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Effect of Presintering Temperature on XRD of Lithium Ferrites

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ABSTRACT: Lithium ferrite with general formula $\text{Li}_{0.5}\text{Fe}_{2.5}\text{O}_4$ were prepared by standard ceramic method by presintering at 500°C , 600°C and 700°C temperatures separately and final sintering for all the samples at 950°C temperature. XRD studies confirms single phase formation and lattice parameter were calculated. Lattice parameter increases with increase in presintering temperature i.e., $8.259 \text{ \AA}$ for 500°C and $8.3028 \text{ \AA}$ for 700°C . With increase in calcining temperature the amount of spinel phase formation increases which are in good agreement with earlier reports.

KEYWORDS: Ceramic oxides, Ferrites, Lithium ferrites

I. INTRODUCTION

Ferrites may be defined as magnetic materials composed of oxides containing ferric ion as the main constituent. Ferrites are ceramic materials dark grey or black in appearance and very hard and brittle. Based on structure of Ferrites they are of three types. 1. Spinel structure – Spinel have a cubic structure and general formula $\text{M}^{2+}\text{Fe}_2^{3+}\text{O}_4^{2-}$ where M^{2+} is divalent metal ion. 2. Garnet structure – with general formula $\text{A}^{3+}\text{Fe}_5^{3+}\text{O}_{12}^{2-}$ where A^{3+} is rare earth element or yttrium ion. 3. Hexagonal structure – with general formula $\text{A}^{2+}\text{Fe}_{12}^{3+}\text{O}_{19}^{2-}$ where A^{2+} is Barium. Lithium ferrites with general formula $\text{Li}_{0.5}\text{Fe}_{2.5}\text{O}_4$ is the spinel ferrites with face centered cubic crystal structure. Lithium ferrite possesses high curie temperature about 650°C and rectangular hysteresis loop characteristic. Lithium ferrite materials have a lot of microwave applications. Suitable substitution in Lithium ferrite develops materials suitable for the microwave devices [1]. Lithium ferrite materials with Co, Mn, Ti and Al substitutions are used in radars. Haurt et al studied Lithium ferrite as n-type semiconducting material in photoelectrochemical cells for solar energy conversion [2].

II. METHOD OF PREPARATION

Hilpert [3] and Snoek [4] had done pioneering work on the development of the ferrite. For the last fifty years various laboratories are involved to cater to the qualitative and quantitative demands of the industry. Saturation magnetization (Ms), coercive field (Hc), initial permeability (μ_i) and its dependence on frequency, temperature and applied magnetic fields are some of the technically important properties for applications. The most acceptable technique which is the commercially used is the classical ceramic method in which starting materials i.e. oxides and carbonates are chosen properly characterized for their purity, particle size and shape. The starting materials should have enough reactivity so that on mixing and calcinations, a homogenous ferrite can be prepared by wet mixing of these oxides and carbonates in the mortar. Two steps of sintering i.e. presintering and final sintering is an important process which controls the extrinsic properties of the ferrite.

III. SYNTHESIS OF LITHIUM FERRITES UNDER INVESTIGATION

Classical ceramic method was used for the preparation of Li- ferrites with general formula $\text{Li}_{0.5}\text{Fe}_{2.5}\text{O}_4$ using high purity AR grade Li_2CO_3 and Fe_2O_3 . These powders were weighed in the required mole proportions on a semimicro balance having least count of 0.001mg and were mixed thoroughly in an agate mortar in acetone medium. This powder was presintered at 500°C , 600°C and 700°C for 10 hours, in a glow bar furnace in air medium separately. Calibrated chromelalumel thermocouple was used to measure the temperature of the furnace. This presintered powder was again grounded in an agate mortar in acetone medium. Thus, obtained fine powders of the ferrites was then sieved through a 100-micron mesh. The mixture was then compressed in the form of pellets by using 1cm die and by applying pressure of about 7 to 8 tons per square inch for 2 minutes. To reduce lithium loss all the three samples were finally sintered at 950°C for 20 hours in air medium and were cooled in furnace atmosphere [5].

For X-ray diffraction studies, the obtained powder by grounding the pellets and sieved through 100-micron mesh were used.

IV. X-RAY DIFFRACTION STUDIES

X-ray diffraction studies in the present case has been used to i. confirm the single-phase formation ii. determine the lattice parameter, bond length and theoretical density iii. To study the effect of presintering temperature on the properties of the Li-ferrite. XRD patterns were recorded by using Philips PW 1718 diffractometer available at CFC, Shivaji university, Kolhapur. The Cu-K α radiation with wavelength (λ) = 1.5418 Å was used. The samples were scanned within the angle (2θ) between 15° to 85° at the rate of 2° per minute. The diffractograms were indexed in the light of the crystal structure of a natural spinel MgAl₂O₄. The prominent peak in the XRD pattern corresponds to (311) plane was used to calculate lattice parameter. For the peaks corresponding to other planes, we have calculated the lattice parameter, interplanar distance and miller indices using the usual procedure

