UNIVERSITI PUTRA MALAYSIA

EFFECT OF SINTERING TEMPERATURE ON DIELECTRIC PROPERTIES, AND MICROSTRUCTURE OF Ni(0.3)Zn(0.7)Fe2O4

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By

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A series of ferrite material with $Ni_{(0.3)}Zn_{(0.7)}Fe_2O_4$ composition have been prepared by the solid state reaction method and sintered at a temperature range of 673 to 1623 K with an interval of 100 K. A total of ten samples was synthesized for this research work. After the samples were sintered, the surface morphology, structure of each sample, density, dielectric and conductivity measurement were made first and then the samples were re-sintered at an increase temperature of 50 K so that the sintered temperatures were 723, 823, 923, 1023, 1123, 1223, 1323, 1423, 1523 and 1623 K. The surface morphology, structure of each sample, density, dielectric and conductivity measurement were made once again after next sintering. Samples were confirmed mixture of crystal phase when sintered under 1173 K and single crystals were formed when sintered above 1173 K via X-ray diffraction measurement. FESEM diagram showed that grain size increased slowly when sintered below 1073 K from 32 to 253 nm and increase drastically when sintered above 1073 K from 1880 to 7590 nm. EDX technique or EDS has been used to determine oxide constituent composition of samples. There was no noticeable change in density for samples sintered below 1173 K and density increased drastically when samples sintered more than 1173 K. Relative dielectric permittivity and loss factor had been measured in frequency ranges from 0.01 to 3 MHz and in the temperature range 303 to 473 K. Experimental data are simulated to a model proposed by Havriliak-Negami (HN) which consists of a combination of several HN functions and a real capacitor and conductance. Relative dielectric permittivity for all samples decrease in the range from 1.5×10^6 to 5 with increased frequency from 10^{-2} to 3 MHz and decrease with increase of 1000/T until the temperatures range from 363 to 403 K and then increase with increase of 1000/T for all fixed frequencies. These changes are due to dielectric relaxation and electron hopping interaction. At low frequency (below 100 Hz) relative dielectric permittivity is dominated by surface charge polarization and the values are in between 10^4 to 1.5×10^6. Dipole occurs in frequency ranges from 100 to 100 kHz with value in between 50 to 150 while electronic polarization occurs above 100 kHz with values in between 5 to 10. Generally relative dielectric permittivity decreases with increasing of frequency and the same trend occurs as sintered temperature increase. This phenomenon can be explained based on the changes microstructures of the samples (resistance of grain and grain boundary). The variation of conductivity versus frequency consists of three parts which are at high frequency, the conductivity depends on the power law, at the middle variation depends on polarization and at low frequency, the conductivity is independent with frequency. Apart from that conductivity at low frequency also increases when sample temperature increase and the value of conductivities of all sample sintering in between 10^{-8} to 10^{-4} S/m. Conductivity phenomenon which occurred in this sample is due to continuous hopping of charge carrier. Impedance spectroscopy has been used in this study to further analyse the conductance of the sample, especially to determine the activation energy. The values of conductance of grains and grain boundary were determined to be in the range of 0.01 to 10 mho and 10^{-9} to 10^{-5} mho respectively. DC activation energy is obtained from the variation of resistivity at low frequency in temperatures range between 303 to 473 K. The values of activation energy in the intrinsic part determined from the high temperature slope region of all sintering temperatures are in between 0.09 to 0.62 eV and the value of extrinsic part determined from the low temperature slope region are in between 0.11 to 0.48 eV. The conductivities decrease when 1/T increases in both samples phase at high temperature region, meanwhile the conductivities increase with no special trends when 1/T increases in the low temperature region of the mixture of crystal phase sample and the conductivities decreases linearly when 1/T increases in the low temperature region at single crystal phase samples except sample with a sintering temperature of 1623 K. The variations of conductivities versus 1/T increases when frequency of external electric field increases. The charges moving in an AC conductivity of the sample are dominant by hopping meanwhile the charges moving in the DC conductivity of the sample is dominated by the free electron. The conductance of these samples have been studied in range 1 nS to 10 µS. At the same time, AC activation energy at various fixed frequencies was determined and the value is in the range of 0.14 to 0.86 eV for sample sintering temperatures from 673 to 1123 K and 0.11 to 0.75 eV for sample sintering temperatures from 1173 to 1623 K. The trend of AC activation energies for all samples are in the decreasing trend when the fixed frequency of external electric field is increased.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia
sebagai memenuhi keperluan untuk ijazah Doktor Falsafah

