STRUCTURAL, OPTICAL AND MAGNETIC PROPERTIES OF SAMARIUM DOPED ZINC PHOSPHATE GLASSES EMBEDDED WITH NICKEL NANOPARTICLES

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In the name of Allah, Most Gracious Most Merciful

All praise and thanks are due to Allah Almighty and
peace and blessing be upon His Messenger

To my beloved parents, siblings and friends
for their love and support

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ABSTRACT

Two series of 40ZnO-(60-x)P_2O_5-$x$$Sm_2O_3$ and 40ZnO-(59-y)P_2O_5-Sm_2O_3-yNiO glasses are prepared by melt-quenching technique with $1.0 \leq x \leq 4.0$ mol% and $0.5 \leq y \leq 2.0$ mol% respectively. The glass is characterized by X-Ray Diffractometer (XRD), Transmission Electron Microscope (TEM), High Resolution Transmission Electron Microscope (HRTEM), Fourier Transform Infrared (FTIR) spectrometer, Raman spectrometer, UV-Vis NIR spectrophotometer, Photoluminescence (PL) spectrometer, Vibrating Sample Magnetometer (VSM) and Electron Spin Resonance (ESR) spectrometer. The XRD pattern confirms the amorphous nature of the glass and the TEM image reveals the existence of nickel nanoparticles (Ni NPs) with average size ~8 nm. Meanwhile, HRTEM reveals the lattice spacing of face centered cubic (FCC) structure of nickel is 0.35 nm at (100) plane. Four major IR absorption peaks are found to be at 723 cm^{-1}, 916 cm^{-1}, 1081 cm^{-1} and 1280 cm^{-1} corresponding to stretching vibration of symmetric (P-O-P), asymmetric (P-O-P), asymmetric ($P-O^-$) and asymmetric (P=O) respectively. Raman spectra display two significant peaks at 708 cm^{-1} and 1201 cm^{-1} attributed to the symmetric and asymmetric stretching vibrations of P-O-P, respectively. The Raman intensities for both peaks exhibit a rapid decrease with the increase of Ni NPs concentration. The absorption spectra of samarium ions consist of five bands attributed to absorption from the ground state $^6H_{5/2}$ to the excited states $^6F_{1/2}$, $^6F_{3/2}$, $^4F_{5/2,}$ $^4F_{7/2}$ and $^6F_{9/2}$. The surface plasmon resonance (SPR) peaks of Ni NPs are detected at 433 nm and 475 nm. Photoluminescence emission exhibits four peaks corresponding to $^4G_{5/2} \rightarrow {}^6H_{5/2}$, $^6H_{7/2}$, $^6H_{9/2,}$ and $^6H_{11/2}$ transitions. It is observed that all peaks experience significant quenching effect with increasing concentration of Ni NPs, suggesting that there is a strong energy transfer from excited samarium ions to the nickel ions. The glass magnetization and susceptibility in magnetic field up to 12 kOe at room temperature is found to be in the range of $(0.11\text{-}2.02)\times 10^{-2}$ emu/g and $(0.09\text{-}1.68)\times 10^{-6}$ emu/Oeg respectively. The obtained hysteresis curve and g-factor value of 1.99 to 2.34 indicate that the glass samples are superparamagnetic. It is concluded that samarium doped zinc phosphate glass embedded with nickel nanoparticles could be useful for magneto-optical and solid state devices.

