STRUCTURAL AND MAGNETIC PROPERTIES OF ALUMINIUM-COPPER SUBSTITUTED COBALT FERRITE NANOPARTICLES SINTERED AT VARIOUS TEMPERATURES

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Dedicated to

My beloved parents, and my supportive supervisor Prof. Dr. Zulkafli Othaman.

Thank you very much for being supportive, helpful and understanding

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ABSTRACT

Nanoferrites, especially spinel ferrites have evoked greater attention in the recent years because of their many potential technological applications. Properties of nanoferrites could be tailored and improved by the incorporation of suitable divalent, trivalent or tetravalent ion impurities into the spinel lattice. Hence the investigation on the properties of substituted nanoferrites helps to improve the performance of these materials and make their applications more diverse. Study of fundamental properties of materials is crucial, as it may lead to the development of new area of potential application. Nanocrystalline copper-cobalt spinel ferrites and nanocrystalline aluminum substituted copper cobalt spinel ferrites with general formula of $Co_{1-x}Cu_xFe_{2-x}Al_xO_4$, ($0.0 \leq x \leq 0.8$) were synthesized using co-precipitation method and sintered from 600 °C to 900 °C. The synthesized nanoferrite samples were characterized using X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FTIR), field-emission scanning electron microscopy (FE-SEM), energy dispersive X-ray spectroscopy (EDX) and vibrating sample magnetometer (VSM). XRD analysis confirmed a single phase spinel structure with the crystalline size between 14-26 nm which is calculated using Scherrer's formula. This size is the most suitable for high density recording media application. The infrared spectra reveal two prominent frequency bands in the wavenumber range of 350–600 cm^{-1}, which confirm the cubic spinel structure and completion of chemical reaction. Substitution of aluminum and copper to cobalt ferrites has made significant changes in the magnetic properties of nanoferrites. Saturation magnetization was observed to decrease by copper and aluminum substitution from 61.6–17.6 emu g^{-1} at 600 °C to 49.5–25.8 emu g^{-1} at 900 °C. The increasing trend of magnetic coercivity from 846.9–1117.2 O_e at 600 °C to 219.4–244.0 O_e at 900 °C was consistent with crystallinity of the samples. The simple, economic and environmental friendly sample preparation method using co-precipitation method has contributed towards the controlled growth of high quality ferrite nanopowder and the suitable values of magnetization that can be applied to high-density recording media and microwave devices.

ABSTRAK

Nanoferit, khususnya spinel ferit telah mendapat perhatian meluas kebelakangan ini disebabkan potensi pelbagai aplikasi dalam teknologi. Sifat nanoferit boleh diubahsuai dan dipertingkatkan dengan menambahkan ion dwi-valensi, tiga valensi atau empat valensi bendasing yang sesuai dalam kekisi spinel ferit. Justeru, penyelidikan ke atas sifat nano ferit gantian membantu meningkatkan prestasi bahan dan membolehkan aplikasinya lebih menyeluruh. Kajian asas sifat bahan adalah penting, kerana ia mungkin boleh menjurus kepada era baru pembangunan aplikasi berpotensi. Nanokristal kuprum-kobalt spinel ferit dan nanokristal penggantian aluminum kuprum-kobalt spinel ferit dengan formula asas $Co_{1-x}Cu_xFe_{2-x}Al_xO_4$, ($0.0 \leq x \leq 0.8$) telah disintesis menggunakan kaedah pemendakan dan pensinteran dari 600 °C hingga 900 °C. Sampel sintesis nanoferit telah dicirikan dengan menggunakan kaedah pembelauan sinar-X (XRD), Fourier spektroskopi inframerah (FTIR), mikroskop pengimbas elektron pengeluaran medan (FE-SEM), spektroskopi sinar-X sebaran tenaga (EDX) dan magnetometer sampel bergetar (VSM). Analisis XRD mengesahkan spinel adalah berstruktur satu fasa, dengan saiz kristal antara 14 -26 nm yarg dikira menggunakan formula Scherrer. Saiz ini paling sesuai untuk aplikasi media perakam ketumpatan tinggi. Spektrum inframerah menonjolkan dua jalur frekuensi yang utama dalam julat gelombang 350–600 cm^{-1}, yang mengesahkan struktur spinel kubik dan tindakbalas kimia lengkap. Penggantian aluminium dan kuprum kepada kobalt ferit telah menyebabkan perubahan ketara pada sifat magnet nanoferit. Pemagnetan tepu didapati menurun dengan penggantian kuprum dan aluminium dari 61.6–17.6 emu g^{-1} pada 600 °C kepada 49.5–25.8 emu g^{-1} pada 900 °C. Pola pertambahan koersiviti dari 846.9–1117.2 O_e pada 600 °C kepada 219.4–244.0 O_e pada 900 °C adalah sekata dengan sampel berfasa kristal. Kaedah mudah, ekonomi dan mesra alam dalam penyediaan sampel menggunakan kaedah pemendakan ini telah menyumbang kepada pertumbuhan terkawal serbuk nano ferit berkualiti tinggi dan nilai pemagnetan yang sesuai diaplikasikan kepada media perakam ketumpatan tinggi dan peranti gelombang mikro.

