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Synthesis of lithium zinc ferrite powders by citrate precursor gel formation method

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Abstract · A nitrate-citrate gel was prepared from metal nitrates and citric acid, inorder to synthesize lithium zinc ferrites The nitrate-citrate gel so prepared exhibits self-propagating combustion behaviour After combustion the synthesized powder is densified at a temperature of 900°C for 3 hours. In this method of synthesis, the sintering temperature is lower than that in ceramic process and calcination is not required

Keywords . Soft Ferrites, Citrate precursor method, Nanoparticles

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1. Introduction

New development and innovative ideas in the area of material processing have often led to the discovery of new materials with interesting and useful properties. Lithium and substituted lithium ferrites have been found to be very good materials in microwave devices due to their low costs, high resistivity and low eddy current loss. In the preparation of lithium based ferrites low temperature sintering is desirable to suppress lithium volatility and oxygen loss during firing [1], which have detrimental effect on their various properties. In order to prepare ferrites at low temperature usually liquid phase agents such as bismuth oxide are added. The additives tend to form glassy phase at grain boundaries enhancing densification [1,2]. However, they may tend to deteriorate some of the properties of certain ferrites [3]. Therefore, low temperature sintered ferrites without additives are a hot topic of research. This has been found to be achieved by using active ultra fine powders, which can be synthesized by wet chemical methods like the citrate precursor synthesis, which has been proved to be very simple and economic, and the particles are of the order of

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nanoscale [3-5]. Nanostructured material hold promises for dramatic changes in future research direction.

In the present work lithium zinc ferrites has been synthesized by using citrate precursor method. The idea behind this technique is a thermally induced anionic redox reaction where the exothermic reaction between oxidant and reductant is high enough to form the desirable phase within a short time.

2. Experiment

Lithium zinc ferrites with compositional formula $Li_{0.5-0.5x}Zn_xFe_{2.5-0.5x}O_4$ where $x = 0.0, 0.2, 0.4$ were prepared by a citrate precursor method. The starting chemicals used in this study were lithium hydroxide, iron nitrate, zinc nitrate and citric acid.

Appropriate amount of lithium hydroxide was dissolved in dilute nitric acid. This was added to a beaker containing stoichiometric amount of zinc nitrate and iron nitrate. The solution was made to mix properly and citric acid was added to it. The ratio of metal cations to citric acid is 1:1. The solution was homogenously mixed with the help of a magnetic stirrer using a magnetic agitator until a transparent clear solution occurs. Ammonia solution was added drop by drop until the pH becomes 7. During this process change in colouration occurs, passing through 3 colour stages. After controlling pH at 7 it was refluxed at 40°C with continuous stirring for about half an hour. The solution was then put in oven at 100°C. Viscosity and colour changed as sol turned into a brown puffy porous dry gel. The dried gel then gets ignited, undergoing a strong auto combustion process with evolution of large amount of gases, which started in the hottest zone of the beaker and propagated from the bottom to the top like the eruption of a volcano, giving rise to dark grey voluminous product with structure similar to a branched tree, as shown in Figure 1 for $x = 0.2$ and 0.4. The ash-synthesized product so obtained is the typical

(a) (b)

Figure 1. Picture of a branched voluminous product obtained from auto combustion for (a) $x = 0.2$ and (b) $x = 0.4$

spinel structured lithium ferrite powder with nanocrystallite size. The synthesized powder was mixed with polyvinyl alcohol as binder and pressed into pellets with 50 kilo Newton pressure. All the three samples were sintered at 900°C for 3 hours in air.

3. Results and discussion

XRD pattern of the prepared samples confirmed the spinel structure and are shown in Figure 2. It has been reported by earlier workers that the spinel phase is generally formed in the ash prepared powder.

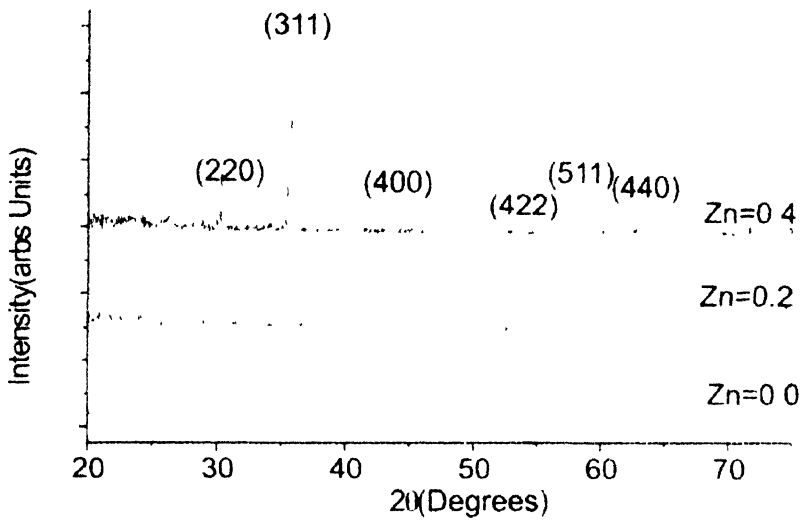

Figure 2. XRD pattern for x = 0 0, 0 2 and 0 4

From an analysis of the XRD data, the average crystallite size of the synthesized powder was estimated using Debye Scherrer equation and is found to be 21nm, 21nm, 29nm for x = 0.0,0.2,0.4 respectively. It is therefore observed that citrate precursor is a simple method to synthesize highly homogenous lithium zinc ferrite with nanosized crystallites. This process has various advantages over the other methods of preparation. The process is based on aqueous system and, therefore, it neither requires expensive chemicals as alkoxide based sol gel process nor special equipment like autoclaves for combustion synthesis. Another advantage is that the ash burnt powder does not require calcination at high temperature as in the conventional ceramic process, to transform into final powder with expected crystal structure. The phase is generally formed right at he moment of auto combustion. Thus the synthesized powder can be sintered at a comparatively low temperature, thereby reducing the loss of lithia at the time of preparation.

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