ABSTRAK

Dua siri kaca 40ZnO-(60-x)P_2O_5-$x$$Sm_2O_3$ dan 40ZnO-(59-y)P_2O_5-Sm_2O_3-yNiO masing-masing dengan $1.0 \leq x \leq 4.0$ mol% dan $0.5 \leq y \leq 2.0$ mol% disediakan menggunakan teknik pelindapan leburan. Kaca tersebut dicirikan dengan pembelauan Sinar-X (XRD), Mikroskop Transmisi Elektron (TEM), Mikroskop Transmisi Elektron Beresolusi Tinggi (HRTEM), spektrometer Infra Merah Transformasi Fourier (FTIR), spektrometer Raman, spektrofotometer UV-Vis-NIR, spektrometer Fotoluminesen (PL), Magnetometer Getaran Sampel (VSM) dan spektrometer Resonans Putaran Elektron (ESR). Corak XRD mengesahkan sifat amorfus kaca tersebut dan imej TEM mendedahkan kewujudan zarah nano nikel (Ni NPs) dengan saiz purata ~8 nm. Sementara itu, HRTEM mendedahkan jarak kekisi bagi struktur kubus berpusat muka (FCC) nikel ialah 0.35 nm pada satah (100). Empat puncak penyerapan utama IR ditemui pada 723 cm^{-1}, 916 cm^{-1}, 1081 cm^{-1} dan 1280 cm^{-1} masing-masing merujuk kepada getaran regangan bagi (P-O-P) simetri, (P-O-P) tak simetri, ($P-O^-$) tak simetri dan (P=O) tak simetri. Spektrum Raman memaparkan dua puncak yang ketara pada 708 cm^{-1} dan 1201 cm^{-1} yang masing-masing berpadanan dengan getaran regangan P-O-P simetri dan tak simetri. Keamatan kedua-dua puncak Raman tersebut mempamerkan penurunan yang mendadak dengan meningkatnya kepekatan Ni NPs. Spektrum penyerapan ion samarium terdiri daripada lima jalur yang berpadanan dengan penyerapan dari keadaan dasar $^6H_{5/2}$ kepada keadaan teruja $^6F_{1/2}$, $^6F_{3/2}$, $^4F_{5/2}$, $^4F_{7/2}$ and $^6F_{9/2}$. Puncak resonans plasmon permukaan (SPR) bagi Ni NPs dicerap pada 433 nm dan 475 nm. Pancaran fotoluminesen memaparkan empat puncak yang berpadanan kepada peralihan $^4G_{5/2} \rightarrow {}^6H_{5/2}$, $^6H_{7/2}$, $^6H_{9/2}$, dan $^6H_{11/2}$. Diperhatikan bahawa semua puncak mengalami kesan penurunan dengan peningkatan kepekatan Ni NPs, yang mencadangkan bahawa berlakunya pemindahan tenaga yang kuat daripada ion samarium teruja kepada ion nikel. Kemagnetan dan kerentanan kaca dalam medan magnet sehingga 12 kOe di bawah suhu bilik ditemui masing-masing dalam julat $(0.11\text{-}2.02) \times 10^{-2}$ emu/g dan $(0.09\text{-}1.68) \times 10^{-6}$ emu/Oeg. Lengkuk histerisis yang diperolehi dan nilai faktor-g antara 1.99 hingga 2.34 menandakan bahawa sampel kaca adalah superparamagnetik. Dapat disimpulkan bahawa kaca zink fosfat berdopkan samarium yang tertanam zarah nano nikel boleh digunakan sebagai peranti optik termagnet dan peranti keadaan pepejal.

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LIST OF SYMBOLS

E	-	Energy
E_g	-	Energy band gap
hυ	-	Photon energy
υ	-	Wavenumber
ΔE	-	Urbach energy
Δ*E*	-	Different energy
n	-	Refractive index
δ	-	Bonding parameter
β	-	Nephelauxetic
τ	-	Decay half-life
I	-	Incident intensity
ρ	-	Density
V_m	-	Molar volume
V_t	-	Ionic packing density
V_i	-	Packing density parameter
x_i	-	Molar fraction
N	-	Ion concentration
R_M	-	Molar refractivity
α_e	-	Electronic polarizability
N	-	Avogadro’s number
H_v	-	Vickers Hardness
L	-	Applied Load
α	-	Face Angle (136°).
d	-	The Mean Diagonal

l_o	-	Length
2θ	-	Bragg angle
d	-	Interatomic spacing
m_s	-	Electron spin energy
g_e	-	Gyromagnetic
μ_B	-	Bohr magneton
μ_o	-	Magnetic field constant
v	-	Frequency
H_r	-	Resonance magnetic field
H,B	-	Magnetic field
V	-	Volume
J	-	Angular momentum
S	-	Spin
L	-	Orbital
M	-	Magnetization
$\overrightarrow{E}$	-	Electric field
M_s	-	Saturation magnetization
M_r	-	Remanence
χ_m	-	Magnetic susceptibility
H_c	-	Coercive force
m	-	Dipole moment