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LIST OF ABBREVIATIONS

EDX - Energy dispersive X-ray
FESEM - Field emission scanning electron microscopy
FT-IR - Fourier transform Infrared
FWHM - Full with at half maximum
LCR - Inductance capacitance resistance
TPP - Two point probe
VSM - Vibrating sample magnetometer
XRD - X-ray Diffraction

LIST OF SYMBOL

μ_0 - Permeability
Å - Angstrom
A - Area
a - lattice constant
a, - Lattice parameter
Co - Cobalt
Cu Kα - Copper K-alpha line
d - Interplanar distance
D - crystalline size
L - Jump length

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CHAPTER 1

INTRODUCTION

1.1 Background of study

For the last decades nanomaterials (crystallite size <100 nm) have been considered the most popular emerging field of research. Many studies have been focused on the preparation of novel nanometal oxides due to their exclusive size based properties. Recently the nanomaterial is of great importance in the field of nanoscience and nanotechnology, and becomes a major concern in scientific research areas such as materials science, optics, electronics and magnetics. In respect of emergent technologies, nanomagnetism has taken a prime place in research in magnetic materials, which has evolved new ideas in the field of nanoscience. In the last few decades interest in nanoscience and nanotechnology has increased manifold due to its promising technological applications [1]. Study of nanomagnetic materials is a remarkable field at the frontiers of physics and chemistry. The domain structure, magnetic interactions and the basic properties of these materials are of considerable importance to modern technology. Developing various methods to generate nanostructures and understanding their complex phenomenon with supporting analysis has been a challenging field in research and development.

Nanosized magnetic oxides are becoming important materials due to their micro magnetic properties such as superparamagnetism, giant magnetic resistance, magneto optic, magneto caloric effect, etc. These materials are useful in variety of applications in electronic industries due to their high saturation magnetization, high resistivity, high permeability and low loss [2]. There is a great demand for high performance and miniaturization of electronic devices. Soft ferrite nanomagnetic materials are extensively used in inductors, transformers, antennas, electromagnetic interference. [3]

Magnetic materials are used in a large variety of devices like passive circuit elements, sensors, reading-writing heads, information storage media, etc. The synthesis and understanding of their properties are essential requirements for their integration in a certain industrial fabrication process. One class of magnetic materials which is of great interest in recent years is that of ultra-soft magnetic materials [4] . These materials can be used to extend the operation frequency range of various passive circuit elements into the GHz regime. Electromagnetic devices operating in the microwave frequency range and using ferrites as an essential ingredient can broadly be subdivided into two categories [5]. The first class of devices is makes use of the nonreciprocal behavior obtainable with ferrites and the second is based on the fact that the microwave behavior can be substantially modified by the application of a biasing field. The first class is comprises primarily isolators and circulators, and the second class is belongs to primarily switches, phase shifters, and tunable filters. For non-reciprocal devices the use of ferrites are appears inevitable for the foreseeable future, since no alternative technology that could achieve similar circuit functions appears on the horizon. For variable devices, however, an alternative solution (usually relying on semiconductors) is available in many instances. Thus the future use of ferrites in these applications will be governed by the details of the technical problem that needs solution as well as by economic considerations [6].