KESAN SUHU SINTER TERHADAP SIFAT DIELEKTRIK DAN MIKROSTRUKTUR $Ni_{(0.3)}Zn_{(0.7)}Fe_2O_4$

Oleh

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Satu siri bahan ferit dengan komposisi $Ni_{(0.3)}Zn_{(0.7)}Fe_2O_4$ yang disediakan menggunakan kaedah tindakbalas bahan pepejal secara pembakaran seramik pada julat suhu 673 hingga 1623 K dengan sela 100 K. Sejumlah sepuluh sampel telah disintesis dalam kajian ini. Setelah sampel disinter, morfologi permukaan, struktur setiap sampel, ketumpatan, pengukuran dielektrik dan kekonduksian sampel-sampel dibuat terlebih dahulu. Setelah pengukuran selesai, setiap sampel dibakar semula dengan kenaikkan suhu sebanyak 50 K, oleh yang demikian suhu pembakaran sampel adalah 723, 823, 923, 1023, 1123, 1223, 1323, 1423, 1523 dan 1623 K. Morfologi permukaan, struktur setiap sampel, ketumpatan, pengukuran dielektrik dan kekonduksian sampel-sampel dibuat sekali lagi selepas pembakaran semula. Sampel-sampel tersebut telah disahkan sebagai struktur campuran kristal apabila suhu pembakaran di bawah 1173 K dan sampel bersifat kristal tunggal apabila suhu pembakaran melebihi 1173 K yang disahkan melalui pengukuran pembelauan sinar-x. Gambarajah FESEM telah menunjukkan bahawa saiz butiran telah meningkat secara perlahan-lahan apabila sampel dibakar pada suhu di bawah 1073 K dari 32 ke 253 nm dan meningkat secara drastik apabila suhu sinter diatas 1073 K dari 1880 ke 7590 nm. Teknik EDX atau EDS juga telah digunakan dalam menentukan komposisi juzuk-juzuk oksida yang terkandung di dalam sampel. Apabila sampel dibakar pada suhu kurang daripada 1173 K, di dapati tiada perubahan ketara di dalam ketumpatan sampel dan ketumpatannya meningkat secara drastik apabila dibakar pada suhu melebihi 1173 K. Ketelusan dielektrik relatif dan faktor kehilangan telah diukur dalam julat frekuensi dari 0.01 hingga 3 MHz dalam julat suhu 303 hingga 473 K. Data dari ujikaji adalah berpadanan dengan model yang telah disyorkan oleh Havriliak-Negami (HN), di mana terdiri daripada kombinasi beberapa fungsi HN dan satu kapasitor nyata dan satu konduktans. Ketelusan dielektrik relatif semua sampel yang dikaji menurun dalam julat dari 1.5×10^6 ke 5 dengan kenaikkan frekuensi dari 10^{-2} ke 3 MHz dan ianya berkurang dengan peningkatan 1000/T sehingga julat suhu dari 363 ke 403 K dan meningkat dengan peningkatan 1000/T untuk setiap frekuensi tetap. Perubahan ini disebabkan oleh santaian dielektrik dan interaksi loncatan elektron. Pada frekuensi yang rendah (dibawah 100 Hz), ketelusan dielektrik relatif didominasi oleh pengutuban cas ruang dan bernilai di antara 10^4 ke 1.5×10^6. Pengutuban dwikutub dan elektronik muncul di dalam julat frekuensi 100 ke 100 kHz dengan nilai-nilai di antara 50 ke 150 sementara pengutuban elektronik muncul di atas 100 kHz dengan nilai-nilai di antara 5 ke 10. Secara umumnya, ketelusan dielektrik relatif menurun dengan kenaikkan frekuensi dan ianya mengikut corak yang sama apabila suhu sinter meningkat. Fenomena ini boleh dijelaskan berdasarkan perubahan mikrostruktur sampel (rintangan butiran dan sempadan butiran). Penyerakan kekonduksian terhadap frekuensi mengandungi tiga bahagian iaitu pada frekuensi tinggi, kekonduksian bergantung kepada hukum kuasa, pada frekuensi pertengahan ianya bergantung kepada pengutuban bahan dan pada frekuensi rendah kekonduksian tidak bergantung kepada frekuensi. Selain itu kekonduksian pada frekuensi rendah meningkat apabila suhu sampel meningkat dan nilai konduktiviti semua sampel yang telah disinter di antara 10^{-8} ke 10^{-4} S/m. Fenomena konduktiviti muncul di dalam sampel adalah disebabkan oleh loncatan pembawa cas berterusan. Spektroskopi impedans telah digunakan di dalam kajian ini untuk analisis lanjutan berkenaan konduktan sampel khususnya untuk menentukan tenaga pengaktifan. Nilai-nilai konduktan butiran dan sempadan butiran telah ditentukan bernilai di antara 0.01 ke 10 mho dan 10^{-9} ke 10^{-5} mho masing-masing. Tenaga pengaktifan DC diperolehi daripada perubahan kerintangan pada frekuensi rendah di dalam suhu di antara 303 ke 473 K. Nilai-nilai tenaga pengaktifan dalam bahagian intrinsik ditentukan daripada bahagian cerun suhu tinggi untuk setiap suhu sinter adalah di antara 0.09 ke 0.62 eV dan nilai untuk bahagian ekstrinsik ditentukan daripada bahagian cerun suhu rendah adalah di antara 0.11 ke 0.48 eV. Konduktiviti berkurang apabila 1/T meningkat dalam kedua-dua fasa sampel pada bahagian suhu tinggi, sementara itu konduktiviti meningkat tanpa bentuk khusus apabila 1/T meningkat dalam julat suhu rendah untuk sampel fasa kristal campuran dan konduktiviti berkurang secara linear apabila 1/T meningkat dalam julat suhu rendah untuk sampel fasa kristal tunggal kecuali sampel yang disinter pada suhu 1623 K. Perubahan konduktiviti terhadap 1/T meningkat apabila frekuensi medan elektrik luar meningkat. Gerakan cas dalam konduktiviti AC sampel adalah dominan secara loncatan sementara gerakan cas dalam konduktiviti DC sampel adalah dominan oleh electron bebas. Konduktan untuk sampel yang telah dikaji adalah dalam julat 1 n ke 10 µS. Dalam masa yang sama, tenaga pengaktifan AC untuk frekuensi tetap yang dikaji juga telah ditentukan dan bernilai dalam julat 0.14 ke 0.86 eV untuk sampel yang disinter dari 673 ke 1123 K dan 0.11 ke 0.75 eV untuk sampel yang disinter dari 1173 ke 1623 K. Corak tenaga pengaktifan AC untuk semua sampel adalah menurun apabila frekuensi tetap medan elektrik luar meningkat.