LIST OF ABBREVIATIONS

A/D	-	Analog/Digital
BB	-	Bragg Brentano
BO	-	Bridging oxygen
CB	-	Conduction band
CCD	-	Charge coupled device
DL	-	Deuterium lamp
DOS	-	Density of state
DTA	-	Differential thermal analysis
ESR	-	Electron spin resonance
ET	-	Energy transfer
FCC	-	Face centered cubic
FFT	-	Fast Fourier transform
FT-IR	-	Fourier transform infra-red
FWHM	-	Full-width at half maxima
GSA	-	Ground state absorption
HL	-	Halogen lamp
HRTEM	-	High resolution transmission electron microscope
J-O	-	Judd ofelt
LED	-	Light emitting diode
LSP	-	Localized surface plasmons
LSPR	-	Localized surface plasmons resonance
M-L	-	Metal-ligand
MRI	-	Magnetic resonance imaging
NBO	-	Non-bridging oxygen

Ni	-	Nickel
NIR	-	Near Infra-Red
NMEF	-	Nanometal enhanced fluorescence
NMR	-	Nuclear magnetic resonance
NPs	-	Nanoparticles
NR	-	Non-radiative
PL	-	Photoluminescence
QEs	-	Quantum efficiency
R	-	Radiative
RE	-	Rare-earth
RI	-	Refractive index
SAED	-	Selected area electron diffraction
SPR	-	Surface plasmon resonance
TEM	-	Transmission electron microscope
TMO	-	Transition metal oxide
UV-VIS	-	Ultra violet visible
VB	-	Valence band
VSM	-	Vibrating sample magnetometer
XRD	-	X-ray diffraction

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CHAPTER 1

INTRODUCTION

1.1 Background of Study

The study of glass laser as a host material deserves larger attentions. This is due to the excellent laser properties of glass possesses low melting temperature, insulating behavior, excellent ability in accommodating large amount for rare-earth (RE) ions and broader emission and absorption spectra as compared to crystalline host make them promising (Brow *et.al.,* 1995; Meyer 1997; Tischendorf *et.al.,* 2001; Ohkawa *et.al.,* 2008; Chen *et.al.,* 2009; Mansour and El-Damrawi 2010; Babu *et.al.,* 2011; Joseph *et.al.,* 2012; Li *et.al.,* 2013; Liang *et.al.,* 2014; Anigrahawati *et.al.,* 2015). Therefore, phosphate exhibits better advantages as a host material. It is reported that P_2O_5 glass is efficient for high average power pumping laser due to the high thermal shock resistance property (Chen *et.al.,* 2009). In other words, this glass has ability to maintain the efficient laser performance (Chen *et.al.,* 2009). Besides, spectroscopic quality factor of RE doped phosphate glasses (Soltani *et.al.,* 2015) is found to be in the same order than other host glasses (Selvaraju and Marimuthu 2013; Bodył *et.al.,* 2009; Agarwal *et.al.,* 2009) possess a good candidate for laser materials.

Despite, the confinement of phosphate glasses due to chemical durability is necessary to add other metal oxides as an intermediate to stabilize the glass structure (Hsu *et.al.,* 2012). Incorporation of metal oxides modifiers such as ZnO, Al_2O_3 and MgO in the phosphate host alters the structural units of the network and thereby improves their chemical durability and physical properties (Brow *et.al.,* 1995; Marzouk *et.al.,* 2013; Thomas *et.al.,* 2013). However, the changes in the overall structure depend very much on their compositions (Liu *et.al.,* 1997). For example, the Zn ions give different form of phosphate chain through the delocalization of P=O and $P\text{-}O^-$ bonds (Liu *et.al.,* 1997; Marzouk *et.al.,* 2013). The addition of other metal oxides is needed to understand the disorder nature of the glassy state and the contribution of P-P bonds. In this study, phosphate in conjunction ZnO metal oxides evaluates the potential structural modification of bridging oxygen (BO) and non-bridging oxygen (NBO) of a host glass. The stable host glass well then suitable for doped with significant RE.

