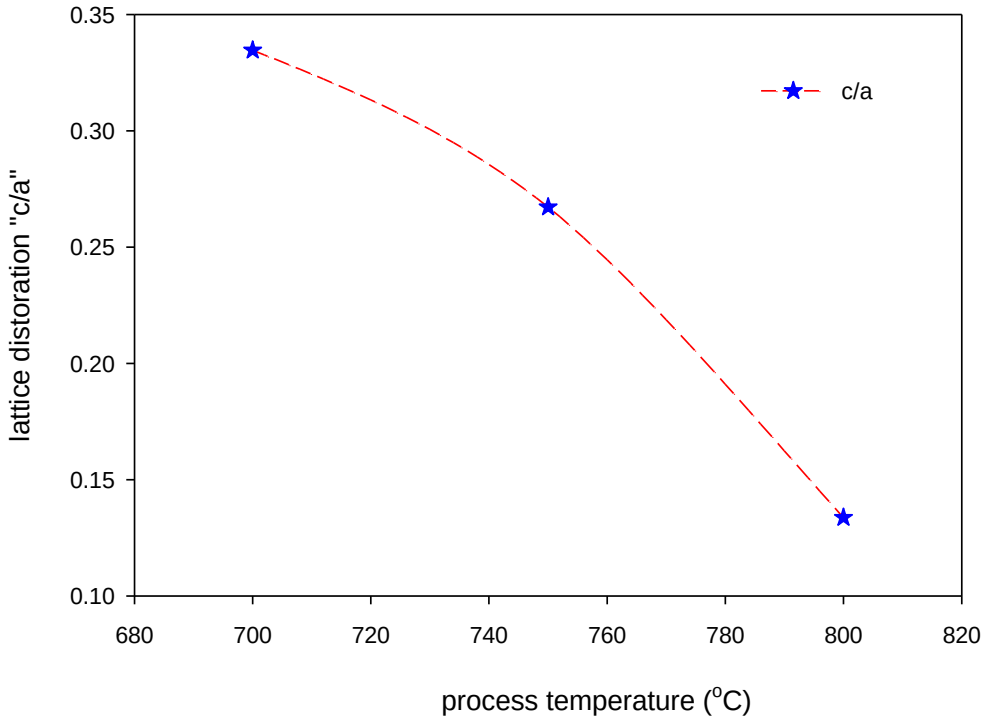

Figure 3 The variation of process temperature with lattice distortion (c/a) of the Pb_2MnO_4 ceramics

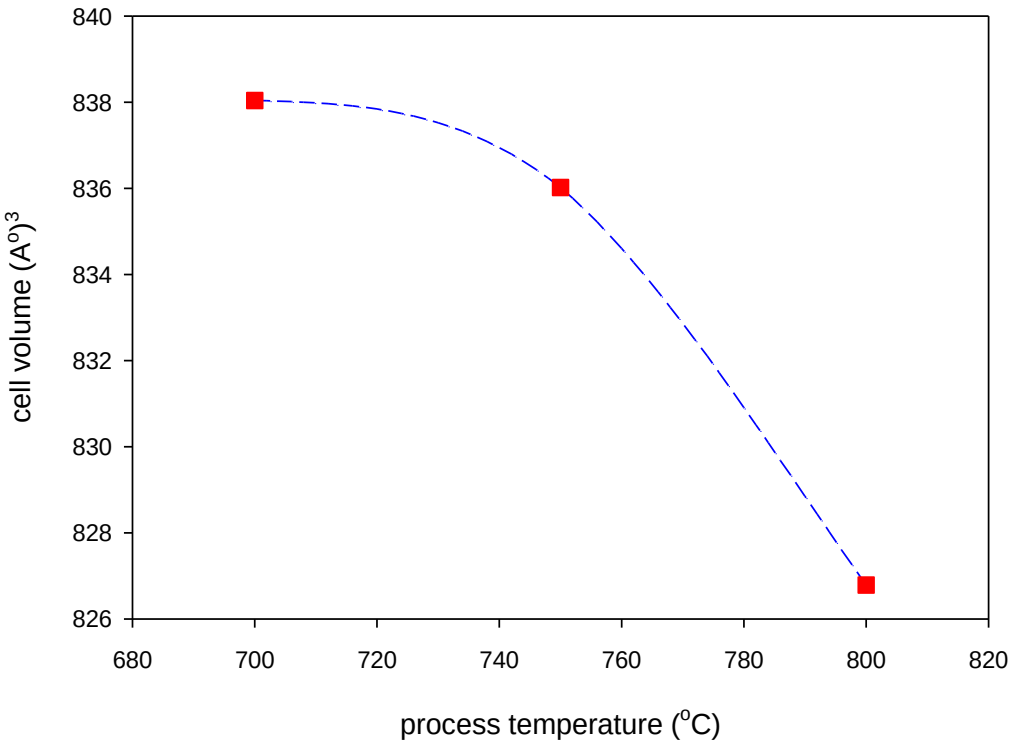

Figure 4 The variation of process temperature with cell volume of the Pb_2MnO_4 ceramics

Table1 Structural properties of Pb_2MnO_4 ceramics.

temperature ^oC	lattice parameter "a(A^o)"	lattice parameter "c(A^o)"	lattice distortion	crystallize size (nm)	micro strain	FWHM of (131) peak(^{o})
650^{o}C	12.8978	5.1162	0.2648	77.26	1.845 x 10^{-3}	0.3973
700^{o}C	12.7657	5.1425	0.3346	59.77	2.387 x 10^{-3}	0.4015
750^{o}C	12.7788	5.1196	0.2672	56.87	2.511 x 10^{-3}	0.4008
800^{o}C	12.6970	5.1285	0.3371	75.82	1.884 x 10^{-3}	0.4045

Conclusion

In this study, spinel Pb_2MnO_4 structure is examined by using conventional solid state sintering method. Experimental data, especially, lattice parameters are nearly the same as standard data.

Solid state sintering method is eco - friendly, requires less times and easy to workup. Solid state reaction mixtures becomes more crystalline on sintering process. Nowadays, research based on spintronic devices are concerned with antiferromagnets. Antiferromagnetic Pb_2MnO_4 structure is one of the possible candidates for spintronic device applications. This work may provide the new direction to the researcher into the development of Pb_2MnO_4 ceramics in short time with eco - friendly and cost effectiveness.

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