

C H A P T E R - II

THEORETICAL BACKGROUND : CHEMICAL DEPOSITION/PHOTOCONDUCTIVITY

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2.1 Introduction :

The various techniques for the deposition of CdS thin films and the advantages of chemical bath deposition over other deposition techniques are described in the previous chapter-I. The CdS thin films have been prepared by chemical bath deposition technique as reported by Pavaskar et al (1), for the study of photoconductivity properties. Chapter-II 2.2A consists the theoretical details of the chemical bath deposition and 2.2B deals with some aspects of photoconductivity.

2.2A Theoretical Background of Chemical deposition :

2.2A.1 Solubility Product and Ionic Product :-

When a sparingly soluble salt AB is placed in water, a saturated solution containing A and B ions in contact with the undissolved solid AB is obtained and an equilibrium is established between the solid phase and the ions in the solution. Thus



Applying law of mass action to this equilibrium

$$K = \frac{C_{A^{+}} \cdot C_{B^{-}}}{C_{AB(S)}} \quad \text{----- 2.2}$$

Where $C_{A^{+}}$, $C_{B^{-}}$ and $C_{AB(S)}$ are concentrations of A, B and AB in the solution respectively.

The concentration of a pure solid phase is a constant number,

i.e. $C_{AB(S)} = \text{constant} = K'$

$$K = \frac{C_{A^+} \cdot C_{B^-}}{K'} \quad \dots\dots \quad \dots\dots 2.3$$

or $KK' = C_{A^+} \cdot C_{B^-} \quad \dots\dots \quad \dots\dots 2.4$

As K and K' are constant, the product KK' is also constant, say K_S , therefore equation (2.4) becomes

$$K_S = C_{A^+} \cdot C_{B^-} \quad \dots\dots \quad \dots\dots 2.5$$

The constant K_S is called as the solubility product and expression $C_{A^+} \cdot C_{B^-}$ is called as the ionic product. When the solution is saturated, the ionic product is equal to the solubility product. It follows that when the ionic product exceeds the solubility product, the solution is supersaturated and precipitation occurs. When the ionic product is less than the solubility product, the solution will be unsaturated. Here temperature, solvent and particle size are three main factors which affect the solubility product (2-5).

2.2A.2 Precipitate formation in the Solution :

The formation of a solid phase from a solution involves two steps; as nucleation and particle growth. The size of the particles of a solid phase is dependent upon the relative rates at which these two competing processes takes place. It also depends on temperature, rate of mixing reagents, concentration of reagents and the solubility of the precipitate during precipitation.

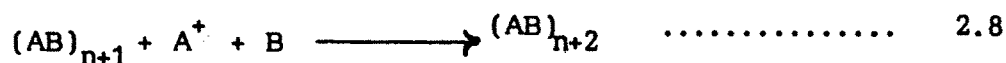
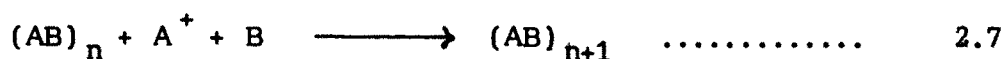
All of these can be related to the relative supersaturation of the system. A state of supersaturation may be achieved by lowering the temperature of an unsaturated solution and formation of these solute in solutions at a fixed temperature.

For any precipitate, there is some minimum number of ions or molecules required to produce a stable second phase in contact with a solution called a nucleus. The rate at which nuclei form in a solution is dependent on the degree of supersaturation. In highly supersaturated solution, the rate of nucleation increases exponentially :

$$\text{Rate of nucleation} = K_0 (Q-S)^X \quad \dots\dots\dots 2.6$$

Where K_0 and X are constants and $X > 1$, Q is the excess concentration and S is the concentration of saturation.

The second step is the growth of the particles already present in the solution. This begins when nuclei or other seed particles are present. In case of an ionic solid the process involves deposition of cations and anions on appropriate sites.



Where n is the minimum number of A and B ions that must combine in order to yield the stable particle $(AB)_n$. The rate of growth is directly proportional to the supersaturation :

$$\text{Rate of growth} = K_0' A (Q-S) \quad \dots\dots\dots 2.9$$

Where A is the surface area of the exposed solid, K_0 is a constant that is characteristic of the particular precipitate. If the supersaturation is maintained at low level throughout the precipitation, the relatively few nuclei formed will grow to give a small number of large particles. With high supersaturation, many more nuclei are formed initially and nucleation may occur throughout the entire precipitation process. As a result there are many more centres upon which the growth process can take place, none of the particles can become very large and a colloidal suspension is formed.

