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Determination of Stress in Strontium Sulphide Thin Films Using Holographic Interferometry

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Abstract-A technique known as double-exposure holographic interferometry (DEHI) was used to explore how strontium sulphide distorted the surface of stainless steel when applied using the dc eletrodeposition method. By changing both solution concentration and deposition duration in a (Srcl2+NaS2O3) solution, the films were deposited. When the hologram is lit, interference fringes can be seen on the surface of the substrate. Furthermore, it was discovered that the fringe spacing varies depending on the concentration of solution and the time elapsed since deposition began. Evaluations were conducted on film thickness, mass of strontium sulphide deposited, and stress.

Keywords: Strontium chloride, sodium thiosulphate, eletrodeposition, X-ray diffraction, holographic interferometry.

1.INTRODUCTION

As the divalent counterparts to the alkali halides, the IA-VIIB compounds, strontium sulphide (SrS) is an alkaline earth metal. Both types of compounds share a common cubic crystal structure [1] and all are composed of ions with electronic configurations comparable to rare gases, which have a closed-shell shell.

Strontium sulphide (SrS) is a popular IIA-VIA chemical due to its high luminous yields. For applications such as infrared sensors and X-ray imaging plates [2], optical storage media [3], and electroluminescent (EL) displays [4,5], these materials have significant potential in thin film form. As a result of its bandgap, strontium sulphide (SrS) is frequently used because of its self-absorption properties.

In terms of holography, interferometry is the most revolutionary and crucial application. Hologram [6] interferometry is an expansion of the interferometric measuring technology in which at least one of the waves that interfere is recreated. In the present work, SrS films were deposited using d.c. eletrodeposition technique was used to study different parameters of ($SrCl_2$+$Na_2S_2O_3$) thin films. The holographic interferometry has been regarded as one of the standard technique for the measurement of small object displacement and surface deformation in strain analysis, non-destructive testing etc**.[7,8].**This technique can be used for the investigation of mechanical properties of materials and in particular, the possibility of measuring important material characteristics **[9].**For example , the stress is the property of thin film can be measured holograpically. This is a useful application of holography because the conventional methods of estimation of strain and stress are rather complicated.

The DEHI technique was employed in this study to investigate the properties of SrS thin films. Material testing can be done non-destructively using the DEHI approach. Comparing each point on an object's surface to itself before and after a change allows for quantitative and qualitative analysis of even the smallest changes in an object's contours with this technique. Since minor dimensional changes occur in materials over time, the DEHI approach has been employed to investigate them. Even on a rough surface, the DEHI approach produces clear interference fringes [10,11]. Engineers can now acquire additional information about an object in a variety of situations by using surface displacement relative to an initial position thanks to the DEHI approach [12]. The application of the DEHI approach is unique and suited for recording long-term changes

in relative surface displacement. If the surface of a double-exposure hologram is significantly deformed between the two exposures, the reconstructed picture will be obscured by interference fringes [13]. Changes to a solid body caused by the shape or finish of its surface can be studied in great detail[14]. Strontium sulphide was deposited into stainless steel using the dc electrodeposition method, and we then used the DEHI approach to examine how this affected the surface deformation [15]. Using this method, we were able to accurately estimate the amount of stress in the strontium sulphide film.

2.EXPERIMENTAL SETUP

The electrodeposition technique was used to create strontium sulphide thin films on a stainless steel substrate (5cm x 1.5cm x 0.1cm) after various preparative parameters, such as solution concentration and pH = 4.0, were optimised. Polished paper, detergent powder, and brasso were used to smooth off the rough edges of the substrate. A second ultrasonic cleaning of these substrates was done in double distilled water. SrCl2 and Na2S2O3 were used to prepare the electrodeposition bath in double-distilled water of analytical quality. In comparison to other metals, strontium has a higher reduction potential (2.899 V Vs SCE) and is therefore more difficult to deposit on substrates. The ligands can be complexed with strontium metal to generate stable complexes, a simple solution to this problem. The complexing agent of choice is EDTA (Ethylene diamine tetraacetic acid). When the complexing agent is added to the bath, additional complexed ions with different characteristics can be formed. As a result, each metal ion is an integral member of the complexing agent ring because it forms the complex with free metal ions. Thus, the cathodic polarisation shifts as a result of this. Substrate surface shape influences deposition potential, as does a substance's affinity for its substrate and its hydrogen overpotential. The deposition potential might go from a reduction potential to a positive or negative value depending on the value of these variables. In an electrolyte bath, EDTA, sodium thiosulfate, and strontium chloride solutions are mixed to produce a high-quality, thin film. A 4cm by 2cm graphite plate polished to 0.2cm was utilised as a counter electrode. For the deposition at room temperature in potentiostastic conditions, the saturated calomel electrode (SCE) was used.