Amongst other host glass, RE doped phosphate glasses are preferred in optical communication, photonics devices and lasing materials (Liu *et.al.,* 1997; Chen *et.al.,* 2009). Particularly, this study is focused on samarium as rare-earth ions doped zinc phosphate glasses due to their functional luminescence properties. The important emission of samarium ions exits from electronic transitions of ${}^4G_{5/2} \rightarrow {}^6H_{5/2}$ (green), ${}^4G_{5/2} \rightarrow {}^6H_{7/2}$ (orange), and ${}^4G_{5/2} \rightarrow {}^6H_{9/2}$ (red). Despite, efficiency of those emission possess many restrictions on their commercial exploitation and usefulness in optical devices. Hence improvement of this emission efficiency by employing of different host glass, co-doped RE and inclusion of sensitizer such as metallic NPs due to concentration and resonance on the energy level structure is promising (Som and Karmakar 2008).

Important consideration on the laser glass characterization is radiative Quantum Efficiency (QEs) analysis. It is reported that radiative QEs can be obtained from the lifetimes by Judd Ofelt (J-O) analysis and laser sources dependent to different applications (Suzuki *et.al.,* 2010). J-O theory has been used to determine the electric and magnetic contribution on total transition probability for desirable emission of RE ions. However, the calculation in all (J-O) analysis usually unconsumed the magnetic contribution. Thus the use of magnetic metal is the unique aspect of magnetic properties to study in order to increase the efficiency of the laser emission. This also due to the particular nature of electromagnetic radiation as coupled magnetic and electric fields' perpendicular to the direction of propagation.

Nickel nanoparticles (Ni NPs) are among the important materials. In periodic table nickel is in group VIIIA which is a magnetic group of material. Ni^{2+} ions in octahedral sites exhibit several strong absorption bands in the visible and NIR regions (Zannoni *et.al.,* 1999; Padmanabham *et.al.,* 2009). Furthermore Feng *et.al.,* shows that nickel occupies the tetrahedral zinc site with cubic symmetry in nickel-zinc phosphate glasses (Feng *et.al.,* 2011). Therefore a study on Ni NPs embedded in glass is very important especially to control the structural, optical and magnetic properties of the glass. In addition, nanometers magnetic particles give a more sensitive response to the change of applied magnetic field and thus exhibit superparamagnetic coexistence of single domain materials (Chudnovsky and Gunther 1988). This reveals interesting optical and magnetic properties such as surface plasmon resonance (SPR) absorption and superparamagnetism (Anigrahawati *et.al.,* 2015; Chen *et.al.,* 2015). The investigation on Mn^{2+}, Fe^{2+} and Co^{2+} (transition metal ions) with RE doped host glasses have significantly increasing the interest in their properties for magneto-optical devices such as isolator, switches and sensing application (Winterstein *et.al.,* 2013; Anigrahawati *et.al.,* 2015; Chen *et.al.,* 2015). From this research advanced understanding on the magnetic properties of rare-earth doped host glass can be studied.

Studies on nano glasses have been introduced and grown due to their interesting quantum size behavior to improve the optical characteristics of the glass. Effect of NPs on spectroscopic properties of RE doped phosphate glasses also being reported elsewhere (Soltani *et.al.,* 2015; Jiménez *et.al.,* 2011; Zhang and Noguez 2008). Nevertheless, the metallic NPs such as silver and gold have been studied to improve their capability in enhancing the luminescence and enhance the non-linear optical properties (Sazali *et.al.,* 2014; Awang *et.al.,* 2014; Yusoff and Sahar 2015).

