16th International Research/Expert Conference
"Trends in the Development of Machinery and Associated Technology"
TMT 2012, Dubai, UAE, 10-12 September 2012

PREPARATION, GROWTH, AND MICROSTRUCTURAL PROPERTIES OF NONVACUUM Mn DOPED ZnMgO NANO PARTICLES

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ABSTRACT
Preparation, growth, and microstructural properties of Mn doped ZnMgO nano particles and thin films were studied. $Zn_{0.94-x}Mg_xMn_{0.05}O$ *precursor solutions were prepared by sol-gel synthesis using Zn, Mg and Mn based alkoxide which were dissolved into solvent and chelating agent.* $Zn_{0.94-x}Mg_xMn_{0.05}O$ *thin films with different thickness were produced on glass and Si (100) substrate using sol-gel dip coating. The nano particles and thin films were annealed at various temperatures and times, were tried to observe the temperature, time and thin films effect on structural properties. The particle size and crystal structure of nanoparticles were characterized by X-Ray diffraction (XRD) and Scanning Electron Microscope (SEM). The surface morphologies and microstructure of all samples were investigated by SEM and XRD.*
Keywords: Sol-gel Chemistry, X-ray diffraction, Thin Films

1. INTRODUCTION

Diluted magnetic semiconductors (DMSs) are quite important for many industrial companies in the world. Their investments and efforts tremendously increase every year to improve the technology behind film and nano particles production. Dietl at al. theoritically predicted that T_c of diluted magnetic semiconductors (DMSs) could be increased above room temperature. Sato and Katayama-Yoshida theoretically demonstrated that a ZnO matrix doped with Transition Metal (TM) atoms including V, Cr, Fe, Co and Ni exhibited FM ordering. Soon After these studies, experimental reports revealed that DMSs exhibited intrinsic room temperature ferromagnetism. Interest in DMSs has increased because of promising applications in spintronics [1-5].

Researchers have focused , ZnO doped with transition metals such as Mn, Co, Cr, Fe, and Ni and deep level impurities such as Cu , As, Sn) to explain the origin of magnetism in the DMSs material and understanding correlating magnetic properties with the TM and deep level doping concentrations. In addition some researchers have focused on the correlelation of Curie temperature with semiconductor band gap. Fabrication and characterization of (Mg/Zn)O alloys are important from the viewpoint of band gap modulation. Magnesium oxide (MgO) has a band gap of 7.8 eV and cubic crystal structure. The ionic radius of Mg^{2+} (0.57 Å) is similar to that of Zn^{2+} (0.60 Å), so replacement of Zn by Mg should not cause a significant change in lattice constants. ZnMgO doped with TM may be possible to obtain ZnMgTMO (TM=Mn, Co, Cr, Fe) with a wider band gap.

The aims of the present work are: to prepare the mixed oxides $Zn_{0.94-x}Mg_xMn_{0.05}O$ as nano-polycrystalline powders by a simple sol–gel process; to grow crack free, pinhole-free, uniform $Zn_{0.94}Mg_{0.01}Mn_{0.05}O$ thin films.

2. EXPERIMENTAL PART

The mixed oxides $Zn_{0.94-x}Mg_xMn_{0.05}O$ were prepared as polycrystalline nano particle powders with various compositions ($0.0 \leq x \leq 1.0$) using sol-gel technique. Zinc acetate dihydrate ($C_4H_6O_4Zn{\cdot}2H_2O$) and Mg 2, 4 pentanedionate ($Mg(CH_3COCHCOCH_3)_2$) and Mn (III) acetate dihydrate ($Mn_3(CH_3COO)_8$ OH) were used as precursor materials and methanol (CH_3OH), glacial acetic acit (GAA:) and acetyl acetone ($C_5H_8O_2$) were as solvents and chelating agent. The appropriate weighing amount of the Zn, Mg an Mn were put all together in a Pyrex container and dissolved in methanol. Following this, glacial acetic acid and acetyl acetone were added to containing solution. They mixed with a magnetic stirrer at room temperature until a transparent solution was obtained. Powder samples were prepared by gelling and drying of sol–gel derived precursor's solutions in a beaker. After that powders were preheated at different temperatures (300-400 °C) for 10 min. The obtained powders were ground and annealed individually in air at temperatures in steps of 100 °C from 500 to 800°C using box furnace.

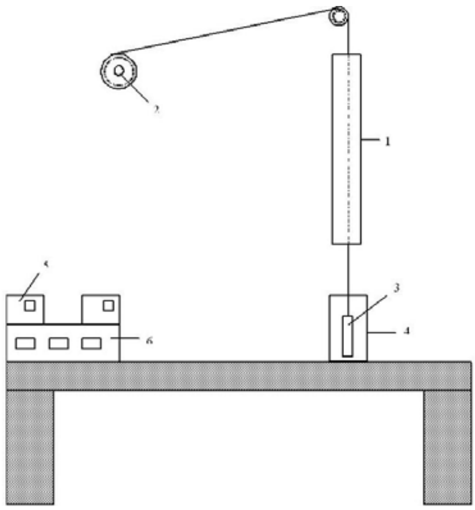

Fig. 1. The sol gel coating system; (1) furnace, (2) take-up spool, (3) substrate (4) solution container (5) electric motor for spool, (6) furnace controllers.

Glass substrates were cleaned in pure acetone and distilled water by using ultrasonic cleaner, respectively. ZnMgMnO films were growth on glass substrate using the sol-gel dip coating technique at a varying withdrawal rate as shown in Fig. 1. XRD scans were recorded using a Rigaku diffractmeter with Cu K_α radiation. Microstructure properties of prepared samples were observed using scanning electron microscope (SEM) (JEOL, JSM-5910LV).