The stainless steel plate was attached to the sample holder using an ultrasonic cleaner. With insulating tape, a stray section of the stainless steel plate was hidden from view from the front. The electrodeposition bath containing (SrCl2 + EDTA + Na2S2O3) solution was then placed on top of the sample holder so that the substrate was submerged in the solution at all times. To conduct the electrodeposition, we used a glass beaker fitted with a sample holder holding 50 ml of water as our electrodeposition bath. While the graphite acts as an anode, the stainless steel acts as a cathedral conductor. Potentiostatic conditions were maintained at room temperature for the depositions. (SrCl2 + EDTA + Na2S2O3) is displayed in Fig. 1 as the cathodic polarisation curve.

The deposition potential was determined by extrapolating the straight line part of the polarisation curve to the voltage axis. -0.1 volts against SCE was found to be the constant potential for the deposition of the films in this study. Double distilled water was used to wash the films after they had been deposited. Structural analyses were carried out utilising a Philips X-ray machine (PW-1710) and a Cu k target, as depicted in Figure 2; the diffraction pattern is presented. Consideration was given to the density of material deposited to determine the film's thickness.

3. HOLOGRAPHIC RECORDING THROUGH DOUBLE-EXPOSURE HOLOGRAPHIC INTERFEROMETRY

The stainless-steel substrate immersed in the (SrCl2 + EDTA + Na2S2O3) solution in electrodeposition bath was employed as an object for a double-exposure holographic interferometry investigation. Strictly speaking, no deposition took place during the recording of the single hologram of an object on a holographic plate. The current was provided across the electrodes and a thin film of strontium sulphide was deposited on the substrate without changing the experimental setup, such as the object's position and the holographic plate's position. The identical holographic plate was used for the second exposure of the distorted substrate. D-19 Kodak developer was used for 3 minutes, followed by a quick fixer (sodium thiosulfate) for roughly 10 minutes to fix the image of the substrate on the holographic plate. In order to see the reconstructed picture of the substrate, the processed hologram was inserted in the reference

beam and replaced. When the image is reconstructed, the interference fringes can be seen to be concentrated on the substrate's surface, where the thin layer was applied to begin with.
Different molarities of (SrCl2 + Na2S2O3) solution, i.e. 0.05, 0.1, and 0.15M, were produced. From 40 seconds to 130 seconds, strontium sulphide was deposited. Film thickness has an effect on the growth of substrate fringes, as may be seen from hologram images that have been digitally reconstructed. The "EX-ACTA VAREX II b" type camera was used to capture these photos, which are shown in Fig. 3. (a-l). The interference fringes may be seen in the black lines on the light background of each shot.
A traditional two-beam off-axis approach using a 3 mW He-Ne laser (= 6328 Ao) on Kodak 8E75HD holographic plates was used to record all of the holograms.

4. MEASUREMENT OF INTRINSIC STRESS OF FILM DEPOSITED

Magill et al.[17] describe a non-destructive method for quantitatively measuring stress in thin films using holographic interferometry. The relation also contributes to the film's internal stress [18, 19].

$$S = \frac{t_s^2 Y_s \delta}{3l^2 t_f} \qquad \text{----------(1)}$$

Where,

S is the stress in dyne/cm^2

t_s is the thickness of substrate.

Y_s is the Young's modulus of substrate.

l is the length of the substrate.

t_f is thickness of thin film and

δ is the deflection of the substrate.

To measure substrate deflection, use this method. The substrate is irradiated with a beam of light that is at an angle to normal, and the resulting hologram is viewed at an angle. A fringe pattern placed on the reconstructed image corresponds to a substrate deflection [20] caused by,

$$\delta = \frac{n\lambda}{\cos\theta_i + \cos\theta_s} \qquad \text{----------(2)}$$

Where,

n is total number of fringes and

λ is the wavelength of laser light used.

In general, the angles θ_i and θ_s are sufficiently small, so that,

$$\delta = \frac{n\lambda}{2} \qquad \text{----------(3)}$$

For the calculation of the thickness of film deposited, weight difference method was used. The density of the deposited material was considered from Joint Committee on Powder Diffraction Standards (JCPDS) data file [75-895] and it was 3.70 gm/cm$^{3.}$

5. RESULTS AND DISCUSSION

X-ray diffraction was used to identify the structure of strontium sulphide film between the diffraction angles of 0o and 100o. Films with polycrystalline orientation were discovered by XRD to

have X-ray patterns along the (1 1 1), (4 0 0, and (4 2 0, respectively) planes of the X-ray pattern. Analysis of XRD d-values showed that the material was face-centered cubic SrS, based on the JCPDS data file [75-895] d-values.