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I certify that a Thesis Examination Committee has met on 9 March 2017 to conduct the final examination of Mohd Noor Mat on his thesis entitled 'Effect of Sintering Temperature on Dielectric Properties, and Microstructure of $Ni_{(0.3)}Zn_{(0.7)}Fe_2O_4$' in accordance with Universities and University Colleges Act 1971 and the University Putra Malaysia [P.U.(A) 106] 15 March 1998. The Committee recommends that the candidate be awarded the Doctor of Philosophy

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τ	Time constant
χ	Dielectric susceptibility
τ_p	Relaxation time
τ_{cc}	Cole and Cole time constant
τ_{DC}	Davidson-Cole time constant
τ_{HN}	Havriliak-Negami time constant
μ	mobility
$\tan\delta$	Loss tangent
A	Area
AC	Alternating current
C	capacitance
C_o	Vacuum capacitance
Cu^{2+}	Copper ion
d	Thickness
DC	Direct current
DVD	Digital Video Disk
E	Electric field
E_a	Activation energy
EDS	Energy dispersive x-ray spectroscopy
EDX	Energy dispersive x-ray spectroscopy
EDXA	Energy dispersive x-ray analysis
EDXMA	Energy dispersive x-ray microanalysis
EM	electromagnetic
EMI	Electromagnetic Interference
ERP	Electron paramagnetic resonance
ESR	Electron spin resonance
f	frequency
FCC	Face center cubic
Fe^{2+}/ Fe^{3+}	Iron ion
Fe_2O_3	Iron oxide
FESEM	Field emission scanning electron microscopy
FMR	Ferromagnetic Resonance
G	conductance
GHz	Giga hertz
GMI	Giant magneto impedance
HCP	Hexagonal center cubic
H_K	Anisotropy field
j	Complex number
k_B	Boltzmann constant
LFA	Low field microwave absorption
M	Molecules weight
MHz	Mega hertz
Mn^{2+}	Manganase ion

MRI	Magnetic resonance imaging
N	Avogadro’s Number
n(T)	Density of state
NZF	Nickel zinc ferrite
O^{2-}	Oxygen ion
P	polarization
P_p	Percentage of porosity
PVA	Poly vinyl alcohol
Q	charge
SEM	Scanning electron microscopy
SMPSs	Switch-mode power supplies
T	Temperature
v_{ph}	Phonons velocity
W_m	The number of valence alternation pairs
XRD	x-ray diffraction
Z^*	Complex impedance
Z'	Real impedance
Z''	Imaginary impedance
Zn	zinc
ZnO	Zinc oxide
α_e	Electronic polarization
$\Delta\varepsilon$	Dielectric field strength
ρ	Resistivity
ρ_D / ρ_s	Density of sample
σ'	Real conductivity
σ^*	Complex conductivity
σ''	Imaginary conductivity
σ_o	Conductivity at $\omega = 0$ Hz
ω	Angle frequency
ω_p	Peak frequency
ε^*	Complex permittivity
ε'	Real permittivity
ε''	Imaginary permittivity
ε_∞	Relative dielectric permittivity at high frequency
ε_o	Free space permittivity
ε_r	Relative dielectric permittivity / dielectric constant
ε_s	Relative dielectric permittivity at low frequency
HN	Function by Havriliak and Negami
NZF	Nickkel Zinc Ferrite

CHAPTER I

INTRODUCTION

1.1 Background Study

Evolution is a change process of forming samples from one stage to next stages (Dias, 2000). The evaluation process can be achieved by using a sintering process. Sintering is a heat treatment process that alters the physical and chemical properties of a material to increase its ductility and reduce its hardness, making it more workable. Sintering is commonly defined as a process which is thermally activated, in which compacted powder density and develop a microstructure with useful properties (Harmer et al., 1979). It involves heating a material above its recrystallization temperature, maintaining a suitable temperature, and then cooling. In sintering, atoms migrate in the crystal lattice and the number of dislocations decreases, leading to the change in ductility and hardness. In the case of ferrite material, this process is performed by heating the material for a while and then slowly letting it cool to room temperature in still air.

Ceramics are classified as inorganic and non-metallic materials that are essential in multipurpose. Ceramic and material engineers are the people who design the processes in which these products can be made, create new types of ceramic products, and find different uses for ceramic products. This category of materials includes things like tiles, bricks, plates and glass. Ceramics can be found in products like watches (quartz tuning forks-the time keeping devices in watches), snow skies (piezoelectric ceramics that stress when a voltage is applied to them), automobiles (spark plugs and ceramic engine parts found in race cars), and phone lines. They can also be found on space shuttles, appliances (enamel coatings), and airplanes (nose cones). Depending on their method of fabrication, ceramics can be dense or lightweight. Typically, they will demonstrate excellent strength and hardness properties; however, they are often brittle in nature. Ceramics can also be formed to serve as electrically conductive materials, allowing electricity to pass through their mass, or as insulator materials preventing the flow of electricity. Some ceramics, like superconductors, also display magnetic properties.