Ferrites are chemical compounds which are composed of a ceramic material and iron oxide as their main component. A ferrimagnetic ceramic compound, ferrites, has a spinel type structure. The magnetic property of the ferrite is due the structure and the distribution arrangement of the ions in the sub lattice. Most of the ferrites have a spinel structure with a formulae AB_2O_4, where “A” are divalent ions such as Mg^{2+}, Co^{2+}, Ni^{2+},Mn^{2+}, and “B” are the trivalent ions such as Fe^{3+}. Spinels structures have an oxygen ion sub lattice, in a cubic close-packed arrangement with cations occupying various combinations of the octahedral (O) and tetrahedral (T) sites. The cubic unit cell contains 8 formula units and containing 32 O and 64 T sites. Spinels are basically categorized into a normal and inverse spinel. In normal spinel, the divalent cations “A” are positioned at the tetrahedral (T) sites and the trivalent cations “B” on the octahedral (O) sites such as $ZnFe_2O_4$. $CuFe_2O_4$ or $CoFe_2O_4$ has an inverse spinel crystal structure. In inverse spinel “A” cation occupies one half of the octahedral coordination sites and half the “B” cation occupies the other half (O) sites as well as all “T” sites. In a spinel structure both atom A and B are exactly antiferromagnetic, and the spins cancel each other. The magnetic property then rises due to the 4 Fe^{2+}, which align themselves on the application of field [7, 8].

Depending on the magnetic properties, ferrites can be categorized as “soft” and “hard” ferrite [9]. Soft ferrites have low coercivity while the hard ferrites have high coercivity, and moderate magnetization. Coercivity stands for the resistance to get demagnetized on the removal of the applied field which satisfies it for being a permanent magnet. The transitions of bulk materials into nano size can lead a number of changes in their physical, electrical and magnetic properties [10] .

Ferrites and their composites has been the subject of an extensive study because of their wide range of technical applications and their importance in understanding the theories of magnetism. They exhibit interesting microstructural, electrical and magnetic properties which depend upon various parameters like method of preparation, sintrations temperature, preparation time, amount of substitution, types and amount of surfactant etc.

In recent years, chemical co-precipitation technique is used to prepare nano ferrites. Many literatures confirmed that co-precipitation technique is a versatile low-cost technique to obtain ultrafine, homogeneous ferrite particles; while micro emulsion and the self-propagating high temperature synthesis techniques are used to prepare nanosized ferrite particles[11] Wet chemical methods are advantageous over the physical methods due to low cost, low reaction temperatures and large scale production possibility[11] .Large scale applications of ferrites with nanoparticles and the tailoring of electrical properties have prompted the development of several widely used methods, including chemical reactions, sol–gel techniques [12], reverse micelles [13], host template [14], co-precipitation [15], micro emulsion procedures

[16], precursor techniques [17], microwave composition [18], mechanical milling for the preparation of stoichiometric [19] and chemically pure ferrite nanoparticles [20]. In the case of spinel ferrites prepared by the co-precipitation method, high temperature is usually employed for the completion of solid-state reaction between the constituent oxides. In the view of the above facts, it is thought that a systematic comparative study would be very much useful and essential in emerging nanotechnology [21].

Within different ferrites, cobalt ferrite ($CoFe_2O_4$) is categorized into a hard magnet due to its high coercivity and moderate magnetization. According to literature, $CoFe_2O_4$ is an inverse spinel ferrite, taken to be collinear ferromagnetic that originating from magnetic moment of anti-parallel spins between Fe^{3+} ions at tetrahedral A sites and $Co^{2+}$and Fe^{3+} ions at octahedral B-sites. The general formula for spinel ferrite is represented by AB_2O_4 and it possesses 64 tetrahedral sites and 32 octahedral sites. Due to its high magnetic coercivity value and good physical and chemical stability, it has been used for various applications [22]. Its high value makes it a perfect candidate for using in high density magnetic storage materials, ferro fluids, medical diagnosis, magneto-mechanical, and torque sensors. However, there are so many limitations by using cobalt ferrite in many applications that need to low magnetic storage [23].