The chemical reduction reaction at the cathode in an aqueous solution (pH, 4) is as follows –

$$SrCl_2 \longrightarrow Sr^{++}_{aq} + 2Cl^{-}_{aq} \quad \text{------- (4)}$$

When water is decomposed into hydrogen and oxygen, the reaction described below takes place,

$$2H_2O + 2e^- \longrightarrow 2H^+ + 2OH \quad - 1.0697 \text{ Vs SCE} \quad \text{------- (5)}$$

A change in pH at the cathode as a result of the production of OH- ions increases the likelihood of hydroxide precipitation[21]. The Sr(OH)2 in this situation, according to –

$$Sr^{2+} + 2OH^- \longrightarrow Sr(OH)_2, \quad pH > 5 \quad \text{-------- (6)}$$

after reaction, pH was measured to be 4 before and after. Electrodeposition of SrCl2 precursor solutions onto stainless steel results in the production of a Sr(OH)2 coating, according to this mechanism. It is similarly electrodeposited from Na2S2O3 solution in aqueous medium (pH = 6).

$$S_2O_3^{2-}(aq.) \rightleftharpoons S + SO_3^{2-}(aq.) \quad \text{---- -0.75V vs SCE ---(7)}$$

It is necessary to mix these two 0.1M normality solutions in the correct ratio to generate a thin film of SrS. These are the chemical reactions that occur as a result of them.

i) $Sr^{2+} + 2e^- \rightleftharpoons Sr$ ------ (8)

ii) $S_2O_3^{2-}(aq.) \rightleftharpoons S + SO_3^{2-}(aq.)$ ------ (9)

iii) $S + 2e^- \rightleftharpoons S^{2-}$ ------(10)

iv) $Sr^{2+} + S^{2-} \longrightarrow SrS$ ---- - 1.0 V vs SCE ------(11)

The pH value remains same before and after reaction (pH, 4). From the reaction mechanism it is concluded that from ($SrCl_2$ +$Na_2S_2O_3$) precursor solution leads the formation of SrS film.

An industry that has grown tremendously has a lot of focus on internal stressors. When it was discovered that thin electrodeposits cracked or peeled away from the electrode in the early days of electrodeposition, researchers began investigating the effects of stress on thin films[22]. S.N.Sahu et.al. [23] used laser beam deflection approach, researchers were able to detect stress in thin films that were formed on a substrate as strips. Anodic oxidation of palladium hydride was electrochemically induced, and stress measurements were made in situ using this approach. Internal stress in tungsten oxide and nickel oxide thin films was determined by F.Decker et al. [24] using laser beam deflection method during electrochromic research of these materials. Electrochromic tungsten oxide thin film electrode stress changes were recorded by D.Dini et.al. [25] using laser beam deflectometry to study the intercalation process.

To quantify substrate deflection and calculate film internal stress, we used the DEHI approach in the current study. There are several reasons why thin film stress parameters can be determined using this method. More accurate data on substrate deflection can be gleaned from this method.

The number of fringes in the hologram is used to determine the deflection of the substrate. Table No. 1 shows the deflections of the substrate as a result of employing Eq. (3).

Fringes appear more frequently on stainless steel plates as the duration of deposition increases, according to the holographic investigation, which is shown in Table 1 of the results. Thickness and mass deposited on the substrate increase as a result. Other two concentrations of (SrCl2 + Na2S2O3) solution, namely 0.1 and 0.15M, yielded similar findings.For (SrCl2 + Na2S2O3) solutions of 0.05,0.15, and 0.15M concentrations, the mass deposition rate on the substrate was determined at 0.0010,0.0016, and 0.002 mg/s using Fig.4. This shows that the rate of depositions increases with the concentration of solution and that this increase was quite minor.

The inherent stress values for varied deposition times were calculated using Eq.(1). As previously mentioned, these numbers can be seen in the same Table 1. The table demonstrates that as deposition time increases, the substrate fringes grow and the thin film stress increases as well. Figure 5 depicts the relationship between computed stress and thickness for a SrS thin film.

The graph shows that as the film thickness grows, the tension on the substrate also rises. According to Cambell et al. [26], the nature of the curve demonstrates that films expand under tensile stress and that stress value is dependent on film thickness.

6. CONCLUSIONS

Strontium sulphide films were prepared by cathodic electrodeosition potential from aqueous acidic bath. The estimated potential was -0.1V versus V_{SCE}. This reveals that films were polycrystalline in nature, according to the XRD pattern. Based on d-values, it was confirmed that films were made of SrS.