Ceramics are typically produced by the application of heat upon processing clays and other natural raw materials to form a rigid product. Ceramic products that use naturally occurring rocks and minerals as a starting material must undergo special processing in order to control, purity, particle size, particle size distribution, and heterogeneity. These attributes play a big role in the final properties of the finished ceramic. Chemically prepared powders are also used as starting materials for some ceramic products. These synthetic materials can be controlled to produce powders with precise chemical compositions and particle size.

The next step is to form the ceramic particles into the desired shape. This is accomplished by the addition of water and/or additives such as binders, followed by a shape forming process. Some of the most common forming methods for ceramics include extrusion, slip casting, pressing, tape casting and injection moulding. After the particles are formed, these "green" ceramics undergo a heat-treatment (called firing or sintering) to produce a rigid, finished product. Some ceramic products such as electrical insulators, dinnerware and tile may then undergo a glazing process. Some ceramics for advanced applications may undergo a machining and/or polishing step in order to meet specific engineering design criteria.

The properties of ceramic materials, like all materials, are dictated by the types of atoms present, the types of bonding between the atoms, and the way the atoms are bound together. This is known as the atomic scale structure. Most ceramics are made up of two or more elements. This is called a compound. For example, alumina (Al_2O_3) is a compound made up of aluminium atoms and oxygen atoms.

The atoms in ceramic materials are held together by a chemical bond. The two most common chemical bonds for ceramic materials are covalent and ionic. For metals, the chemical bond is called the metallic bond. The bonding of atoms together is much stronger in covalent and ionic bonding than in metallic. That is why, generally speaking, metals are ductile and ceramics are brittle. Due to ceramic materials having a wide range of properties, they are used in a various applications. In general, most ceramics are hard, wear-resistant, brittle, refractory, thermal insulators, electrical insulators, nonmagnetic, oxidation resistant, prone to thermal shock and chemically stable.

1.2 History of Ferrites

The most common soft ceramic magnets are ferrites with the general chemical formula of spinel ferrites $[M.Fe_2O_4]_8$, where the indicated iron is Fe^{3+} a trivalent iron ion, M denotes a divalent metal ion, may be Fe^{2+}, Ni^{2+}, CO^{2+}, Mn^{2+}, Zn^{2+} and so on. Each ferrous ion has lost two electrons. These are the two 4s electrons, the six 3d electrons remain to give four unpaired electrons and a ferric ion has lost the two 4s electrons and one 3d electron (Lawrence, 1989).

Ferrites may be defined as magnetic materials composed of oxides containing ferric ions as the main constituent (Valenzuela, 2005). The term is often restricted to such materials having the cubic crystal structure of the mineral spinel, but it is also loosely applied to magnetic oxides in general irrespective of their crystal structure (Bragg, 1915). Magnetite or ferrous ferrite is an example of a naturally-occurring ferrite. It has been known since ancient time and its week permanent magnetism found application in the lodestone of the early navigators. Hilpert in 1909 published the first systematic study of the relation between the chemical and magnetic properties of a number of binary iron oxides but experienced difficulty in identifying the magnetic phases of his preparations. Around 1928 Forestier in France and Hilpert and Wille in Germany made quantitative investigations into the relation between the chemical composition, the saturation magnetization, and the Curie temperature. Magnetic oxides were also studied by Japanese workers between 1932 and 1935 (Snelling, 1988).

In 1936 Snoek was studying magnetic oxides in the Netherlands; similar studies in Japan were reported by Takei in 1939 (Snelling, 1988). Snoek and his co-worker realized that the most important property of a mineral intended as a core for an inductor is the loss tangent divided by the permeability, the so-called loss factor. By 1945, Snoek had laid the foundations of the physics and technology of practical ferrites and a new industry came research era. Since that time the use of ferrites has become established in many branches of telecommunication and electronic engineering and they now embrace a very wide diversity of composition, properties, and applications. A more detailed account of the history of ferrite research and development has been given by Wijn (Snelling, 1988; Goldman, 1990).

Ferrites are ceramic materials, dark grey or black in appearance, very hard and brittle. A ferrite core is made by pressing a mixture of powders containing the constituent raw materials to obtain the required shape and then converting it into a ceramic component by sintering. The magnetic properties arise from interactions between metallic ions occupying particular positions relative to the oxygen ions in the crystal structure of the oxide. In magnetite, in the first synthetic magnetic oxides and indeed in the majority of present-day magnetic soft ferrite, the crystal structure is cubic; it has the form of the mineral spinel.

The early practical ferrites are represented by the combination of elements cuprum-zinc-iron, manganese-zinc-iron or nickel-zinc-iron. The first of these compounds was soon discarded and the other two generally referred to as manganese zinc ferrite and nickel zinc ferrite respectively were developed for a wider range of applications where high permeability and low loss were the main requirements.

