Faculty of Women for
Arts, Science and Education
Ain Shams University

Characterization of the Passive State on Zinc in Alkaline Buffer Solutions

A Thesis
Submitted for Partial Fulfillment of the Requirements for The Master Degree of Science (M. Sc.)
in Chemistry (Physical Chemistry)

Presented *by*
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(W. R. Abd-Ellatif)
B.Sc. 2013

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To whom it may concern

This is to certify that: Walaa Ragab Abd Ellatif Ahmed has attended and passed successfully the following postgraduate courses as a partial fulfillment of the requirements of the degree of master science:

1. Writing scientific research SCR 610
2. Instrumental analysis CHEM 601
3. Advanced thermodynamics CHEM 612
4. Electrochemistry CHEM 613
5. Ethics of scientific research SCR 620
6. Spectroscopy CHEM 630
7. Surface and kinetics CHEM 615
8. Polymer CHEM 617

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Aim of the work

The present work is intended to investigate the electrochemical and semiconducting properties of spontaneously formed, as well as electrodeposited thin passive films on pure zinc in alkaline carbonate/bicarbonate buffer mixtures. The effect of different operating parameters such as: solution pH over the range (9.2-9.8), ambient temperature, immersion time, formation voltage, in addition to dopant nature and its concentration were carefully examined and discussed. The techniques used to fulfill these objectives include open circuit potential (OCP), electrochemical impedance spectroscopy (EIS) and potentiondynamic polarization measurements, in addition to Mott-Schottky analysis and surface characterization techniques using FE-SEM and EDX.

Undoped and doped electrodeposited thin oxide films on zinc metal were formed potentiostatically under controlled potential from -1.04 to -0.99 V (vs. Ag/AgCl), while the choronoamperometric trace was recorded during the deposition method. The processing solution was pH 9.2 borate buffer solution free or containing the dopant Li^{+} or In^{3+} ions.

Summary

Zinc (Zn) metal and its oxide (ZnO) are recognized as being extensively used engineering materials for widespread applications in many industries. Therefore, the electrochemical and semiconductor properties of spontaneously formed and electrodeposited passive films on pure zinc metal were carefully investigated to gain more information about their behaviors in alkaline carbonate/bicarbonate buffer mixtures.

The effect of different operating parameters such as: solution pH, temperature, immersion time and formation voltage (V_a), in addition to the dopant (nature and its concentration) were examined and discussed.

The study was performed using different electrochemical measurements, Mott-Schottky analysis and surface characterization techniques. Undoped and doped electrodeposited thin oxide films on zinc metal were formed potentiostatically at -1.04 to -0.99 V (vs. Ag/AgCl), while the choronoamperometric traces were recorded during the deposition method. The processing solution was pH 9.2 borate buffer solution free or containing the dopant Li^+ or In^{3+} ions.

The results revealed direct correlation between each of buffer pH, temperature, immersion time and V_a with the total film

resistance (R_f) and its thickness (δ_f). Generally, zinc passivation is enhanced with increasing pH over the range 9.2 to 9.8, decreasing the ambient temperature, increasing immersion time or the formation voltage (V_a), in parallel with the increase of its film thickness. In the meantime, (R_f) values for the undoped anodic ZnO films are always higher than those for the doped ones.

Mott-Schottky results showed that the charge carrier donor concentration (N_D) increases by increasing either pH, V_a or In^{3+} doping level, but it decreases by increasing the solution temperature or Li^+ dopant concentration, commensurate with the increasing or decreasing trends, respectively in the thickness of the formed oxide film.

CHAPTER I

Introduction