The thickness of SrS films and the mass deposited at various concentrations were determined using the DEHI technique.From that deflection of the substrate and intrinsic stress of film were easily determined. This study helps one to prove as the time of deposition for thin film increases, number of fringes on substrate increases consequently the deflection of substrate increases and hence the intrinsic stress of the film also increases.

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Table No.1 - Observed Data for SrS Thin Films

1) For 0.05 M solution of ($SrCl_2$ + $Na_2S_2O_3$)

Time of Deposition t sec	No. of Fringes n	Thickness t_f A^0	Mass of SrS Deposited mg	Deflection of substrate δ A^0	Calculated Stress S x 10^{10} dyne/cm^2
40	4	1247.40	0.15	12656	0.608754
70	5	1496.88	0.18	15820	0.634118
100	6	1746.36	0.21	18984	0.652236
130	8	1995.84	0.24	25312	0.760942

2) For 0.1M solution of ($SrCl_2$ + $Na_2S_2O_3$)

Time of Deposition t sec	No. of Fringes n	Thickness t_f A^0	Mass of SrS Deposited mg	Deflection of substrate δ A^0	Calculated Stress S x 10^{10} dyne/cm^2
40	4	1330.56	0.16	12656	0.570707
70	5	1496.88	0.18	15820	0.634118
100	7	1995.84	0.24	22148	0.665824
130	11	2328.48	0.28	34804	0.896825

3) For 0.15 M solution of ($SrCl_2$ + $Na_2S_2O_3$)

Time of Deposition t sec	No. of Fringes n	Thickness t_f A^0	Mass of SrS Deposited mg	Deflection of substrate δ A^0	Calculated Stress S x 10^{10} dyne/cm^2
40	5	1496.88	0.18	15820	0.634118
70	6	1746.36	0.21	18984	0.652236
100	8	2245.32	0.27	25312	0.676393
130	13	2577.96	0.31	41132	0.957312

FIGURE CAPTIONS

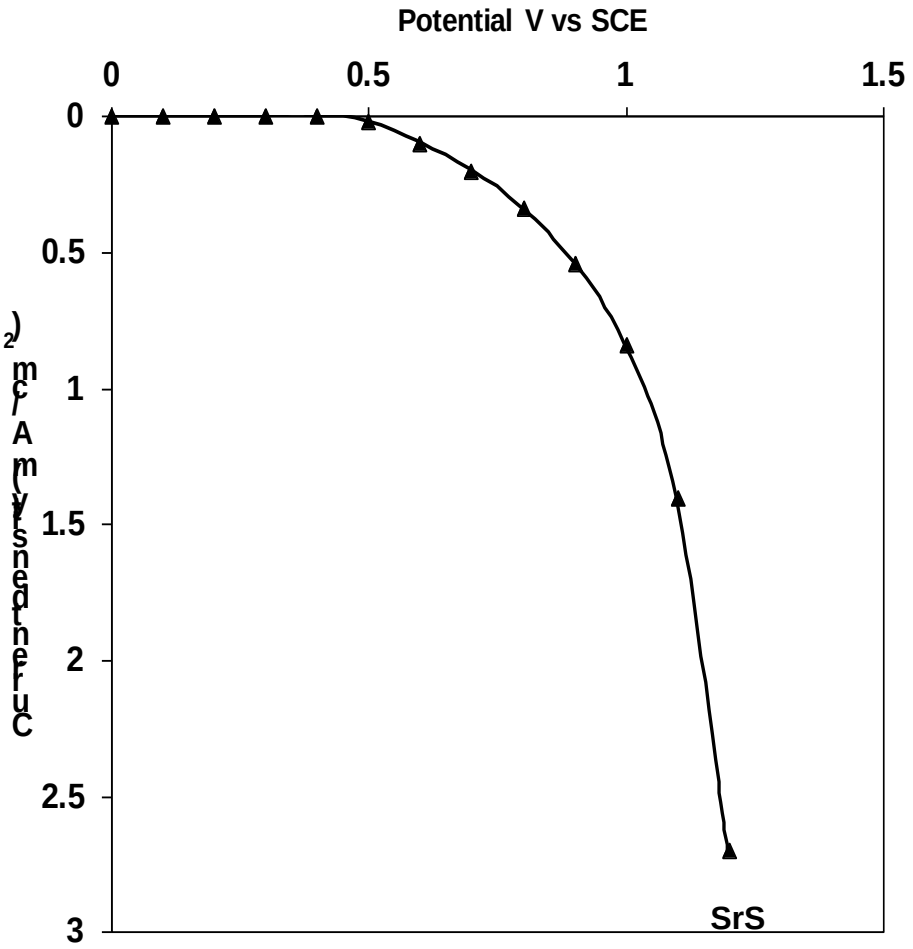
Fig.1 The cathodic polarization curve for a bath containing 0.1M ($SrCl_2$+$Na_2S_2O_3$) solution with stainless steel substrate.