1.3 Application of Ferrites Materials

Ferrite material research has started since several years ago. Various electronic equipment using ferrite material has served many purposes in telecommunications, automotive, computer memory, electromagnet wave pollution suppressor, transformer, radar and so on (Valenzuela, 2012). Ferrites are primarily used as inductive components in a large variety of electronic circuits such as low noise amplifiers, filters, voltage-controlled oscillators, impedance matching networks, for instance.

In addition, to provide a high permeability at the operating frequency, the ferrite film should be prepared by a process compatible with the integrated circuit manufacturing process. Sputtering provides films with high density, but the composition is sometimes difficult to control with accuracy, and the annealing processes can attain high temperatures. Pulsed laser deposition leads to high-quality films; however, a method involving the preparation of the ferrite film by a combination of sol-gel and spin-coating seems easier and with a lower cost (Yang et al., 2006).

Layered samples of ferrites with piezoelectric oxides can lead to a new generation of magnetic sensors. The basis of their performance is the capability of converting magnetic fields into electrical voltages by a two-step process. First, the magnetic field produces a mechanical strain on the magnetic material (due to its magnetostriction); this strain then induces a voltage in the piezoelectric layer. These sensors can provide a high sensitivity, miniature size, and virtually zero power consumption. Sensors for AC and DC magnetic fields, AC and DC electric currents, can be fabricated. Sensors based on nickel ferrite ($Ni_{1-x}Zn_xFe_2O_4$ with x = 1 – 0.5)/lead zirconate-titanate ($PbZr_{0.52}Ti_{0.48}O_3$) have shown an excellent performance (Fetisov et al., 2006).

Transformers in electrical appliances such as computer, television, radio, are used to reduce the voltage from the main source to a voltage level that suits the appliances in use to prevent damage. It is used in many types of electronic instruments such as transformer core attenuation and motor electrical component. Although the core size is small but the output is particularly satisfying (Valenzuela, 2012). In a computer configuration, a wave suppressor electromagnet is used in wire extension from the main source to a computer in order to reduce interference in a computer system. This device is able to absorb and suppress undesirable wave and allow wave entailment only through it (Li et al., 2006).

In a computer system, material ferrite functions as a transducer and active ingredients to store information data. For example, the cartridge magnetic tape was a tool to keep data. It is made of thin ribbon polyester and coated with magnetic material on one surface. Cassette recorder connects with microcomputer through the standard circuit to change and duplicate audio signal. With magnetic tape driver the data that is kept can be manipulated again. While floppy disk magnet, drive zipper, hard disk and DVD (Digital Video Disk) also uses a similar technique, they have larger storage space and are easier to access.

To perform a satisfactory switch operation requires a wide flux swing in the magnetic material which must allow the transfer of energy between saturated and unsaturated states, producing a correspondingly fast rise time for a switch. Losses and rounding of transition into saturation are dependent on the domain walls in the material.

Requirements for reliable, high energy pulsed power systems for lasers and accelerators have rapidly increased. Amorphous magnetic alloys and Ni-Zn ferrite materials are used in pulse power transformers and in magnetic pulse compressor network in these systems. Magnetization rates to produce pulses on the order of 100 ns are required. The magnetization rates involved are up to about 5 T/µs. The magnetization behavior of magnetic materials at dc is entirely different from dynamic / pulsed conditions. Therefore, the dynamic studies of magnetic materials have been taken up to know how the ferrite behaves when its magnetization reverses in the order of 100 ns.

It is well known that ferrites were used as a magnetic recording heads because of their superior magnetic properties at high frequencies and their wear resistance. These properties are due to the high electrical resistivity, mechanical hardness and chemical stability of the ferrites.

In general, the following characteristics are desired for magnetic core materials: high permeability, high saturation magnetization, low coercive force and high electrical resistivity, hardness for wear stability, high Curie temperature, low porosity and reasonable cost.

The polycrystalline Ni-Zn ferrite is a suitable material for magnetic head (Resleszu and Resleszu, 1987) because it is mechanically strong and can be produced by a similar method to the conventional one, which is impossible for Mn-Zn ferrites. When a polycrystalline is used for magnetic heads, it is desirable for the ferrite to have a high density to enable it to form a mechanically strong magnetic gap.

Ferrite materials as a microwave absorbers are very useful in many applications like radar cross-section reduction, EMI/EMC problems, etc. Moreover, with the recent rapid increase in the use of portable telephones, cell phones, and other wireless devices, public concerns regarding potential health hazards due to the electromagnetic energy emitted by these devices have been growing (Pardavi, 2000).

Ferrites materials as an inductive components in a large variety of electronic circuits such as low noise amplifiers, filters, voltage-controlled oscillators and impedance matching networks, for instance. Their recent applications as inductors, among other tendencies, to the general trend of miniaturization and integration as ferrite multilayers for passive functional electronic devices. The multilayer technology has become a key technology for mass production of integrated devices; multilayers allow a high degree of integration density. Multilayer capacitors penetrated the market a few decades ago, while inductors started in the 1980s. The basic components to produce the inductances are a very soft ferrite and a metallic coil. In addition, to provide a high permeability at the operation frequency, the ferrite film should be prepared by a process compatible with the integrated circuit manufacturing process. Sputtering provides films with high density, but the composition is sometimes difficult to control with accuracy, and the annealing processes can attain high temperatures.

