MICROSTRUCTURE AND PHYSICAL PROPERTIES OF THIN INDIUM FILMS

M. Sc. THESIS
Submitted to Faculty of Science
Ain Shams University

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(B. Sc.)

1982

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ACKNOWLEDGEMENTS

I want to express my deep gratitude to Prof. Dr. Abdel Aziz Ali Mohamed, Head of Physics Department, Faculty of Science, Ain Shams University, for his continuous supervision and helpful suggestions.

My deep thanks to Dr. Fathy Semey Saeed, Associate Prof. of Physics, Faculty of Science, Ain Shams University, for his interest and valuable advice during supervision.

Also my deep gratitude for Dr. Elhamy Aziz Abou-Saif, Associate Prof. of Physics, National Research Center, Dokki, for his stimulating supervision and valuable suggestions and discussions.

My thanks are for all my colleagues at Electron Microscope and Thin Films Laboratory, NRC, Dokki and my colleagues at Physics Department, Faculty of Science, Ain Shams University, for their true help.

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بسم الله الرحمن الرحيم

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CONTENTS

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SUMMARY

The microstructure and optical properties of thin indium (In) films of different thicknesses were studied. The indium films were deposited onto crystalline mica substrate with rate of deposition 1 Å/sec. by thermal evaporation technique. The vacuum evaporation coating plant from Carl Zeiss Jena, DDR of vacuum 10^{-5} Torr was used in the present work.

The structural investigation of indium films of thicknesses in the range from 200 to 800 Å was carried out by using transmission electron microscopy and diffraction techniques. The analysis of electron diffraction patterns has shown that films of thicknesses 200, 320 and 450 Å are of polyctystalline structure, i.e., the crystals are aggregated in random orientation. On the other hand, the patterns of films of thicknesses 600, 750 and 800 Å have single crystalline structure.

The average volume growth was found to be 26.86×10^{-18} cm^3/sec for film thicknesses 320 and 800 Å. The morphological characteristics of the films had shown the presence of primary, secondary and tertiary nuclei. The epitaxial growth was observed.

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The optical measurement of indium films deposited on discs of mica as a substrate, were carried out in infrared region (i.e. in the range from 2.5 to 23.25 u). The optical constants, refractive index n and extinction coefficient K, were determined for film thicknesses 1500 and 1800 Å. Moreover, the optical dielectric constants ϵ_1 and ϵ_2 were calculated showing variation with photon energy. The spectra of energy loss function were also determined. Besides, the number of conduction electrons per unit volume n_c, the velocity at Fermi surface μ, the relaxation time τ and the static bulk conductivity σ_0 of indium films were determined from plasma frequency ν_0 and damping frequency ν_R.

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INTRODUCTION

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INTRODUCTION

Thin solid films were first obtained by electrolysis in (1838). Bunsen and Grove obtained metal films in (1952) by means of chemical reaction and glow discharge sputtering. This historical review has been mentioned by K.L. Chopra (1969)$^{(1)}$. Faraday (1857)$^{(2)}$ obtained metal films in (1857) by thermal evaporation and explosion of current carrying metal wire. Thin films have been extensively studied for over a century. However, until this decade sufficient technological progress has not been made to give reasonable scientific confidence to thin film research. The development in this decade has made directly or indirectly, significant contributions to many areas of basic and applied solid-state research. Till now a very considerable part of our industry is involved with thin films. Some of this is old-established, as in the use of electroplated films for protection or decoration. Much of it was of interest and involves the use of techniques which have been developed over the last few years$^{(3)}$.

Most physical properties of films, i.e., optical chemical, magnetic, dielectric, ... etc. were of importance in scientific and technical applications. The technical interests which stimulated these studies have also been rewarded in the form of useful inventions. These inventions were such as variety

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of active and passive microminiaturized components and devices, solar cells, radiation sources and detectors, magnetic memory devices cyclotom, balometers, interference filters, reflection and interflection coatings ... etc. Moreover, after the oil crisis of (1973), the cost of energy sources is going very-high. More and more people were trying to use solar energy. Here, collectors were the main problem. Glasser (1977)$^{(4)}$ put forward a very revolutionizing idea to use satellites for generating power in space, then to transmit to the earth preferably without any cable. To avoid massiveness of the generating satallites, thin films of suitable materials can be used as collectors.

The important parameters affecting the deposition of thin metallic films by evaporation are, the type of substrate, the substrate temperature, the film thickness, the rate of deposition, the residual gas pressure, the contamination, and the vacuum pressure.

1- Effect of Substrate

The substrate is of primary importance in the formation of the film. Material of the substrate and crystallographic state, (amorphous, polycrystalline, single crystalline) surface roughness and surface contamination are important parameters. The material of film and substrate are of considerable importance since the binding forces between the atom of the film and those of the surface determine the mode of growth (continuous island formation etc. (1963))(5). Furthermore, contamination of the substrate is extremely important since even a monoatomic layer can alter the growth conditions completely (1963)(5).

Thin metalic films deposited by evaporation either on collodion or carbon substrate were investigated as early as (1949) by Levinstein(6), followed by Sennett and Scott (1950)(7). It was found that vapour diposited layers of metals at room temperature consisted of randomly oriented polycrystals. On the other hand, few defects appeared in the metallic films deposited on smooth surface of single crystals. Although cleaned surface of rocksalt and mica have been frequently used, they contain irregular cleavage steps (1963)(8).

When the cleavage steps are large, correspondingly, large steps appeared in the evaporated films (1939)(9). Bassett (1958)(10) showed that although monoatomic steps are preferable nucleation sites, they do not produce misorientation of nuclei. For some purposes, metal substrates were desirable, because film deposited on them become continuous at lower thickness and exhibited less twining than films grown on ionic substate (1963)(8). But when metallic substrates are used, two difficulties are encountered(8). First, the presence of any oxide layer that might interfere with the film growth, so the

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surface must be free from any oxide traces. The second difficulty is alloying at the interface which can be minimized by using law substrate temperature.