Fig. 2 X-ray diffraction of strontium sulphide thin film.

Fig. 3 Reconstructed images of substrate from holograms for different moralities (M) of ($SrCl_2$ + $Na_2S_2O_3$) solutions and different deposition times with number of fringes n for: (a) 0.05M, 40s, n = 4; (b) 0.05M, 70s, n =5; (c) 0.05M, 100s, n = 6; (d) 0.05M, 130s, n = 8; (e) 0.1M, 40s, n = 4; (f) 0.1M, 70s, n =5; (g) 0.1M, 100s, n = 7; (h) 0.1M, 130s, n = 11; (i) 0.15M, 40s, n = 5; (j) 0.15M, 70s, n =6; (k) 0.15M, 100s, n = 8; (l) 0.1M, 130s, n = 13.

Fig. 4 The plot of mass of SrS versus time of deposition.

Fig. 5 The plot of stress of SrS thin film versus thickness of film.


Potential V vs SCE
0
0.5
1
1.5
0
0.5
1
1.5
2
2.5
3
SrS

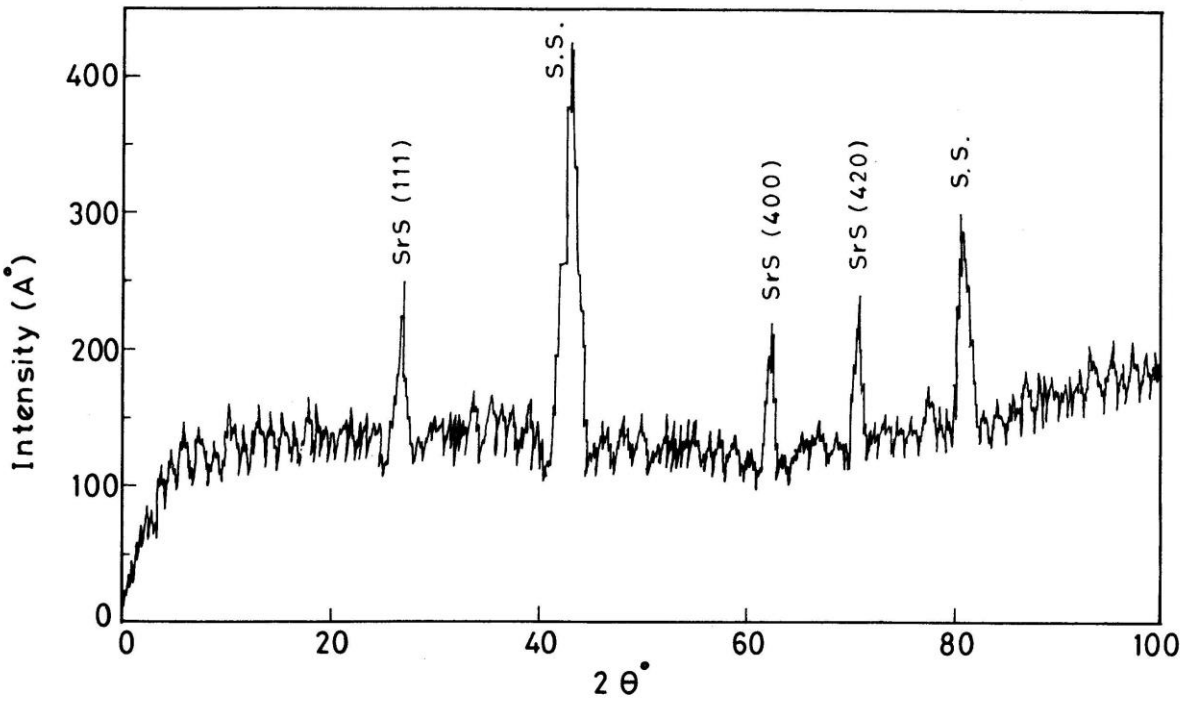
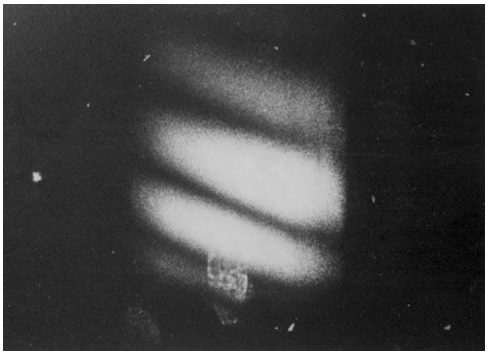
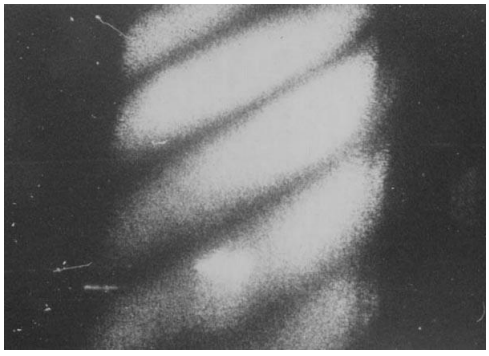