Both ferrite and zirconate-titanate films are prepared by tape casting; typically, 11 ferrite layers were combined with 10 piezoelectric layers (both High Frequency). There has been an increasing demand for magnetic materials for high-frequency applications such as telecommunications and radar systems, as microwave technology requires higher frequencies and bandwidths up to 100 GHz (Fetisov et al.,2006).

Ferrites are nonconducting oxides and therefore allow total penetration of electromagnetic fields, in contrast with metals, where the skin effect severely limits the penetration of high-frequency fields. At such frequencies, domain walls are unable to follow the fields (dispersion of domain walls typically occurs about 10GHz), and absorption of microwave power takes place by spin dynamics. The usual geometry is to align spins first with a DC magnetic field, H, and apply the microwave field perpendicular to H. The spins process around their equilibrium orientation at the frequency of the microwave field. Ferromagnetic resonance (FMR) is associated with the uniform (in phase) precession of spins. The upper limit of applications of ferrites is FMR since the interaction with the microwave field becomes negligible. Spinels are therefore applied at frequencies up to 30 GHz, while this limit is about 10 GHz for garnets and can attain 100 GHz for hexaferrite. The absorption of microwaves by ferrites involves losses (Pardavi, 2000).

In polycrystalline ferrites, losses are associated with defects and the anisotropy field distribution, and with electrical conduction, a common problem, especially in spinel's, is the presence of Fe^{2+}, which promotes a hopping conduction process in combination with Fe^{3+}. The physical origin of losses in polycrystalline ferrites, through its effects on the line width, has been recently investigated in detail; the dominant role of grain boundaries is apparent.

Some of the ferrite applications rely on the fact that the spin rotation depends on the orientation of the external field, which allows the control of the interaction with the microwave field. Typical devices are circulators, isolators, phase shifters, and antennas. Circulators were developed for radar systems and are now used in mobile phones. They allow the use of the same device for transmission and reception of the response signal.

Circulators are usually fabricated with garnets. Recently, a nonresonant absorption of microwave power at very low magnetic fields has been receiving attention. This absorption, known as LFA (for low-field microwave Absorption), has shown to be clearly dominated by the anisotropy field, H_K , of the ferro- or ferrimagnetic material. Recent results obtained in a Ni-Zn ferrite in many aspects (Valenzuela et al., 2007); this absorption is similar to giant magneto impedance, GMI (Monteil et al., 2005). A significant difference with GMI is that LFA does not need that the electrical conductivity of the sample is of the metallic type. No practical applications of LFA have been proposed, so far; however, it is possible to expect that similar applications to GMI can be made (sensors for the magnetic field, DC electric current, mechanical stress, etc.), with the additional advantages over classic GMI of high frequencies and insulator magnetic materials (Knobel et al., 2003).

A recent approach to increase efficiency of the ferrite cores is based on the decrease of eddy currents, by increasing resistivity. Besides the use of nonconducting additives that locate preferentially on grain boundaries (and limit the intergrain conductivity), MnZn and NiZn are combined as $Mn_xNi_{0.5-x}Zn_{0.5}Fe_2O_4$ and obtained through a citrate precursor method (Verma et al., 2006).

An additional difficulty appears in the case of power applications at high temperature, as is the case of some automotive power devices. Due to the closeness to the car engine, the working temperature increases from the usual 80–100°C for standard applications, to 140°C. A proposed solution involves the modification of the MnZn ferrites (used previously for these applications) in order to produce a higher fraction of Fe^{2+} (Zaspalis et al., 2004), such as $(Mn_{0.76}Zn_{0.17}Fe^{2+}{}_{0.07})Fe_2O_4$. This ferrous concentration presents a minimum in the magnetocrystalline anisotropy close to 140°C, and therefore, a minimum in losses appears at this temperature. The change is obtained through a careful control of the oxidation atmosphere during sintering and cooling.

As in all other applications, a strong need for miniaturization has also marked the developments in this area in the last few years. In addition to standard methods to obtain ferrite thin films (such as sputtering, laser ablation, sol-gel), screen or stencil printed ceramic-polymer composites have been investigated (Brandon et al., 2003), combining the remarkable magnetic properties of ferrites with the processing ability of polymer thick films. These polymers thick films can be cured at temperatures of 200°C or less, leaving only the polymer binder and the ferrite filler. This technique allows the fabrication of highly integrated power circuits. The coil is obtained by patterning copper on a flexible Polyimide substrate; the ferrite-polymer composite film is then printed above and below the plane of the coil. The magnetic ceramic filler is formed by MnZn ferrite particles about 10 μm, obtained by a standard method. The inductance value remains stable up to 124 MHz. A different method based on a batch-fabrication method of 3D transformers and inductors has also been proposed (Amalou et al., 2001).

The magnetic core is made of two half magnetic pieces and a printed circuit board (or flexi foil) carrying the electric windings around the core. The 3D ferrite cores are microstructures out of a 1mm thick ferrite wafer using a newly developed batch-type micro powder blasting process (Belloy et al., 2000). These transformers are well suited for low-power applications at working frequencies up to 1 MHz. Losses in ferrites depend essentially on hysteresis loss at low frequencies, conductivity (or eddy current) loss, and relaxation-resonance loss at high frequencies; their modelling is complex. A model based on the Preisach theory (Preisach, 1935) has been applied to predict the hysteretic behavior of soft ferrites for applications in power electronic (Angeli et al., 2000), with good results at low frequencies.