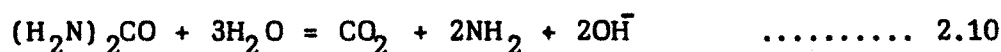
The colloidal suspension consists of finely divided solid particles in a liquid phase with diameters of 0.01 to 0.1 micron. Under some circumstances colloidal particles can come together and adhere to one another and the resulting solid is called a colloidal precipitate and process by which it is formed is termed coagulation or agglomeration. Colloidal particles when agglomerated have quite different properties from a crystalline solid since particles are arranged irregularly.

2.2A.3 Controlled Precipitation in the Solution :

The degree of supersaturation can be reduced by the slow, dropwise addition of the reagent and by the use of dilute solutions of both ions to be precipitated and the reagent. In precipitation from homogenous solution, the precipitating agent is chemically generated in the solution. Local reagent excesses does not occur because of the precipitating agent appears slowly and

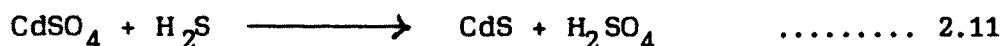
homogeneously throughout the entire solution; the relative supersaturation is thus kept low.

Urea is often employed for the homogeneous generation of hydroxide ion. The reaction can be expressed by the equation



This reaction proceeds slowly at temperatures just below the boiling point and typically 1 to 2 hours are needed to produce sufficient reagent for the completion of a precipitation. Homogenous precipitation of crystalline precipitates also results in marked increase in crystal size as well as improvement in purity.

Controlled precipitation can be obtained when a solution reacts with reacting gases like NH_3 or H_2S (6-9). When a gas is bubbled through the bulk of the solution, precipitation is obtained while if the solution is slowly exposed to the gas a thin solid film is obtained. The thickness and quality of films can be controlled by optimizing the preparative parameters. A typical reaction involved in precipitation of CdS film by reaction of H_2S gas with CdSO_4 solution is as follows :

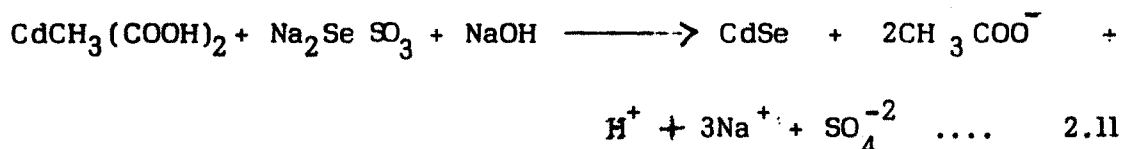


2.2A.4 Growth of thin films by the bulk precipitation in the solution :

Thin films of metal chalcogenides are obtained by bulk precipitation of solution. The reaction of metal solution takes place with an aqueous solution of a compound capable of giving

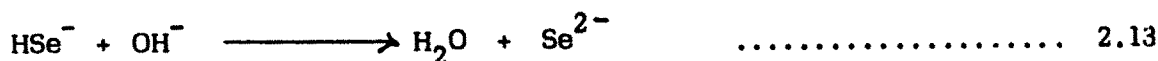
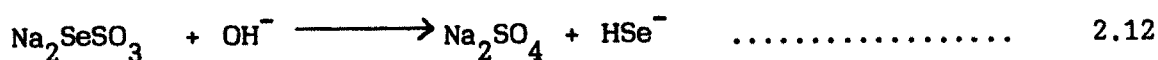
chalcogenide ions under suitable conditions. Thiourea, Thioacetamide or sodium selenosulphate are the commonly used compounds which furnish sulphide or selenide ions respectively by hydrolysis in alkaline media (1,10-12).

As a representative of the procedure, the deposition of CdSe is considered (1). Appropriate amounts of cadmium salt and ammonia solutions are mixed together and the desired pH is adjusted. To this, sodium selenosulphate is added and substrates are dipped in the solution which is heated to the desired temperature with continuous stirring of the solution. The overall chemical reaction is as follows :



The films are grown by ion by ion or the cluster by cluster mechanism depending upon the preparative conditions. The reaction mechanism and growth kinetics of CdSe film formation is proposed as :

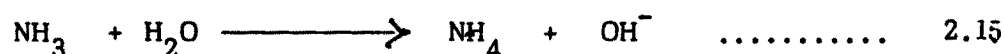
Sodium selenium sulphate (Na_2SeSO_3) hydrolyzes in alkaline solution to give Se^{2-} ions according to



In the presence of cadmium ions (Cd^{2+}) in the solution, CdSe will be formed if the ionic product of Cd^{2+} and Se^{2-} exceeds the solubility product of CdSe.