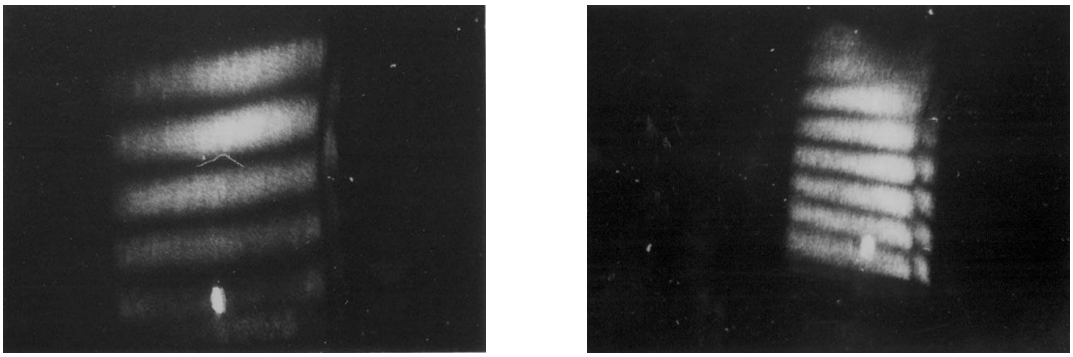
Fig 2 - X-ray diffraction of SrS thin film.



(a)

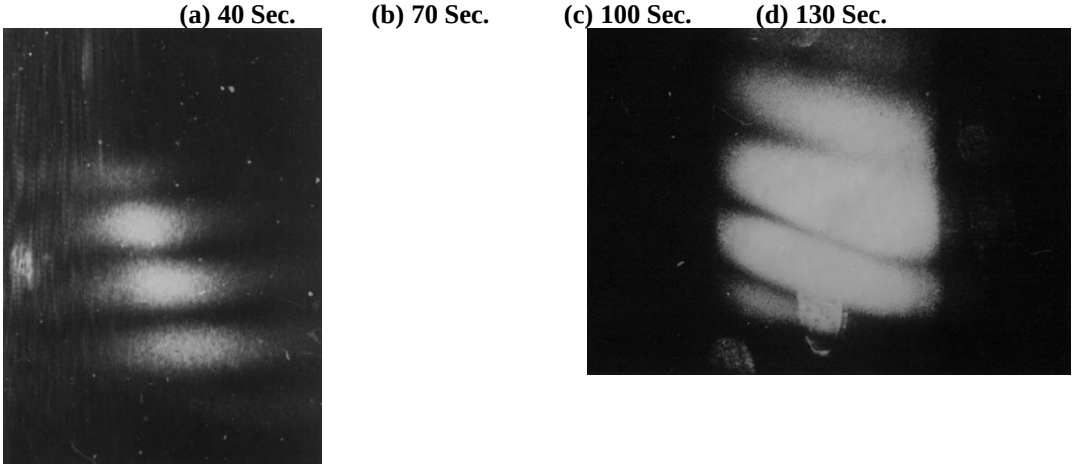


(b)

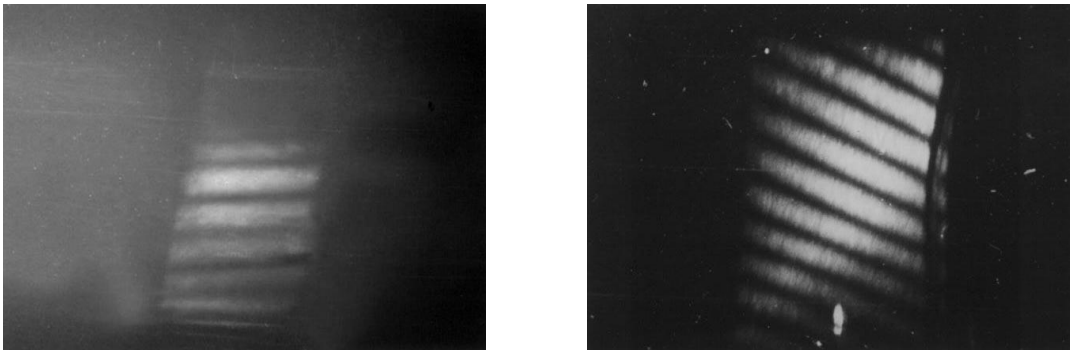


(c) **(d)**

Fig 3: (i) Photographs of Interferograms of SrS Thin Film (0.05M)
(a) 40 Sec. (b) 70 Sec. (c) 100 Sec. (d) 130 Sec.