The significant increase in the amount of electronic equipment such as high-speed digital interfaces in notebooks and computers, digital cameras, scanners, and so forth, in small areas, have seriously enhanced the possibility of disturbing each other by electromagnetic interference (EMI). In particular, the fast development of wireless communications has led to interference induced by electric and magnetic fields. Electromagnetic interference can be defined as the degradation in performance of an electronic system caused by an electromagnetic disturbance (Stojanovic et al., 2006). The noise from electric devices is usually produced at frequencies higher than circuit signals. To avoid, or at least reduce EMI, suppressors should work as low-pass filters, that is, circuits that block signals with frequencies higher than a given frequency value. There are several approaches to build EMI suppressors: soft ferrites (Li et al., 2006), ferromagnetic metals (Feng et al., 2006), ferromagnetic metal / hexaferrite composites (Li et al., 2006), encapsulated magnetic particles (Che et al., 2006), and carbon nanotube composites (Xiang et al., 2005).

Ferrite components for EMI suppressors have been used for decades. In the recent years, however, there have been special needs of these materials as a consequence of the miniaturization trends, increase in integration density, and increase in higher clock frequency, especially in communication, computing, and information technologies. Ferrite multilayer components have been developed as a response to these needs, formed essentially by a highly conductive layer embedded in a ferrite monolithic structure, produced by ceramic compressing technologies. Typically, Ni-Zn ferrites are used for the 20–200 MHz frequency range. Multilayer suppressors behave like a frequency-dependent resistor; at low frequencies, losses in the ferrite are negligible.

Hexaferrites represent an interesting alternative to cubic ferrites as EMI suppressor components; they possess higher resonance frequencies, relatively high permeability (at microwave frequencies), and high electrical resistivity. Metallic ferromagnetism, in contrast, shows a larger saturation magnetization, but, as frequency increases, they exhibit a strong decrease in permeability due to eddy currents. However, in combination with hexaferrite, they have shown a strong potential for EMI suppressor devices (Li et al., 2006). Co_2Z and Zn_2Y hexaferrite particles (10–30 μm), mixed with metallic Ni particles (2-3 μm), and prepared with a polymer (polyvinylidene fluoride) by hot pressing at low temperature led to high shield effectiveness.

Carbon nanotube-polystyrene foam composites have shown a high EMI shielding effectiveness based on a mechanism associated with the reflection of electromagnetic radiation (Yang et al., 2005); no magnetic material is used. Ferrite nanoparticles in combination with carbon nanotubes can efficiently absorb microwave radiation. Carbon nanotubes/$CoFe_2O_4$ spinel nanocomposite was fabricated by a chemical vapor deposition method using $CoFe_2O_4$ nanoparticles as catalysts (Che et al., 2006). The microwave absorption (2–18 GHz range) was enhanced. For carbon nanotubes, the dielectric loss contributes to the energy loss of electromagnetic waves, while, for the pure Co ferrite, the effects of microwave absorption are associated with magnetic losses, but both isolated mechanisms are poor absorbers. For the nanocomposites, however, the microwave absorption improves because of a better match between the dielectric loss and the magnetic loss, which originates from the combination of paramagnetic nanotubes and ferromagnetic material. The dispersed $CoFe_2O_4$ nanoparticles absorb the microwave signals by resonance effects due to shape anisotropy and dipolar interactions between particles. Such effects are weakened in congregated particles.

Magnetic materials in the form of magnetite (Fe_3O_4), are present in various living organisms and can be used in a number of applications (Lowenstam, 1962). Magnetic nanoparticles can, of course, be prepared in the laboratory by means of the well-known methods; however, magnetic biogenic particles have better properties than synthetic ones: they have a definite size range and width/length ratio and high chemical purity, they are almost perfect crystallographically, and sometimes they possess unusual crystallographic morphologies. Extracellular production of nanometer magnetite particles of various types of bacteria has been described (Zhang et al., 1998). In many cases, the biogenic particles retain a lipid layer which makes them very stable and easily biocompatible. Many biotechnological applications have been developed based on biogenic and synthetic magnetic micro- and nanoparticles (Fu et al., 2005).

Magnetic nanoparticles have been used to guide radionuclides to specific tissues. An approach has been developed to directly label a radioisotope with ferrite particles in vivo liver tissue in rats. Therapeutic applications are feasible by further conjugation with other medicals. In magnetic resonance imaging (MRI), the magnetite superparamagnetic particles are selectively associated with healthy regions of some tissues (liver, for instance); since these particles change the rate of proton decay from the excited to the ground state (which is the basis of MRI), a different, darker contrast is obtained from these healthy regions of tissue.

Thermal energy from hysteresis loss of ferrites can be used in hyperthermia, that is, the heating of specific tissues or organs for the treatment of cancer. The temperature in tumor tissues rises and becomes more sensitive to radio or chemotherapy. In addition, of magnetite, several spinel ferrites (M–Zn, with M = Mn, Co, Fe^{2+}, and Fe^{2+}–Mn) are under investigation as well as hexaferrite. Enzymes, oligonucleotides, antibodies, and other biologically active compounds can be immobilized, as an important technique used in biotechnology. Such immobilized compounds can be targeted to a specific place or can be removed from the system by using an external magnetic field.