In the present case, ammonia hydrolyzes in water to give OH^- according to



With hydrolysis constant $K_{\text{hyd}} = 1.8 \times 10^{-5}$.

When ammonia solution is added to Cd salt solution, $\text{Cd}(\text{OH})_2$ starts precipitating when the solubility product (SP) of $\text{Cd}(\text{OH})_2$ is exceeded i.e.



$$\text{With SP} = [\text{Cd}^{2+}] [\text{OH}^{-2}] = 2.2 \times 10^{-14} \quad \dots\dots\dots 2.16$$

The $\text{Cd}(\text{OH})_2$ precipitate dissolves in excess ammonia solution to form the complex cadmium tetra-amine ion



The solubility constant of $\text{Cd}(\text{NH}_3)_4^{2+}$, $\text{PK} = 7.12$, i.e.

$$\frac{[\text{Cd}^{2+}] [\text{NH}_3]^4}{[\text{Cd}(\text{NH}_3)_4^{2+}]} = 7.56 \times 10^{-8} \quad \dots\dots\dots 2.18.$$

Conversion to logarithmic concentration and elimination of $\text{P}[\text{Cd}^{2+}]$ from equation (2.16) and (2.17) leads to

$$\text{pH} = 10.73 + 1/2 \text{ P}[\text{Cd}(\text{NH}_3)_4^{2+}] - 2\text{P}[\text{NH}_3]$$

If excess ammonia is added to dissolve the $\text{Cd}(\text{OH})_2$ Precipitate, then $\text{P}[\text{Cd}(\text{NH}_3)_4^{2+}]$ can be approximated by PC salt, hence

$$\text{pH} = 10.73 + 1/2 \text{ PC salt} - 2\text{P}(\text{NH}_3)$$

In the present case, $PC \text{ salt} = -\log (4.8 \times 10^{-2}) = 1.3$

therefore, $pH = 11.38 - 2P(NH_3)$ 2.19

Equation (2.19), when plotted, is a straight line (A) as shown in figure 2.1. Points lying on the line and in the region I represents a homogeneous system consisting of stable $Cd(NH_3)_4^{2+}$ complex in equilibrium with Cd^{2+} ions. In region II, which can be reached by the addition of an alkali to the solution, conditions for the spontaneous formation $Cd(OH)_2$ exist so that the points lying in the region II represent a heterogeneous system consisting of the complex $Cd(NH_3)_4^{2+}$ and a solid phase of $Cd(OH)_2$ simultaneously in equilibrium with Cd ions. CdSe films have been obtained in the region II for conditions corresponding to three points on the line A. In the heterogeneous system of $Cd(OH)_2$ and $Cd(NH_3)_4^{2+}$, the region enclosed by the two dotted lines B and C represents conditions for obtaining films of measurable thickness.

Similar type of ion-by-ion growth reaction mechanisms have proposed for PbSe, CdS, PbS and HgS (12,13). According to Lundin and Kiteav (11) nucleation on substrates takes place by adsorption of the colloidal particles and growth takes place as a result of surface coagulation of these particles, resulting in thin and adherent films. This is known as 'Cluster-by-Cluster' growth of a film.

The growth mechanism of CdS films from aqueous solutions of Cadmium Sulphate and Thiourea in alkaline medium is first suggested by Lundin (14).

The processes involved are :

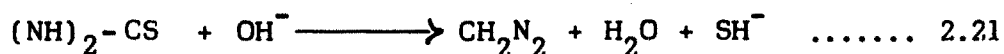
i) Formation of quaternary complex compound

Cadmium sulphate reacts with aqueous ammonia to form cadmium tetra ammonium sulphate.

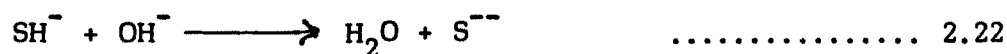


ii) Diffusion of the complex ion, OH^- and Thiourea to the catalytic surface of CdS.

