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Effect of thermo-physical properties of Zn precursors on ZnO thin films grown by ultrasonic spray

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ABSTRACT

In the present work we have investigated temperature and precursor solution concentration influences on the density, viscosity and surface tension of zinc aqueous solutions. Three zinc salt solution sources, namely acetate, nitrate and chloride, have been investigated. The study was carried out at different concentrations ranged from 0.02 to 0.20 mol/l and solution temperatures varied from 20 to 60 °C. The measurements results show, for the whole studied salt sources, a linear increase in density, surface tension and viscosity with salt concentration. While an inverse behavior of these properties is observed with increasing solution temperature. Zinc acetate has the lower surface tension and viscosity, while zinc chloride has the largest ones. Droplets Weber, Reynolds numbers and surface enthalpy formation have been estimated from solution properties measurements. Ultimately a correlation between the used salts, concentrations and the obtained ZnO thin films morphologies and structures is addressed. Solution viscosity and surface tension are key parameters controlling the films growth and morphology. At fixed substrate temperature, films with smooth surface can be produced by reducing the surface tension and the viscosity of the starting solution.

1. Introduction

During the last decades, zinc oxide (ZnO) thin films have emerged as one of the most promising oxide materials. This is motivated by its interesting optical, electrical properties, high chemical and mechanical stability. Beside this, its abundance and non toxicity promote ZnO thin films as low cost potential candidate for several applications such as solar cells [1–3], gas sensors [4,5] anti-bacterial activity [6,7], photo catalysis [8,9] piezoelectric devices [10,11] and UV detectors [12].

Various techniques have been used to produce ZnO thin films, among them the chemical methods which are achieved in a liquid medium based on solutions prepared from different precursors. The most important techniques using this process are: sol-gel [13,14], electrodeposition [15,16], chemical bath deposition (CBD) [17,18] and spray pyrolysis technique [19,20]. In these techniques, the thermo-physics properties of the used solutions, namely density (ρ), viscosity(μ), surface tension(σ) are key parameters that may control the properties of the obtained films. However, there is a serious lack of studies dealing with the effect of deposition parameters on the thermo-physical properties of the starting solution.

Mainly, zinc acetate [21,22], zinc nitrate [23,24] and zinc chloride salts [25,26] are used as starting solution for ZnO thin films deposition by chemical methods. Many researches were carried out on the investigations of solution precursor nature and concentration effect on ZnO thin films properties. Bacaksiz et al. [27] have prepared ZnO thin films using various solution precursors such as zinc chloride, zinc acetate and zinc nitrate. They observed that the precursor nature affects the structural and optical properties of ZnO thin films. Similar study was carried out by Lehraki et al.[28]. They found that, films deposited with zinc acetate are characterized by a smooth surface, dense structure with high transparency, while films deposited with zinc chloride have a rough surface, better crystallinity and low optical transmittance. Alami et al. [29] have investigated the effect of zinc nitrate solution concentration on ZnO films morphology and optical properties. They reported that increasing precursor concentration leads to an enlargement of crystallite size and a reduction in films optical transmission in the visible range. The same observations have been reported by Gaikwad et al. [30] and Baneto et al.[23] in the cases of zinc acetate and zinc chloride solutions.

In the most published studies dealing with ZnO thin films deposition, researchers attempt to correlate post deposited films properties with the experimental conditions (i.e chemical source nature, deposi-

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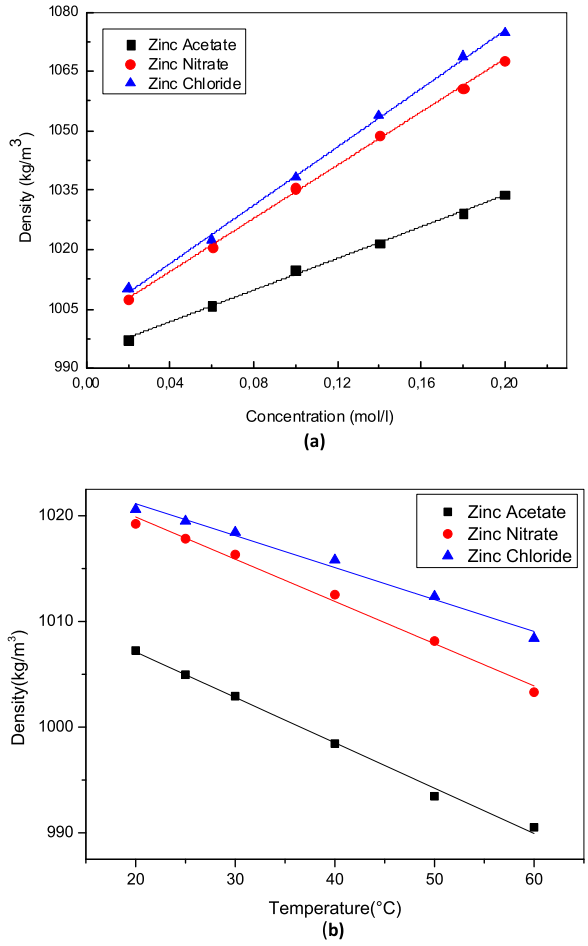

Fig. 1. (a) Density variation versus concentration for the three zinc aqueous solutions at fixed temperature (30 °C), **(b)** Density variation versus temperature for the three zinc aqueous solutions at fixed concentration (0.05 mol/l).