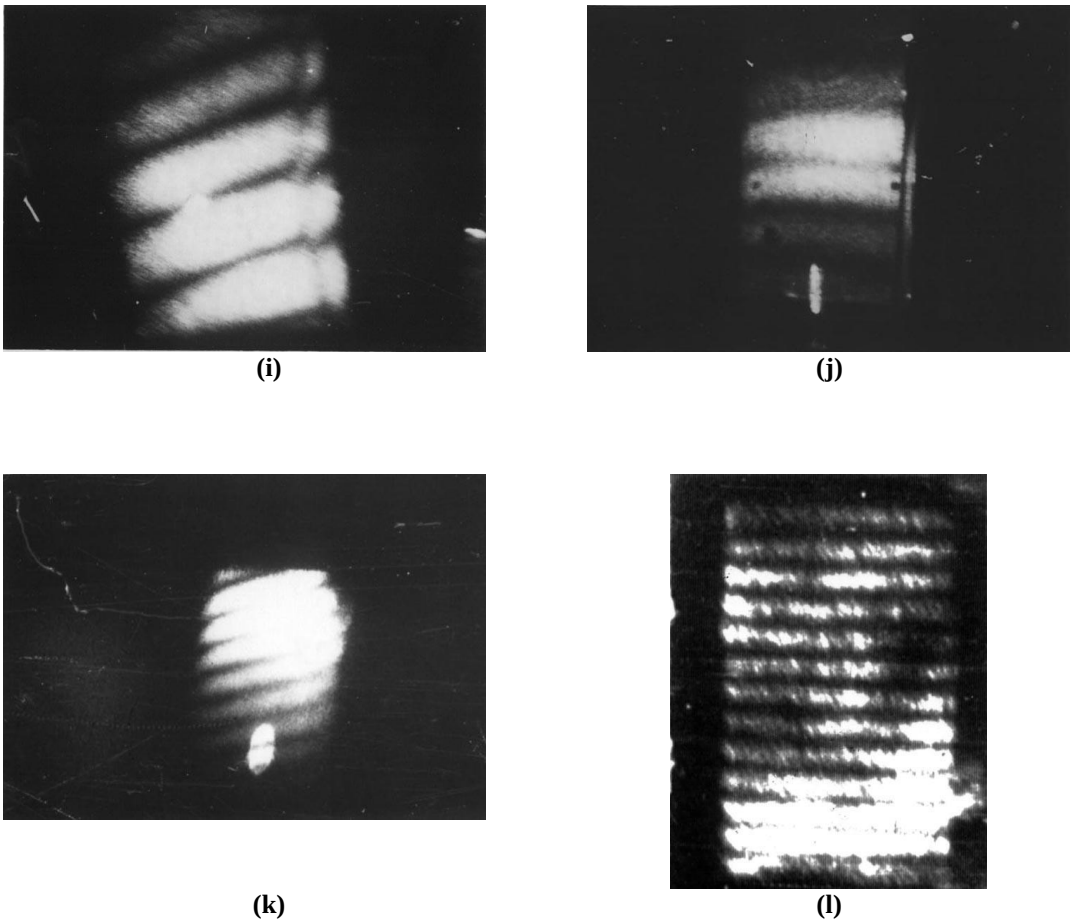


(e) **(f)**



(g) **(h)**

Fig 3 : (ii) : Photographs of Interferograms of SrS Thin Film (0.1M)
(e) 40 Sec. (f) 70 Sec. (g) 100 Sec. (h) 130 Sec.



(i) **(j)**

(k) **(l)**

Fig 3 : (iii):Photographs of Interferograms of SrS Thin Film (0.15M)
(i) 40 Sec. (j) 70 Sec. (k) 100 Sec. (l) 130 Sec.