Particularly, NPs can increase the yield of their weak optical transitions by generation of intense electric fields upon electromagnetic excitation where plasmonic metal nanostructures in the vicinity of the rare-earth (RE) ions alter their free space spectroscopic properties (Som and Karmakar 2008). This mechanism so-called nanometal enhanced fluorescence (NMEF) and it is due to the localized surface plasmons resonance (LSPR). Three main phenomena that govern the origin of the electromagnetic field enhancement are; the localized surface plasmons (LSP), the change of confinement at the metallic nanoparticles extremities and coupling the effect (Guillot and Chapple 2012).The metal nanocluster exhibited delocalized conduction electron which give absorption in the visible region due to intraband transitions (Udayabhaskar *et.al.,* 2014). Thus excitation near to resonant value of the SPR is ascribed to the amplified local field. Due to this phenomenon, it is reported that energy transfer (ET) between ions RE and NPs is possible (Awang *et.al.,* 2014). Thus nanoparticles would be the enhancer or quencher to the fluorescence of samarium ions in this glass. Therefore, this study will focus on the optical properties of the samarium ions for an optical tuning of the solid state luminescence materials.

In this study, the structural, physical, optical properties of Ni NPs on samarium doped zinc phosphate glass followed by a magnetic contribution are studied. X-Ray Diffraction (XRD), density, FT-IR and Raman analysis are able to characterize the alteration of Ni NPs on physical and structural properties of Sm^{3+} doped zinc phosphate glass synthesized via the melt-quenching technique. Transmission electron microscopy will be used to study the size distribution of the nanocluster and UV-Vis absorption studies due to SPR band. Quenching and enhancement in fluorescence with excitation near to resonant value of the SPR attributed to the amplified local field is interested in the aspect of magnetic contribution. Meanwhile vibrating sample magnetometer and electron spin resonance will be used to study the magnetic moment of the local magnetic properties due to the nature of spin-spin interaction.

1.2 Problem Statement

Throughout the previous research especially on the glass involving silver and gold nanoparticles embedded in RE doped glasses, it can be seen that the enhancement on the luminescence properties of RE ions have been successfully achieved (Jiménez *et.al.,* 2011; Mahraz *et.al.,* 2013; Awang *et.al.,* 2014; Sazali *et.al.,* 2014; Soltani *et.al.,* 2015). This is due to the effect of NPs metallic particles which yields the Surface Plasmon Resonance (SPR) phenomena. SPR that governs the confining light to nanostructures interfaces (plasmon and photonic signal) can generate an intense local electromagnetic fields (Aghlara *et.al.,* 2015). This phenomenon is dominated by the force exerted by the incident electric and magnetic field on the electric charge in the matter would increase the quantum efficiency and luminescence intensity of significant RE-doped glass emission. This phenomenon is successfully contributed by the electrical dipole moment via the existence of NPs for optical studies and applications has been extensively reported (Sazali *et.al.,* 2014; Awang *et.al.,* 2014). In spite of that, the investigation of RE-doped glasses incorporated with magnetic NPs elements are of interest for magnetic studies that could lead to the development of new magneto-optical materials. This is essential for Ni NPs elements to incorporate in the glass system due to the important magnetic properties of this metal (Yeshchenko *et.al.,* 2008). Therefore, the study of the magnetic properties of samarium doped zinc phosphate glasses embedded with Ni NPs is crucial due to lack of comprehensive data to be found in literature.

Nickel is a transition metal and generally exists in valence ions which are extremely stable in the glass matrix (Rao *et.al.,* 2008). Additionally, nickel oxide may induced of a significant changes on the structural, spectroscopic and dielectric properties of the host glasses (Rao *et.al.,* 2008; Hussein and Moustaffa 1972; Elzahedb 1994; Gandhi *et.al.,* 2011; Rao *et.al.,* 2011; Perrière *et.al.,* 2013). In addition, nickel is practically importance due to their interesting in contributing the magnetic properties in the glass system. It is found that glass embedded with Ni^{2+} ions has been an increasing demand for getting laser emission due to $^3T_2(F) \rightarrow {}^3A_2(F)$

transition in NIR region (Rao *et.al.,* 2008; Rao *et.al.,* 2011; Gandhi *et.al.,* 2011). Thus is very significant to bring Ni NPs inside the magnetic environment in the glass.

The distribution of nickel ions in glassy matrix which depend on its size and shapes is a current issue. Furthermore, properties due to the formation of non-bridging oxygen and structural units of constituent atoms in glass matrices are far from being understood. Therefore, the aims of this work are to study the structural, optical and magnetic properties of samarium doped zinc phosphate glasses embedded with nickel nanoparticles.

