Title	Polycrystalline Phase Formation of Magnesium Ferrite, $MgFe_2O_4$ Investigated by X-Ray Diffraction and Fourier Transform Infrared Spectroscopic Methods
All Authors	[1]Shwe Sin Oo, [2]Thaung Aye, [3]Win Kyaw & [4]Win Win Thar
Publication Type	Local publication
Publisher (Journal name, issue no., page no etc.)	Journal of the Myanmar Academy of Arts and Science (Jour. Myan. Acad. Arts & Sc. 2012 Vol. X. No.2)
Abstract	Magnesium ferrite spinel, $MgFe_2O_4$ was prepared by conventional solid state reaction method at 1100°C for 22 h in vacuum chamber. Structural and vibrational characteristics of the sample were studied by X-ray diffraction (XRD) and Fourier Transform Infrared (FTIR) spectroscopic methods. Crystal structure, lattice parameters and crystallite size of the sample were examined. Vibrational characteristics and mode assignments of the sample were analyzed to investigate the polycrystalline phase formation of the sample.
Keywords	Magnesium ferrite, $MgFe_2O_4$, XRD, FTIR, polycrystalline phase formation
Citation	
Issue Date	2012

Polycrystalline Phase Formation of Magnesium Ferrite, $MgFe_2O_4$ Investigated by X-Ray Diffraction and Fourier Transform Infrared Spectroscopic Methods

[1]Shwe Sin Oo, [2]Thaung Aye, [3]Win Kyaw & [4]Win Win Thar

Abstract

Magnesium ferrite spinel, $MgFe_2O_4$ was prepared by conventional solid state reaction method at 1100°C for 22 h in vacuum chamber. Structural and vibrational characteristics of the sample were studied by X-ray diffraction (XRD) and Fourier Transform Infrared (FTIR) spectroscopic methods. Crystal structure, lattice parameters and crystallite size of the sample were examined. Vibrational characteristics and mode assignments of the sample were analyzed to investigate the polycrystalline phase formation of the sample.
Key words: Magnesium ferrite, $MgFe_2O_4$, XRD, FTIR, polycrystalline phase formation

Introduction

Spinels type AB_2X_4 compounds exist in a large variety of combinations of cations (A, B) and several different anions (X) such as sulphur, oxygen, or selenium. This leads to compounds with a wide range of physical properties, although they all have the same structure. The highly stable spinel structure allows different cations to be located on the same type of site, and owing to the site preference of the cations, many selective magnetic substitutions and various degrees of magnetic dilutions in the two sublattices may be obtained. Many oxide spinels, such as $MgFe_2O_4$, show a varying extent of cation disorder but $ZnAl_2O_4$ (Zn aluminate) is an exception, showing only small departures from the cation distribution of an ideal spinel structure. The aluminate spinels offer a good combination of physical and chemical properties, such as high melting point, high resistance and high mechanical strength; consequently, they can be used in metallurgical industry, cement, glass, etc.. These spinels also occur naturally as solid solutions [1, 5, 6].

During the last years, many kinds of ceramic oxides have been investigated actively as humidity sensing materials. Humidity sensors based on semiconducting oxides have certain advantages compared to other types of humidity sensors, such as low cost, simple construction, small size and ease of placing the sensor in the operating environment. Basically, a ceramic sensor can detect humidity on the principle of measuring a change in the

[1]Demonstrator, Department of Physics, University of Distance Education
[2]Assistant Lecturer, Department of Physics, Nationalities Youth Resource Development Degree College
[3]Lecturer, Department of Physics, University of Yangon
[4]Professor and Head, Department of Physics, University of Yangon

resistance by water vapor adsorption. Many researchers have reported the utilization of ferrites for water vapors detection as humidity sensitive active elements. Because the ferrites behave as a n type semiconductor, conductivity will be increased in the presence of water vapor. Much interest was focused on Mg ferrite owing to its high electrical resistivity and high porosity [3, 7, 8].

Materials and Methods

Preparation of Magnesium Ferrite, $MgFe_2O_4$ Spinel

The starting materials of Analar grade Magnesium Oxide, MgO and Ferric Oxide, Fe_2O_3 were weighted with stoichiometric composition. The mixtures of the powders were grounded by an agate motor for 3 h to be homogeneous and fine grain powders. The powders were then annealed at 1100°C for 22 h in the vacuum chamber by using thermal resistive heating coil that controlled FOTEK MT-20 temperature controller (2000°C) [3, 4 10]. The K-type thermocouple (1300°C) was used as the temperature sensor to read-out the real temperature of the sample. Although after reaching the annealing time, the vacuum-pump was stilled pump-down until the temperature of sample and chamber that reach to room temperature. Finally, the candidate material of $MgFe_2O_4$ ferrite spinel was obtained. Photographs of experimental arrangement of sample preparation system and $MgFe_2O_4$ ferrite are shown in Fig 1(a) and (b).