Copper ferrite ($CuFe_2O_4$) is one of the important spinel ferrites because it exhibits phase transitions, changes semiconducting properties, shows electrical switching and tetragonality variation when treated under different conditions in addition to interesting magnetic and electrical properties with chemical and thermal stabilities. It is used in wide range of applications such as in gas sensing, catalytic applications, color imaging, bioprocessing, and magnetic refrigeration and ferro fluids. Although, $CuFe_2O_4$ assumes great significance because of its high electrical conductivity, high thermal stability, however, need to controlled the electrical properties of this spinel ferrite for different applications [24].

Cobalt-Copper (Co-Cu) ferrite nano crystalline magnetic materials are extensively investigated for their interesting deviations in magnetic, optical, electrical and thermal properties compared to that of their bulk counter parts. Among the spinel ferrites, Co-Cu ferrite is a hard magnetic material that has high coercivity, moderate saturation magnetization, high cubic magneto crystalline anisotropy, excellent chemical stability and mechanical hardness. The structural and magnetic properties of nano crystalline copper ferrites are also quite interesting. The introduction of Cu^{2+} ions in the structure of $CoFe_2O_4$ is expected to modify its structural and magnetic properties, making it suitable candidature as sensors, catalyst and treatment for industrial effluents. Spinel ferrites are suitable for many uses, and with control size and electrical properties, Co-Cu ferrite can make it usable in an industrial application and chemistry as catalyst application [25].

Recently, research shows that substitution of Al^{3+} ions have some beneficial effects on power handling capability [26]. Addition of Al^{3+} ions changes structural properties, switching characteristics in electrical properties, magnetic properties, dielectric properties and elastic properties of spinel ferrite materials in an interesting manner. Aluminum substituted ferrite is a soft ferrite having low magnetic coercivity and high dc resistivity. The high electrical resistivity and good magnetic properties makes ferrite an excellent choice for power transformer in electronic, telecommunication application and chemistry as catalyst or catalyst supports [27].

1.2 Problem statement

Ferrites are the most important materials for high frequency applications because of their large resistivity, low conductive losses and reasonably high permeability. Among those ferrites, $CuFe_2O_4$ and $CoFe_2O_4$ have been most extensively studied systems, because they exhibit the typically normal and inverse spinel ferrites respectively. Chemical co-precipitation method has advantages over other methods due to its processing simplicity, low cost, good control of size, and the efficiency of more homogeneous mixing of the component materials that lead to the formation of nanocrystallites.

Aluminum has recently been shown to have an interesting effect on the electric, magnetic properties and particle size of ferrite [28]. Aluminum substituted Co-Cu ferrites due to their high electrical resistivity, low eddy current losses, square nature of hysteresis loops, high stability and high value of saturation magnetization are promising candidates for vast technological application over wide range of frequency [29, 30]. Recently, the diamagnetic substitution in mixed ferrites has received special attention. The role-played by the substituents in modifying the physical properties of basic ferrites and the mechanism behind enhanced magnetic response are not widely studied. Fabrication of ferrite materials of high quality, low cost and low loss at high frequency for power applications is ever demanding.

Thus in this study, nanocomposition for ferrimagnetic material was varied to change the magnetic properties such as magnetization and resistivity $Co_{1-x}Cu_xFe_{2-x}Al_xO_4$ ferrite provides high resistivity and magnetostriction coefficient which favors the magnetic effect, and is suitable as one of the material used for the recording media application. Hence $Co_{1-x}Cu_xFe_{2-x}Al_xO_4$ with $x = 0.0, 0.2, 0.4, 0.6, 0.8$, is suitable for the media application.