1.3 Objectives

The objectives of this study are:-

(a) To prepare series of samarium doped zinc phosphate glass at varying concentration of included nanoparticles by melt quenching method.
   i) 40ZnO- (60-x)P_2O_5-$x$$Sm_2O_3$; where x = 1.0, 2.0, 3.0 and 4.0 mol%.
   ii) 40ZnO- (59-y)P_2O_5-1Sm_2O_3-yNiO; where y = 0.5, 1.0, 1.5 and 2.0 mol%.

(b) To verify the amorphous nature of glass, estimate the size distribution and d-spacing of nickel nanoparticles.

(c) To determine the influence of nickel nanoparticles (mol%) on the physical, structural, optical and magnetic properties of samarium doped zinc phosphate glasses.

1.4 Scope of Study

In this study the melt-quenching technique is exploited to prepare the samarium doped zinc phosphate glasses embedded with Ni NPs. The glass nature is confirmed by X-Ray Diffraction (XRD) and the presence of nanoparticles is probed by TEM and HRTEM. These are very useful methods to provide morphological and crystallographic information on nickel nanoparticles distributed in samarium doped zinc phosphate glasses.

The glass density is measured by Archimedes method which is then be used to determine the molar volume and polarizability. The structural units of host glass are investigated by means of Fourier Transform Infrared (FTIR) spectroscopy and Raman analysis. The optical energy band gap, Urbach energy, refractive index, bonding characteristic and Ni NPs surface plasmon resonance mediated transient paramagnetic response are determined by using absorption spectroscopy. Meanwhile, the luminescence properties will be accomplished from Photoluminescence (PL) spectroscopy. Magnetic properties such as saturation magnetization, coercivity force, and *g*-factor value of Ni NPs are evaluated by means of Vibrating Sample Magnetometer (VSM) and Electron Spin Resonance (ESR) spectroscopy.

1.5 Significance of Study

This research is of significance to the domain of *'nanoglass'* (glass containing nanoparticles) technology as it extended the basic knowledge that currently exists in area. Plasmonic metal nanostructures which is due to the localized surface plasmons resonance (LSPR) phenomena have been used in diverse application such as electromagnetic field enhancement, high resolution optical imaging and biosensing (Aghlara *et.al.*, 2015; Choi *et.al.*, 2015; Liu *et.al.*, 2015).

One of the important property of surface plasmon is modes of electromagnetic radiations that are strongly coupled to collective oscillation of free-electrons at the interface of conductive materials (Chui and Lin 2014). It is motivated by possibility that surface plasmon are found to enhance the transmission of electromagnetic wave.

In this study, Sm^{3+}-doped phosphate shows several luminescence lines. The handfuls of Ni nanoparticles exhibiting novel optical properties of RE doped zinc phosphate glass with their extremely small dimensions, special surface nature and magnetic behavior. Having justified its significances, this research is poised to expand the studies for further research into the area of magnetic-optical properties.

It is known that lasing properties are due to the electrical and magnetic contribution. However, all of the calculation in JO analysis is based on the ignorance of magnetic factor because it can be assumed that the glass is non-magnetic. Thus the study on the emission efficiency due to magnetic contribution is the new area to explore. Despite of spectroscopies, magnetic behaviour in terms of magnetization and *g*-factor findings will modify the optical response of samarium doped zinc phosphate glass due to presence of Ni NPs.

1.6 Organization of Study

The entire project is documented in this thesis and it consists of five chapters. Chapter 1 describes the introduction of research background, problem statement, research objectives, scope and the significance of the study. Chapter 2 is on the literature review on the active medium materials development. It contains the review of physical, structural, optical and magnetic properties of the related glass comparison with another glass system. Chapter 3 elaborates the preparation of the samples and the employed analytical techniques. The measurement technique and instrumentations are discussed in this chapter. Chapter 4 is on the accomplished discussion of the results obtained. Finally, the conclusion and future work of current research are given in chapter 5.

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