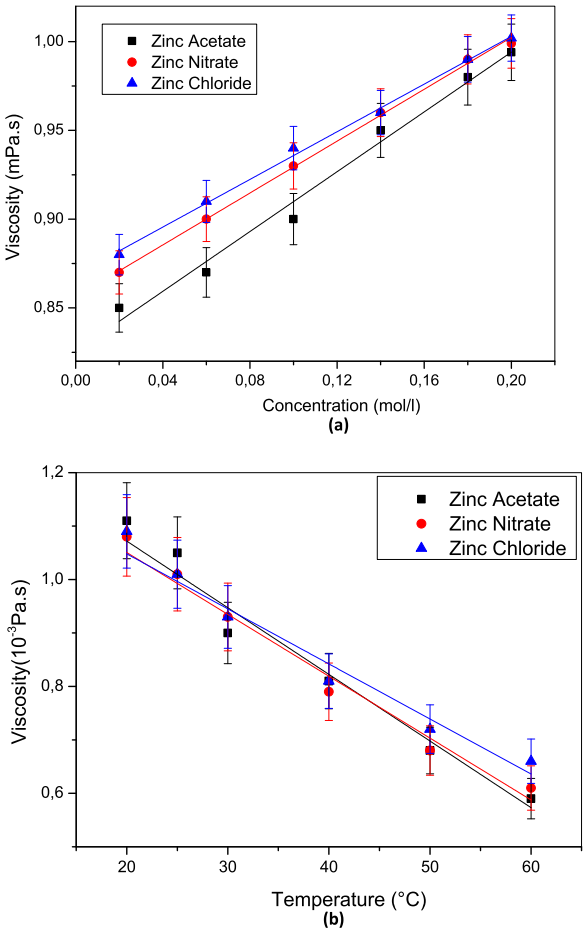

Fig. 2. (a) Viscosity-Concentration dependence for three zinc aqueous solutions at fixed temperature (40 °C), **(b)** Viscosity- temperature dependence for three zinc aqueous solutions at fixed concentration (0.10 mol/l).

tion temperature, concentration, flow rate, substrate ...etc). While, actually the experimental parameters alter indirectly the films properties and growth through their direct influence on thermo-physical properties (i.e. density, viscosity, surface tension) of the starting solution. To the best of our knowledge, few attentions have been paid to the thermo-physical properties studies of the used aqueous solutions prepared from different zinc salts for ZnO thin films deposition by wet chemical routes [28].

In the present work we have investigated the influence of solution concentration, nature and temperature upon solution density, viscosity and surface tension, Weber number (We), Reynolds number (Re) and surface formation enthalpy (H) of the three commonly used zinc salts. These quantities control the shape and the dynamic of the droplet during their landing on the substrate. A correlation between solution thermo-physical properties and structural properties of the post deposited films is addressed.

2. Material and measurements

Zinc acetate dihydrate ($C_4H_6O_4Zn{\cdot}2H_2O$), zinc nitride hexahydrate ($Zn(NO_3)_2{,}6H_2O$) and zinc chloride ($Zn\,Cl_2$) were used in this study. To prepare aqueous solutions, each salt was dissolved in distilled water, six different concentrations 0.02, 0.06, 0.10, 0.14, 0.18 and 0.20 (mol/l) were used. The obtained solutions were transparent. The solutions thermo-physical properties measurements were carried out in temperature ranged from ambient 20 °C to 60 °C (just before solution evaporation).

The kinematic viscosity of aqueous zinc solutions was measured by Ubbelohde type viscometer. The viscometer was suspended inside a water bath heated with thermoregulation. 15 ml of testing solution is used, the temperature measurement was ranged from ambient to 60 °C. Before each measurement, the used vessel is cleaned with a solution composed of 15% H_2O_2 et 15% HCl followed by methanol rinsing to remove any impurities that may hinder the liquid motion during measurement. The time taken by the liquid free surface to pass, during its flowing, through two marked positions is used for kinetic viscosity measurement. The dynamic viscosities were calculated by multiplying the measured kinetic viscosities by the solution densities. Each reported viscosity value was the average of at least four readings. The surface tension of the aqueous zinc solutions was carried out using a Dunouy tensiometer. The viscosity measurement is based on the determination of the force necessary to take off a platinum ring, with a diameter of 3 cm, immersed in the solution. The ring was thoroughly cleaned and dried before each measurement. The solution was placed in thermostated bath with varied temperature from the ambient to 60 °C. Each reported data point was taken as an average of five readings.

ZnO thin films were deposited by ultrasonic spray pyrolysis, on glass substrate, using stating solutions prepared with the three studied salts with three concentrations 0.05, 0.10 and 0.15 M. The ultrasonic generator delivers 40 kHz frequency signal atomizing the solution to a steam composed with uniform fine droplets having a diameter of 40 µm. The substrate temperature was fixed at 300 °C, the nozzle substrate distance was equal to 5 cm, the deposition time was 10 min. The obtained films thicknesses are almost equal to 200 nm.

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