2- Effect of Substrate Temperature :

The film formation is also affected by the substrate temperature, this determines the surface mobility of the arriving film atoms conjunction with the forces between film and substrate atoms. The surface mobility also influences the number of collisions among film atoms on the surface of the substrate(1955)$^{(11)}$, since nuclei for the formation of crystallites can result from such collision at least in the initial stages of film formation.

Heating the substrate aids to clean its surface, increases the surface and volume diffusion and provides some activation energy (1965)$^{(12)}$. This activation energy is required to allow the deposited atoms to take up positions of minimum potential associated with ordered epitaxy.

There are three major effects (1963)$^{(8)}$ of substrate temperature on the critical size of the grains formed by evaporation on the substrate, namely, the rate of formation of the nuclei, the mobility of the adsorbed atoms and the annealing defects in condensed films. Pashely (1959)$^{(13)}$ showed that elevated substrate temperature leads to films of larger grain

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size, when single crystal substrates were used(13). Oriented growth was more likely to occur at higher temperatures. Moreover, it was found(13) that the nature of the orientation depends upon the substrate temperature and film thickness.

3- Effect of Film Thickness :

As early as (1939), Bruck(14) showed that the film thickness could be influenced by rising the temperature which could occur during the deposition. This rise in temperature resulted either from the radiant heat from the source or from the thermal energy carried by molecules.

Kirchner and Gramer (1938)(15) found that the crystallographic structure in oriented metallic deposits varied with the mean thickness. Pines and Chank (1966)(16) showed that a change of the film thickness was accompanied by changes in the structural characteristics and strength. Also, the increased strength is dependent on film thickness and did not depend on the nature of substrate.

4- Effect of Deposition Rate :

The rate of deposition influences the grain size of the deposit. Campbell et al (1962)(17) found that the grain size of a polycrystalline LiF deposit being normally finer when the deposite rate is faster.

As the deposition rate increased, the film becomes less perfect(8), this may be due to an increased rate of nucleation or to more misoriented nuclei. Pashley (1965)$^{(12)}$ stated that there was a critical concentration of atoms adsorbed on the substrate surface. Above this critical value, the nucleation rate was large and below it the nucleation rate was very small.

5- Effect of Residual Gas Pressure :

It was found (1964)$^{(18)}$ that a small amount of gas molecules adsorbed on the substrate surface had a great effect on the orientation of some metal films. The effect of oxygen in inhibiting edge break up and agglomeration of tin atoms was attributed to a reduction in surface mobility of the atoms$^{(19-20)}$ (1961-64). Pashely (1964)$^{(21)}$ stated that the presence of the gas has some possible effect on the mode of growth, and hence the microstructure of the deposit. Recently, it was shown by Presot and Wilmann (1967)$^{(22)}$ that the contamination of the film by gases influenced the growth process and the structure of the film. It was shown that (1962)$^{(23)}$ the step structure on a rocksalt surface cleaved in vacuum was very different from the step structure on a surface cleaved in air. The critical temperature required for the epitaxial growth was found (1962)$^{(24)}$ to be lower than that for the growth on surface cleaved in air. Pashely et al (1965)$^{(12)}$ found that contaminations due to adsorbed hydrocarbon could form a bridge between two nearly touching islands.

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6- Effect of Vacuum Pressure :

Murr and Inman (1966)$^{(25)}$ studied the effect of the vapour pressure upon the sup-structure of thin evaporated films of Ag, Cu, Ni, and Al. A decrease in the nominal vapour pressure before evaporation stimulated a marked increase in grain size with consistent trend towards a preferred orientation.

Accordingly, all of the forementationed parameters have great influence on the physical properties and the microstructure of thin metallic films. Hence, the optical properties and the microstructure of one material of particular interest in this field, which is Indium, will be studied in the present work. From the literature, it was found that Indium, In, has the tetragonal unit with $a_o = 3.244 \overset{o}{A}$ and $C_o = 4.938 \overset{o}{A}$ (1963)$^{(26)}$. This will be carried out taking into consideration some of the parameters, which, affecting the deposition of thin Indium film by thermal evaporation. This study is taken up with a view to understand the behaviour of thin films of Indium since these films are of great importance in industrial application.

The reflectance measurements of Indium films between 2.5 and 1.5 eV have been studied by Lemonnier (1975)$^{(27)}$. These films were obtained by evaporation in Ultra-high vacuum

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on cooled substrates. Anomalous absorption was observed on unannealed samples and remained after annealing.

The optical constant were obtained by Kramers - Kranig analysis of reflectance data. The optical conductivity σ and real part of the dielectric constant ϵ_1 were deduced and compared with the results obtained from Ashcroft's theory (See Ashcroft and David Mermin 1976)(28). The agreement was good up to 9 eV, beyond this energy the effect of d-transitions shifted the experimental curve. The energy loss function peaks were observed at 11.42 ± 0.02 eV(28).

Island density and grain size variation as a function of film thickness and substrate temperature of vapour deposited Indium and Tin thin films were investigated (1972)(29). It was observed that the nucleation and growth characteristics of Indium and Tin films were different even when the substrate temperature were in the same ratio of their melting points. These differences were ascribed to the differences in interfacial surface energy, nucleation barrier and critical thickness. Island density curves systematically illustrated the occurence of primary, secondary and tertiary nucleation. This feature was also apparent from transmission and scanning electron micrographs of films deposited at 130°C. The presence of broad peaks in island density curves is due to the formation and growth of secondary nucleated island.

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