Fig 1 Photographs of (a) experimental arrangement of sample preparation system and (b) $MgFe_2O_4$ ferrite

XRD and FTIR Spectroscopic Measurements

XRD pattern of the sample was observed by RIGAKU, MULTIFLEX X-ray diffractometer using Ni-filter with CuK_α radiation, λ = 1.54056 Å. FTIR transmission spectrum of the

sample was recorded on FTIR-8400 (SHIMADZU) Spectrophotometer using Potassium Bromide, KBr pellet method.

Results and Discussion

Powder X-ray diffraction pattern of the sample is shown in Fig 2. The collected XRD data of diffraction angle (2θ), atomic spacing (d), intensity (I) and miller indices (hkl) are assigned by JCPDS (Joint Committee on Powder Diffraction Standards) and are found to agree with JCPDS. The diffraction line of (311) plane at 35.75° is the strongest one. According to XRD pattern, $MgFe_2O_4$ ferrite belongs to cubic structure at room temperature. The lattice parameters are evaluated by using crystal utility of the equation of $\frac{\sin^2\theta}{(h^2+k^2+l^2)} = \frac{\lambda^2}{4a^2}$ [2, 7]. The lattice parameters of the sample are $a = b = c = 6.44$ Å**.** The crystallite size of the sample is examined by using the Scherrer formula, $t = \frac{0.9\lambda}{B\cos\theta}$, where t = thickness of the crystallites (nm), λ = wavelength of incident X-ray (nm), θ = diffraction angle of the peak under consideration at FWHM (°) and B = the observed FWHM (radians). The crystallite size is 51.86 nm. The line at 35.75° or (311) is used to examine "*t*".

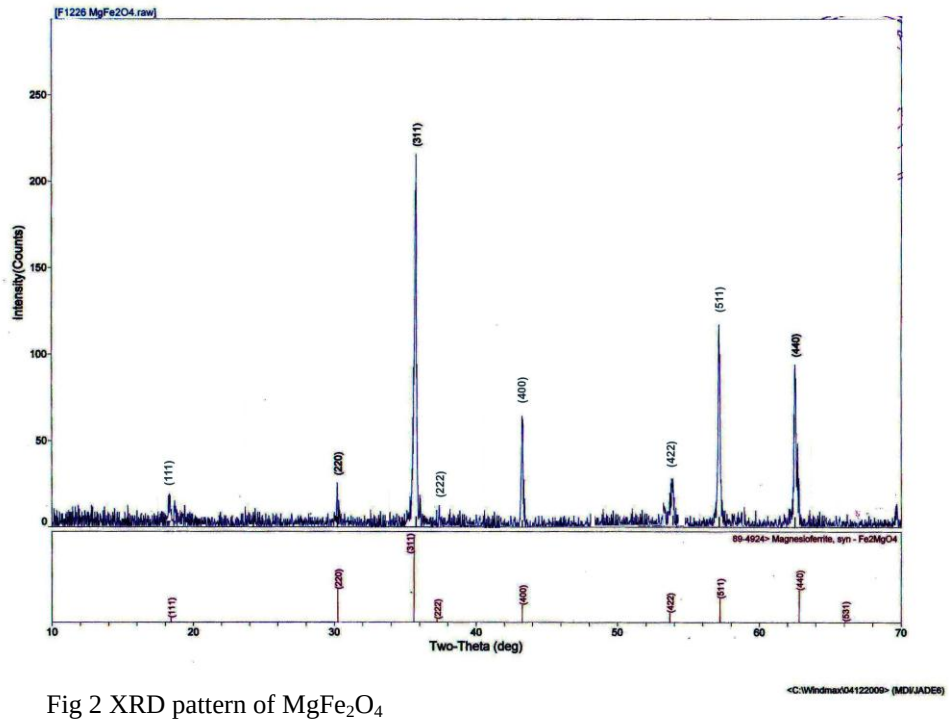

Fig 2 XRD pattern of $MgFe_2O_4$

FTIR transmission spectrum of the sample is shown in Fig 3. The observed wavenumbers and corresponding vibrational characteristics and modes assignments of molecules are tabulated in Table 1. Generally, $MgFe_2O_4$ is composed of two molecular groups; name as MgO (called group *A* molecule) and Fe_2O_3 (called group *B* molecule). The starting materials of MgO has

two types of vibrations, namely; longitudinal-optical (LO) and transverse-optical (TO) stretching vibrations. However, vibrational frequencies of LO modes are located in lower