The compounds can exert their activity on the specific place or a tissue or can be used as affinity ligands to trap the cells or target molecules. Magnetic nanoparticles can also be used in a variety of applications: modification, detection, isolation, and study of cells, and isolation of biologically active compounds.

The review of the application of ferrites materials are listed in this part as in Table 1.1. These table consists three part such as composition, preparation and properties of ferrites materials and family.

Table 1.1: The review of the literature as application of ferrites materials

No	composition	preparation	properties
1	Ferrite film	Combination of sol-gel and spin-coating	Inductive component such as low noise amplifiers, filters, voltage-controlled oscillators and impedance matching networks
2	$Ni_{1-x}Zn_xFe_2O_4$ with x = 1 – 0.5 / lead zirconate-titanate ($PbZr_{0.52}Ti_{0.48}O_3$)	Combination of sol-gel and spin-coating	Magnetic field sensor
3	Ferrite materials	Conventional solid state method	Transformer, wave suppressor electromagnet and transducer
4	Ferrite materials on thin ribbon polyester	Combustion of sol-gel and spin coating	Cartridge magnetic tape, floppy disk magnet, drive zipper hard disk and DVD
5	Amorphous magnetic alloys and Ni-Zn ferrite	Citrate precursor method	Switched mode power supplies
6	Polycrystalline Ni-Zn ferrite	Citrate precursor method	Magnetic head, EMI suppressor, microwave absorbers, circulators, isolators, phase shifter and antennas for radar system
7	MnZn ferrite	Conventional solid state method	Power application at high temperature, transformer and inductive magnetic core
8	Co_2Z and Zn_2Y hexaferrite particles mixed with metallic Ni particles	hot pressing	EMI suppressor devices
9	Carbon nanotubes mixed with	Chemical vapor deposition method	EMI shielding effectiveness absorbers

	ferrite nanoparticles		
10	Magnetic materials nanoparticles	Chemical vapor deposition method	Guide radionuclides to specific tissues, modification, detection, isolation, study of cells and isolation of biologically active compounds

All ferrite applications were highlighted in this study are in magnetic properties and most of them in high frequency applications. Application of ferrite materials especially in dielectric studies unreported by previous researchers. Therefore this study also will report about dielectric and conductivity properties when microstructure of the samples changes as the proliferation of knowledge.

1.4 Problem Statement

Ferrite material is a soft magnetic material and its use is diversifiable. Composition selection is one of the factors preferred according to usage suitability. The production of ferrite material on a large scale based on the soft magnetic material with specific composition for specific application is costly. Therefore another alternative should be explored in an effort to generate a product with restriction, but also has extensive usage. In this era users require products that are small in size, but able to perform tasks in large capacity quickly and accurately.

Lack of research had been done from nano to micron sized of nickel zinc ferrite on the conductivity of structural, electrical and dielectric properties. Alternative solutions to this problem, the samples were propose using nano-sized oxide constituent with a composition by using heat treatment (annealing process). There will be some issues that may need a great effort to overcome this deadlock. (1) Among the issues are - do heat treatment capable of producing structures that can fulfil usage per product? (2) How is the dielectric property altered when the material structure changes? (3) Does microstructure of a certain material is altered when the sintering temperature changes? (4) Does conductivity altered when the microstructure of certain material changes? (5) Does impedance of a certain material is changed when the microstructure change? (6) How the charges carrier move in a materials when the microstructure changes?

There are several hypothesis in order to solve the problem in this study:

i. when the sintering temperature of the sample increases, the crystallite sized of the samples also would be increased.
ii. when the frequency of external field increases, the conductivity of the sample would be increase.
iii. when the temperature of the sample increases, the conductivity of the sample would be increase.

iv. when the conductivity of the samples varies with frequency and temperature changes, the behaviour of carriers would be change.

v. when the conductivity of the samples varies with frequency and temperature changes, the AC and DC activation energy carriers would be change.

1.5 Objectives

The evolution of ferrite sample which is parallel with the changes in microstructure is the main objective of this study. This study investigates the samples with micron sized particles oxide and nano-sized powders. The nanoparticles oxide is prepared using dry mixing method. The sample was shaped and evolved through dry preparation technique which is crucial in this study, especially the investigation of their electrical properties. The main objectives in this study are as follows:

i) To study the effect of sintering on the microstructural and crystallite size of the ferrite samples sintering.

ii) To establish dielectric and conductivity with frequency and temperature dependencies of Nickel Zinc Ferrites.

iii) To construct an equivalent circuit model for the sample to determine the dielectric field strength of each sintering temperatures

iv) To investigate the AC and DC activation energy of the ferrites samples of each sintering temperatures.

1.6 Thesis Outline

This thesis has six chapters. Chapter 1 explains the general introduction and the applications of ferrite. Chapter 2 discusses on the literature review on the structure and dielectric properties of ferrite. Chapter 3 mentions the fundamental structure of ferrite, the general theory of dielectric and conductivity. Chapter 4 discusses the experimental procedure and theoretical aspects that have been employed in this research. Chapter 5 discusses the experimental result and finding in the present work. Finally, the conclusion and recommendation for future work are described in Chapter 6. Various results and calculation are included in the Appendices to supplement the main text within this thesis.

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