

Room temperature single-step electrosynthesized copper ferrite thin films and study of their magnetic properties

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Abstract

A single-step electrosynthesis of copper ferrite thin films from aqueous bath (which avoids anodization step for an incorporation of oxygen species into deposit) has been carried out at room temperature. Observed tetrahedral structured nanocrystalline copper ferrite thin films showed smooth, uniform and compact surface morphology. After annealing, increase in dielectric constant and reduced dielectric loss were observed. The saturation magnetization for annealed films was 292 emu/cm³ comparable to that of other reported ferrites.

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1. Introduction

Granular magnetic materials, consisting of ferri or ferromagnetic monodomain particles, diluted in non-magnetic matrix are of great interest [1]. Copper ferrites have distinctive features such as phase transition from tetragonal to cubic due to the Jahn Teller distortion, electrical switching, and change in semiconducting properties, etc. [2].

High-density magneto-optic recording devices, color imaging, bioprocessing, magnetic refrigeration and ferrofluids, magnetic cores, optomagnetic devices, bubble memory devices [3,4], etc. are some of the well-known applications of copper ferrites whose intensive detail study in thin film form is still waiting. There are some reports on synthesis of copper ferrite thin films using physical methods [5,6].

Oxides are usually synthesized at relatively high temperature under a controlled pressure of oxygen. An electrochemical intercalation and de-intercalation of oxygen,

under the controlled polarization find one of suitable chemical methods in air, at room temperature and in alkaline media gives rise to oxide formation [7]. Also by cathodic electrodeposition various oxide materials have deposited at room temperature [8]. Instead of anodization, oxide materials can be cathodically electrodeposited by applying higher deposition potential from highly alkaline bath. Surface precipitation of hydroxides resulting from an increase in pH of solution adjacent to the cathode results into the formation of oxides [9].

Ferrite thin film synthesis by physical methods involves mainly two steps; first, the alloyed films are formed with appropriate compositions on conducting substrates and then oxidized at high temperature. The high-temperature oxidation is critical and affects the reproducibility and quality of the films and therefore electrochemical oxidation (anodization) at ambient temperature has received considerable interest.

Electrodeposition is a powerful and interesting process that can be applied to the synthesis of films and powders because of the high energy density accumulated in a solution near to an electrode's surface. It has advantages compared with other techniques, which include: (i) low

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processing temperature, (ii) low cost of raw materials and equipment, (iii) control of the film composition and morphology using the electrochemical parameters, and (iv) the ability to deposit a film on a complex surface. This is probably the easiest, non-vacuum method for preparing large-area electrodes.

Few years back, we already reported synthesis of ferrite thin films in two-steps using at ambient temperature, i.e. formation of alloyed film and then its conversion into oxides through the process of anodization in 1 M KOH solution [10]. To the best of our knowledge there are no reports in the literature to prepare single-step synthesis of ferrite thin films. We here reported for the first time a novel, more economical, simple and easy going process for copper ferrite thin films synthesis in one-step onto stainless steel substrate and characterized for structural, surface morphological properties. The effects of bath composition, deposition potential, current density, deposition time, and bath temperature were studied and optimized to get good quality copper ferrite thin films. Further, the changes in magnetic and dielectric properties after annealing were studied.

2. Experimental and characterization details

The solutions of copper sulphate, iron sulphate, citric acid, and NaOH were prepared in triply distilled water. Atomic absorption was preferred for 1:2 chemical elemental stoichiometry copper and iron with the bath composition of 0.05 M CuSO_4 and 0.1 M FeSO_4 solutions. To make alkaline bath (pH~10–11), 1 M NaOH solution was added. The hydroxide precipitation was avoided with 0.1 M citric acid as a complexing agent. Direct single-step deposition was carried out onto stainless steel substrates at room temperature (300 K) by fixing the deposition potential from cyclic voltametric curves.