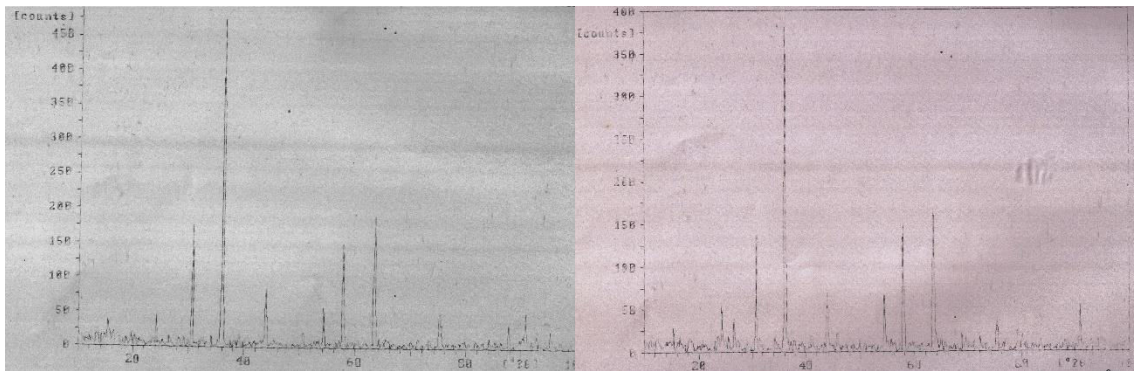


Fig.1 XRD pattern of sample sintered at 500°C Fig.2 XRD pattern of sample sintered at 600 °C

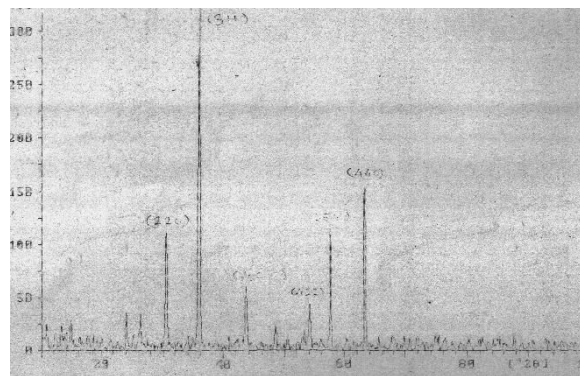


Fig.3 XRD pattern of sample sintered at 700 °C

The data on lattice parameter and interplanar distance is given in the table 1,2 and 3. The observed and calculated d values are in good agreement for all indexed planes.

The lattice parameter for Li-ferrite has been reported by many workers [6,7,8]. In our case the lattice parameter of the sample presintered at 700°C is 8.3028 Å. Kang et al (9) reported the effect of presintering temperature on the properties of Mg-Mn ferrites. They found that the grain size and density of the specimen sintered were dependent on calcining temperature. Calcining temperature also determines the amount of spinel formation which increases with increasing calcining temperature and saturates above 800 °C in case of Mg-Mn ferrites. In our case the lattice parameter is also increases with increasing calcining temperature.



Table 1
X-ray diffraction data of $\text{Li}_{0.5}\text{Fe}_{2.5}\text{O}_4$ ferrite presintered at 500 °c
Wavelength-1.5418 Å⁰
Lattice parameter (a) – 8.259 Å⁰
Structure – Cubic

Sr.No.	2θ, deg.	d calc. Å ⁰	d Obs.Å ⁰	(hkl)
1.	30.66	2.9201	2.9131	(220)
2.	36.03	2.4903	2.4903	(311)
3.	43.71	2.0648	2.0692	(400)
4.	54.22	1.6859	1.6902	(422)
5.	57.74	1.5895	1.5954	(511)
6.	63.35	1.4660	1.4668	(440)

Table 2
X-ray diffraction data of $\text{Li}_{0.5}\text{Fe}_{2.5}\text{O}_4$ ferrite presintered at 600 °c
Wavelength-1.5418 Å⁰
Lattice parameter (a) – 8.2394 Å⁰
Structure – Cubic

Sr.No.	2θ, deg.	d calc. Å ⁰	d Obs.Å ⁰	(hkl)
1.	30.67	2.9130	2.9122	(220)
2.	36.12	2.4843	2.4843	(311)
3.	43.84	2.0598	2.0634	(400)
4.	54.25	1.6818	1.6893	(422)
5.	57.75	1.5856	1.5950	(511)
6.	63.42	1.4565	1.4655	(440)
7.	75.10	1.2564	1.2639	(533)
8.	79.08	1.1892	1.2100	(444)

Table 3
X-ray diffraction data of $\text{Li}_{0.5}\text{Fe}_{2.5}\text{O}_4$ ferrite presintered at 700 °c
Wavelength-1.5418 Å⁰
Lattice parameter (a) – 8.3028 Å⁰
Structure – Cubic

Sr.No.	2θ, deg.	d calc. Å ⁰	d Obs.Å ⁰	(hkl)
1.	30.51	2.9319	2.9271	(220)
2.	35.88	2.5004	2.5004	(311)
3.	43.57	2.0732	2.0753	(400)
4.	54.08	1.6927	1.6942	(422)
5.	57.55	1.5959	1.6002	(511)
6.	63.18	1.4659	1.4704	(440)



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