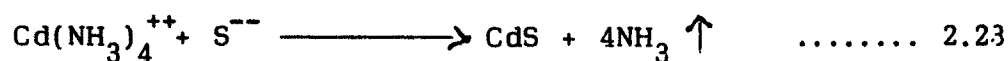
iii) Dissociation of the Thiourea on the catalytic CdS surface in alkaline medium.



iv) Bivalent sulphide ion formation

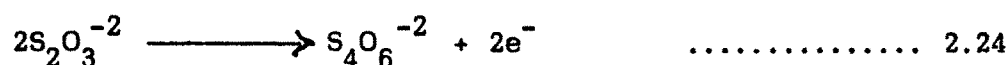


v) Formation of CdS

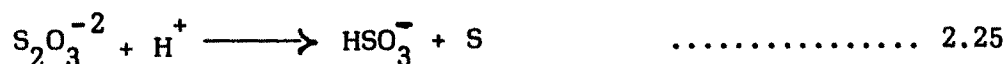


The growth of some chalcogenide thin films from acidic baths using $\text{Na}_2\text{S}_2\text{O}_3$ as a sulphur source has been reported (15-17). For example, the reaction mechanism for SnS_2 deposition is proposed as follows :

$\text{Na}_2\text{S}_2\text{O}_3$ is a reducing agent by virtue of the half cell reaction as,



In the acidic medium, the dissociation of $\text{Na}_2\text{S}_2\text{O}_3$ takes place :



The released electrons from (2.24) react with sulphur released from (2.25) as :



and formation of SnS_2 takes place as :



2.2A.5 Effect of preparative parameters :

2.2A.5.1 Effect of pH :

The reaction rate and the rate of deposition depend on the supersaturation : the lower the supersaturation, the slower the formation of MX. If the concentration of OH^- ion in the solution is higher, the M ion concentration will be lower and the reaction rate will be slow. With an increase in pH as the M ion concentration decreases, the rate of formation of MX is decreased. At a certain pH the concentration of M ions decreases to a level such that the ionic product of M and X becomes less than the solubility product of MX and a film will be formed.

2.2A.5.2 Effect of complexing agent :

The metal ion concentration decreases with increase in concentration of complexing ions. Consequently, the rate of reaction and hence precipitation is reduced leading to a larger terminal thickness of the film.

2.2A.5.3 Effect of temperature :

The dissociation of the complex and the anion of the compound [X compound] depends on the temperature. At higher temperatures, the dissociation is greater and gives higher concentrations of M and X ions which result in higher rates of deposition.

The thickness increases with increase in the bath temperature depending on the conditions under which films are prepared. At low pH values, supersaturation is high even at low temperature and increases further with increasing temperature. This results in formation of precipitation and consequently lower thickness is obtained. At high pH values, precipitation is limited due to the low supersaturation and most of the product is formed on the substrate surface. Further the thermal dissociation of complex and anion compound is increased at higher temperatures so that more M and X ions are available for MX formation and thus higher thickness is obtained.

2.2A.5.4 Effect of anion compound [X compound] concentration :

The increase in X compound concentration leads to an increase in X ion concentration and a film of MX with larger thickness is obtained. However, above a certain concentration when rate of reaction becomes high and precipitation also becomes important leading to a lesser amount of MX on the substrate and hence lower thickness.

2.2A.5.5 Effect of substrate :

Film formation can take place only under certain conditions i.e. either under optimum conditions for MX formation or when the substrate has special properties facilitating for formation of single crystal films. The second condition favours the formation because when the lattice of the deposited material matches well with that of the substrate the free energy change is smaller, thereby facilitating nucleation.

2.2A.5.6 Effect of doping :

Impurities in the starting materials can be incorporated in to the films only if the impurities form insoluble chalcogenides under the same conditions of deposition and provided their corresponding ionic product is greater than the solubility product. The doping affect film thickness and properties.