After the deposition, annealing of copper ferrite was carried out for 5 h at 773 K and slowly cooled in the furnace. Above 773 K, due to the different thermal expansion coefficients of stainless substrate and copper ferrite material, the films were peeled off from the substrate. Structural characterization of as-deposited and annealed copper ferrite thin films was carried out using X-ray diffraction and infrared spectroscopy techniques. Philips PW-3710 X-ray diffractometer with a copper anode using Ni filtered $\text{CuK}\alpha$ radiations operated at 25 kV and 20 mA was used for structural analysis. For the surface morphological studies, scanning electron microscopy (SEM) and atomic force microscopy (AFM) techniques were used. Dielectric properties of stainless steel substrate- CuFe_2O_4 -copper press contact (M-O-M) structure were studied with the help of LCR Meter Bridge for various frequencies at room temperature. Magnetic properties were studied using vibrating sample magnetometer (VSM) technique, applying magnetic field in the plane of films.

3. Results and discussion

3.1. Optimization of preparative parameters for stoichiometric single-step ferrite film formation and the growth mechanism

The composition of electrodeposited material in binary metals depends upon concentration of parent metals in an electrolyte. The variation of metal ions the concentration of parent metals, in a fixed quantity of electrolyte is one of the easy way to optimize the bath composition [11].

Bath of fixed quantity (20 cc) contains mixture of 0.05 M CuSO_4 and 0.1 M FeSO_4 solutions. We have optimized bath composition by varying quantity of 0.1 M FeSO_4 solution. The copper ferrite thin films from various bath compositions were electrodeposited onto stainless steel substrates to which pre-electroforming treatment was given [12]. After the deposition, film was detached from the substrate and dissolved into nitric acid and further nitric acid was diluted to known quantity. By using atomic absorption spectroscopy technique, atomic percentage of Fe in the deposit was estimated and is shown in Fig. 1 for fixed quantity of 0.1 M FeSO_4 solution in the bath.

From Fig. 1, it is observed that the film deposited using 4 cm^3 of 0.05 M CuSO_4 and 16 cm^3 0.1 M FeSO_4 solution contains Cu~33% and Fe~66% in the deposit. Therefore for further deposition, above solution composition was fixed. Oxygen incorporated into the deposit during the deposition was reduced when film was dissolved into concentrated nitric acid as it does not affect the elemental chemical stoichiometry. An optimized bath composition of 0.05 M CuSO_4 and 0.1 M FeSO_4 (pH~2.4) solution was used for direct synthesis of soft copper ferrite thin films.

Fig. 2 shows the current transient obtained during copper ferrite thin film deposition at applied potential of -0.5 V vs. SCE (obtained from cyclic voltametry curves of copper, iron, and copper-iron solutions). The three

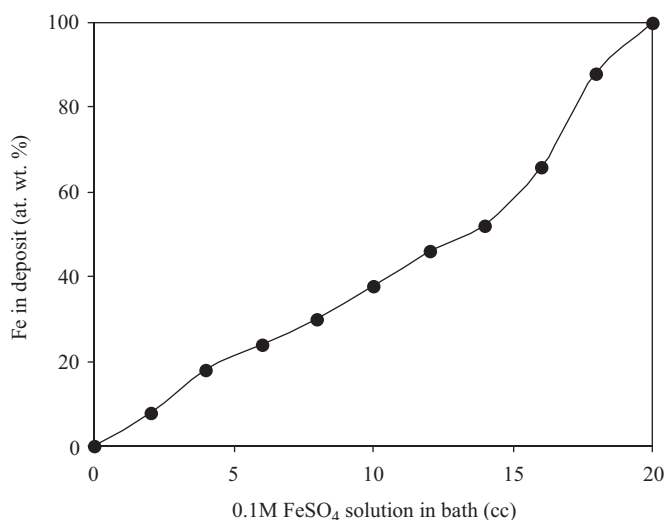


Fig. 1. The Fe (wt%) content in deposit against the quantity of 0.1 M FeSO_4 solution in bath.

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