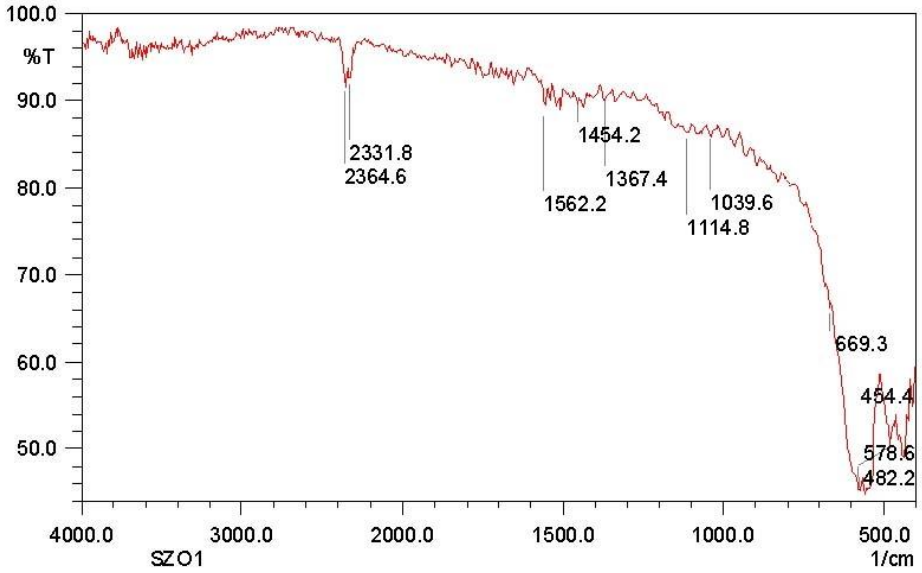

Fig 3 FTIR transmission spectrum of $MgFe_2O_4$

Table 1 Wavenumbers (frequencies) and corresponding vibrational characteristics and mode assignments of $MgFe_2O_4$ spinel

Line No	Wavenumber (cm^{-1})	Vibrational Characteristics and Mode Assignments	Molecules
1	454	ν_2-mode [(Fe---O)(E_{1u})]-bending	Fe_2O_3 (starting material)
2	482	ν_3-mode [(Fe---O)(F_{1u})]-symmetric deformation	Fe_2O_3 (starting material)
3	579	ν-internal mode of tetrahedron	Fe_2O_3 (B) in $MgFe_2O_4$
4	669	ν-internal mode of octahedron	MgO (A) in $MgFe_2O_4$
5	1040, 1115, 1367, 1454, 1562	ν-molecular group vibration	$MgFe_2O_4$
6	2332, 2365	ν_2-mode (O---C---O)-bending	CO_2

wavenumbers region (< 400 cm^{-1}). Also, Fe_2O_3 has six types of normal vibrations such as ν_1-mode [(Fe---O)(A_{1u})]-stretching, ν_2-mode [(Fe---O)(E_{1u})]-bending, ν_3-mode [(Fe---O)(F_{1u})]-symmetric deformation, ν_4-mode [δ(O---Fe---O)(F_{1u})]-bending (in plane), ν_5-mode [δ(O---Fe---O)(F_{2g})]-bending (out of plane) and ν_6-mode [δ(O---Fe---O)(F_{2u})-bending (out of plane) vibrations. Among them, bending vibrations of ν_4-mode, ν_5-mode and ν_6-mode are located in low frequencies region of 200 cm^{-1} - 400 cm^{-1} [9].

As shown in recorded FTIR spectrum, eleven absorption lines are observed and assigned by using molecular vibrational theory. The lines at 454 cm^{-1} and 482 cm^{-1} are indicated by the ν_2-mode [(Fe---O)(E_{1u})]-bending and ν_3-mode [(Fe---O)(F_{1u})]-symmetric deformation of starting materials Fe_2O_3. The lines at 579 cm^{-1} and 669 cm^{-1} are represented by ν-internal mode of tetrahedron and ν-internal mode of octahedron of the sample. The lines at 1040 cm^{-1}, 1115 cm^{-1}, 1367 cm^{-1}, 1454 cm^{-1} and 1562 cm^{-1} are ν-molecular group vibrations of $MgFe_2O_4$. Moreover, the line at 2332 cm^{-1} and 2365 cm^{-1} are represented by the ν_2-mode (bending) of CO_2 (carbon dioxide) molecules.

Conclusion

Magnesium ferrite, $MgFe_2O_4$ was prepared by conventional solid state reaction method. The sample was characterized by XRD and FTIR spectroscopy. From the XRD pattern, the sample belongs to cubic structure at room temperature and the lattice parameters are $a = b = c = 6.44$ Å. From the FTIR spectrum, eleven absorption lines are observed and these lines are represented by four types of molecular vibrations. The sample is single-phase polycrystalline materials and it can be considered as the transformer cores and humidity sensors.

Acknowledgement

The authors would like to acknowledge Professor Dr Pho Kaung, Pro-Rector and Director of Asia Research Centre (ARC), University of Yangon for his permissible to use the Universities Research Centre (URC) facilities.

References

1. Candeia R A et al 2004 Materials Letters 58 569
2. Cullity B D 1978 "Elements of X-Ray Diffraction" (Reading: Wesley)
3. Moulson A J & Herbert J M 1997 "Electroceramics Materials, Properties & Applications" (London: Chapman & Hall)
4. Nell J & Wood B J 1991 American Mineralogist 76 405
5. Sinha M M & Kim J S 2003 Journal of Korean Physical Society 43 (2) 237
6. Pillai S O 2006 "Solid State Physics" (New Delhi: New Age)
7. Suryanarayana C & Norton M G 1998 "X-Ray Diffraction: A Practical Approach" (New York: Plenum)
8. Ross S D 1972 "Inorganic Infrared and Raman Spectra" (London: McGraw-Hill)
9. 2001 “Vacuum Technique and Thin Film Deposition” Experiment 3, 1 Laboratory Instruction, (California Institute of Technology: California)