3. RESULTS AND DISCUSSION

$Zn_{0.94-x}Mg_xMn_{0.05}O$ nano particle powders were obtained with various compositions ($0.0 \leq x \leq 0.05$) using sol-gel technique. $Zn_{0.94}Mg_{0.01}Mn_{0.05}O$ nano particles were annealed in the range of 500-800 °C from 30 minute under air using box furnace. The X-ray diffraction of $Zn_{0.94}Mg_{0.01}Mn_{0.05}O$ nano particles at various temperatures (500-800 °C) for 30 min in the air is shown in Fig.2. The reflections correspond to ZnO hexagonal wurtzide structure.

$Zn_{0.94}Mg_{0.01}Mn_{0.05}O$ thin films were deposited on glass substrate using the sol-gel dip coating system. The quality of thin film depends on withdrawal rate, drying, heat treatment condition and sol structure such as chemical composition, purity of precursor solvent catalyst materials and pH value of starting and stabilized solution.

Fig. 3a-c depicts morphologies of $Zn_{0.94}Mg_{0.01}Mn_{0.05}O$ nano particles. The grain size of the nano particles was observed to be around 90-100 nm. As seen Fig. 3d surface is smooth, dense and without crack and porosity. Thickness of the coating was controlled by viscosity of solution, number of dipping, changing withdrawal rate and temperature of the furnace.

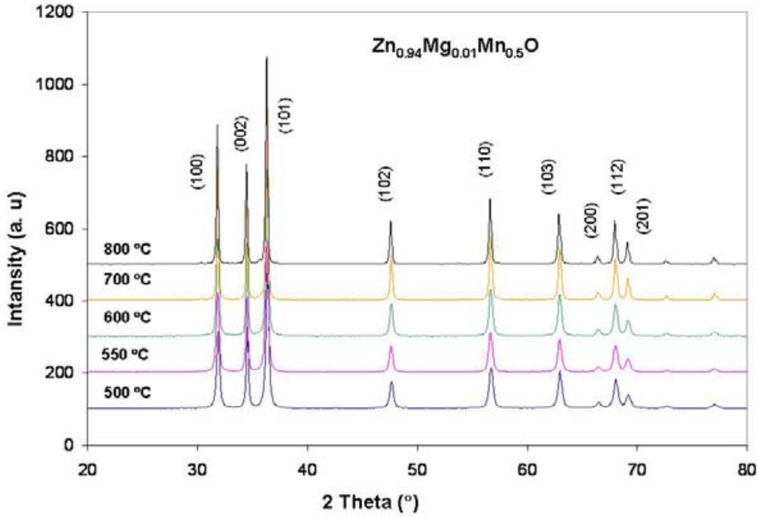

Fig.2. XRD patterns of the $Zn_{0.94}Mg_{0.01}Mn_{0.05}O$ nano particles were annealed different temperature (500, 550, 600, 700, and 800 ℃) arranged from bottom to top.

4. CONCLUSIONS

$Zn_{0.94}Mg_{0.01}Mn_{0.05}O$ thin films were coated on the glass substrate using sol-gel dip coating system. The thickness of the Mn doped ZnMgO thin film increases by increasing the number of dipping, withdrawal speed, and solution density. Smooth, dense and crack free thin film was produced. The $Zn_{0.94}Mg_{0.01}Mn_{0.05}O$ layer was observed uniform on the glass substrate by using SEM. The grain size of Mn doped ZnMgO were 90-100nm.

5. ACKNOWLEDGEMENTS

The present work was supported by the Research Fund of Bahcesehir University.

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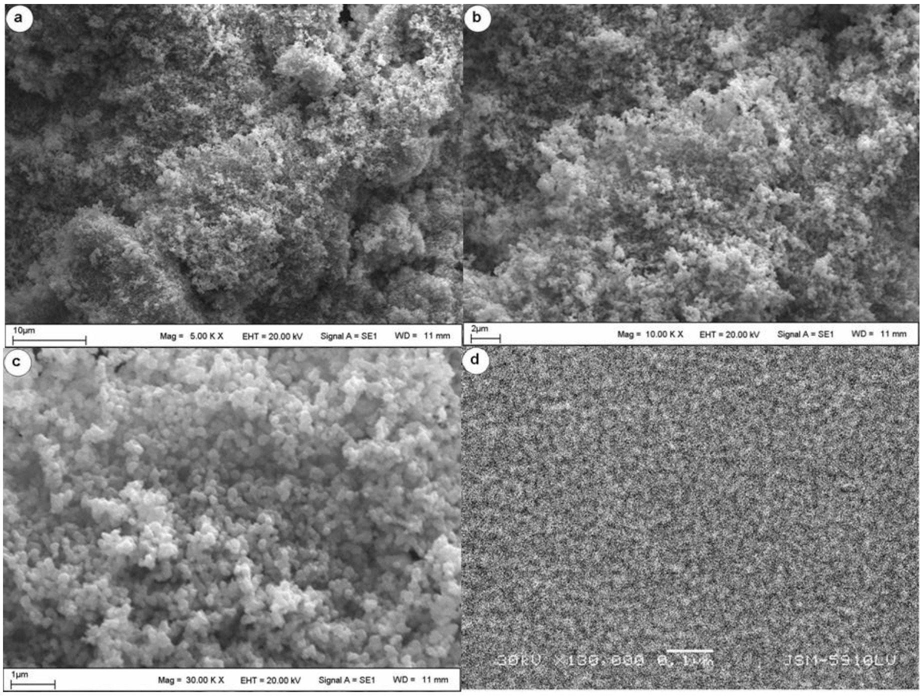

Fig.3 depicts the SEM picture of $Zn_{0.94}Mg_{0.01}Mn_{0.05}O$ nano particles and thin films. The grain size of the nano particles were observed to be around 90-100 nm.