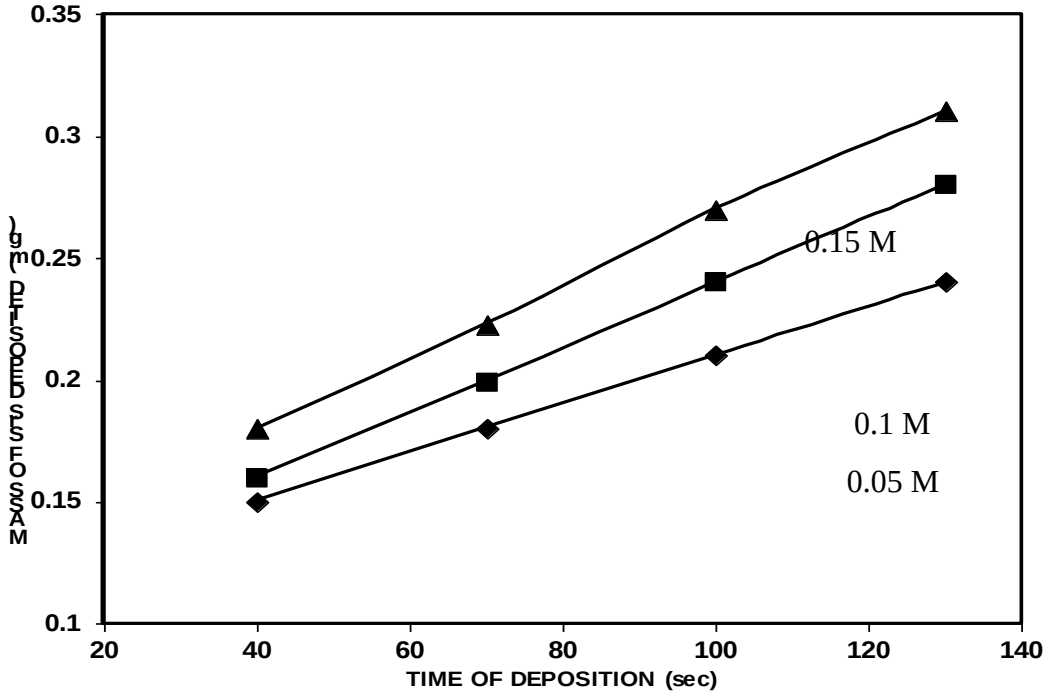


Fig. 4 : Varation of mass with time of deposition.

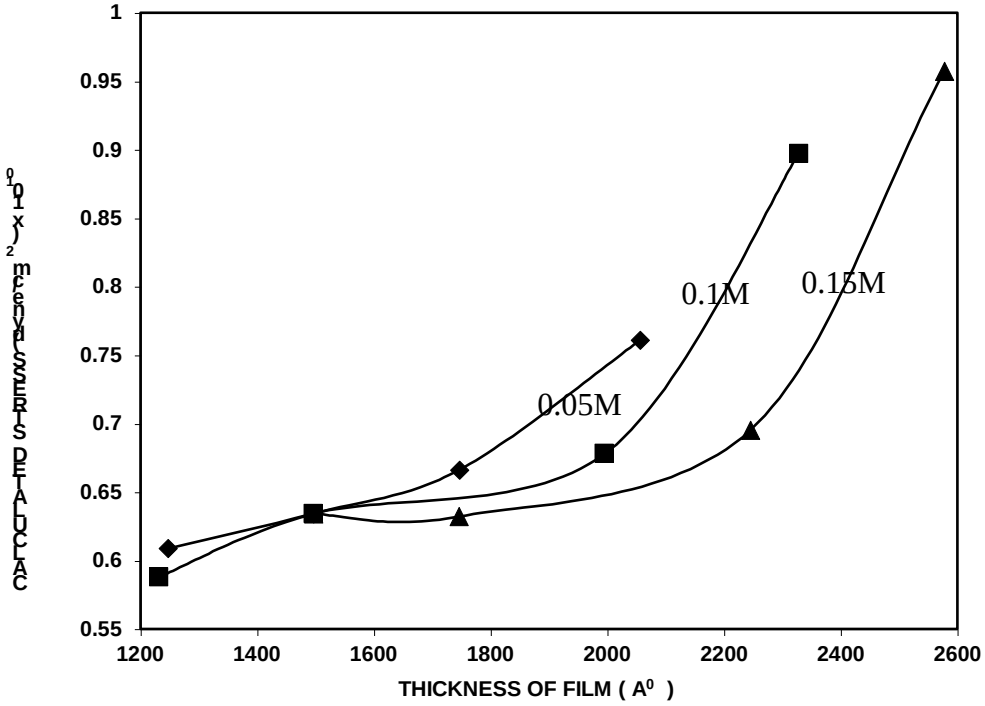


Fig.5: The Plot Of Calculated Stress Versus Thickness Of Film.