The study of solubility of Cu-Al cobalt ferrite is interesting not only from the academic point of view related to its influence on electromagnetic properties but also for their technological applications considering the vast potential of applicability in the microwave region and recording media application. Moreover, since the independent substitution of these ions for Co/Fe brings improvement in some of the magnetic and dielectric properties, the co-substitutions of Cu-Al in cobalt ferrite would be expected to throw more light in determining compositions for low dielectric losses.

1.3 Objectives of research

This study presents the following objectives:

I. To synthesize nanoparticles of copper aluminum substituted cobalt ferrite by using chemical co-precipitation technique.
II. To determine the effect of sintration temperature and concentration on grain size and structural of copper aluminum substituted cobalt ferrite nanoparticles.
III. To determine the effect of sintration temperature and concentration on morphological and chemical content properties of copper aluminum substituted cobalt ferrite nanoparticles.
IV. To determine the effect of sintration temperature and concentration on magnetic properties (M_s, H_c, M_r) of copper aluminum substituted cobalt ferrite nanoparticles.

1.4 Scope of research

Present work consists of the synthesis, characterization and investigation of structural, magnetic and electric properties of nanophase $Co_{1-x}Cu_xFe_{2-x}Al_xO_4$ with ($0.0 \leq x \leq 0.8$). This material is selected for this study in view of their technological importance.

Copper aluminum substituted on cobalt ferrite $Co_{1-x}Cu_xFe_{2-x}Al_xO_4$areprepared by co-precipitation method by using cobalt acetate, copper, aluminum and iron nitrates. The crystallinity of the powder is developed by annealing at 600 °C, 700 °C, 800 °C and 900 °C.

Crystalline phase is investigated by using X-ray Diffractometer (XRD). The average crystallite size for nanoparticles is determined by Scherrer's formula. Fourier Transform Infrared Spectroscopy (FT-IR) is one of the preferred methods for infrared spectroscopy to identify the chemical and structural changes occurring in particular sample. The morphology of the annealed sample is investigated by field emission scanning electron microscopy (FE-SEM). Energy dispersive X-ray spectrometer (EDX) is used for elemental analysis of the sample. To understand the magnetic properties of $Co_{1-x}Cu_xFe_{2-x}Al_xO_4$ferrite samples, the field dependence magnetization of all the samples is measured using vibrating sample magnetometer (VSM). Chemically synthesized samples are used to prepare nanoparticles and subjected at a different sintering temperature in order to study the effect of sintering on various properties of nanoparticles.

1.5 Significant of study

These materials have special applications in the field of high frequency devices as they possess more electrical-resistivity, lesser magnetic coercivity, and small eddy current loss. The morphological stability of the ferrites is significant in each case. The magnetic nature of the spinel ferrite is understood in depth with the information of cations displacement and spin alignment. The cation displacement within A and B position is important for studying chemical as well as physical characteristics of these ferrite. The variation in magnetic structure, ferromagnetic order, and spin alignment depends on distribution of non-magnetic atoms in the ferrites as reported by others. Co has important magnetic properties whereas adding Cu lowers the firing temperature and enhances the density as reported earlier. Aluminum substitution shows influences on the product ultrafine structure and behavior. Due to these potential applications, interest in the above mentioned ferrites has been developed and work carried out in this field.

Present research gives a better understanding into synthesis of nanomaterials in particulate form by making use of hydroxide. Co-precipitation method is almost preferred to be able to obtain high quality product. By reliably controlling the size, morphology, composition, and crystallinity of these nanostructures, their properties can be tuned for a specific purpose. The copper-aluminum doped cobalt ferrite finds a wide range of applications in recording media and microwave, where controllable size, and minimum electrical and magnetic losses are required [27].A low-coercivity, high-remanence, soft magnetic material, having a square hysteresis loop, is required for recording media and sensing applications. Magnetic nanoparticles are being used or have the potential use as a catalyst, with controlled size of ferrite nanoparticle make it suitable for using as catalyst or catalyst support. Catalyst support is the material, usually a solid with a high surface area, to which a catalyst is affixed [31].

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