2.2B Theoretical Background of Photoconductivity :

2.2B.1 General Mechanism :

All the materials are classified in to three groups viz, metals, semiconductors and insulators depending on their width of forbidden energy band. Electrical conductivity can arise only from these electrons which are excited from the filled band to the conduction band. For the formation of free charge carriers in a semiconductor, energy is required to overcome the energy gap that is between allowed band or local impurity levels and the filled band. Under normal conditions, this energy is drawn from the

thermal energy present in the crystal. The free electrons (n) and holes (p) liberated due to thermal ionization are called equilibrium carriers and thermal electrical conductivity for n-type semiconductor is given by relation.

$$\sigma = ne\mu_n \dots\dots\dots 2.28$$

In contrast to thermal energy, energy used to generate the excess carriers is retained by electrons, and average thermal energy of the crystal lattice remains practically unaffected. Consequently, the thermal equilibrium between the lattice and electrons is disturbed. The carriers formed in some way other than thermal ionization are called 'non-equilibrium' carriers. Thus there is lattice difference in equilibrium carrier densities. The total density is the sum of equilibrium and non-equilibrium carrier densities. As a result, equation for conductivity becomes :

$$\sigma = e (n \cdot \mu_n + \Delta n \cdot \mu_n) \dots\dots\dots 2.29$$

The change in conductivity $\Delta\sigma$, due to non-equilibrium density is, therefore, gives as $\Delta\sigma = e (\Delta n \cdot \mu_n)$ assuming that mobilities of equilibrium and non-equilibrium conductivity appears as a result of illumination, it gives rise to photoconductivity due to electron transitions from the valance band to the conduction band by the absorption of light quanta. In general, when mobility also changes by the illumination, the change in conductivity i.e. photoconductivity

$$\Delta\sigma = \Delta ne\mu_n + ne\Delta\mu_n \dots\dots\dots 2.30$$

Since the number of free carriers is related to the excitation rate

F and the free carrier life-time τ_n by

$$n = F \cdot \tau_n \dots\dots\dots 2.31$$

and $\Delta n = \Delta F \cdot \tau_n + F \cdot \Delta \tau_n$

Therefore, $\Delta \sigma = e \mu_n \tau_n \Delta F + e \mu_n F \Delta \tau_n + e R \tau_n \Delta n \dots\dots\dots 2.32$

Thus, the change in photoconductivity can be related to three factors :

- i) $e \mu_n \tau_n \Delta F$, which simply relates to the change in excitation rate which is obviously the concept of photoconductivity.
- ii) $e F \tau_n \Delta \mu_n$ relates to mobility changes that results from an increase in the density of ionized impurity scattering centers; inhomogeneities of crystal potential or transitions from lower to higher mobility states.
- iii) $e \mu_n F \Delta \tau_n$ which means the change in photoconductivity is because of change in carrier life time. A positive $\Delta \tau$ leads to an increase in sensitivity and hence a superlinear dependance of photocurrent on excitation intensity.

2.2B.2 Life Time :

The life time of photoexcited carriers is the key parameter to understand the photoconductivity (18). If light falling on a photoconductor creates electron-hole pairs per second per unit volume of the photoconductor,

then,

$$F \tau_n = \Delta n \dots\dots\dots 2.33$$

$$f\tau_p = \Delta P \quad \dots\dots\dots 2.34$$

Where τ_p is the life time of an electron, τ_p is the life time of hole, and Δn and ΔP are respectively the additional free electron and hole densities present as a result of absorption of light.

The following types of 'life-times' must be distinguished :

- 1) Free life-time : The free life-time is the time that the charge carrier is free to contribute to the conductivity. It is the time that an excited electron spends in the conduction band, or the time that an excited hole spends in the valance band. The τ_n and τ_p are the free life-times.
- 2) Excited life-time :- The excited life-time is the total time the carrier is excited between the act of excitation and the act of recombination. The excited life-time includes any time that the carrier may spend in traps; it is therefore usually larger than the free life time.
- 3) Pair Life-time :- The pair life time is the free life-time of an electron-hole pair. If either electron or hole is captured, the pair life-time is terminated.

The usual termination of the life time of free carrier is by recombination. This recombination may take place in various ways, one is direct recombination between free electrons and holes. More often recombination takes place after one of the carrier has been captured by discrete states in the forbidden gap. These states are due to imperfections in the lattice such as impurities, vacancies or dislocations. Many of the most important photoconductivity

properties are associated with the effects of imperfections in crystals. One of the common types of imperfection is the impurity imperfection which enhances the activation of a photoconductor.

2.2B.3 Photo-Sensitivity :

When the term photosensitivity is used it means photoconductivity per unit excitation intensity i.e. change in conductivity caused by excitation, divided by excitation intensity.

2.2B.4 Photoconductivity gain, G :

The fundamental equation expressing the gain of a photoconductor is (19).

$$\text{Gain} = \frac{\text{Number of charges passed through crystal}}{\text{Number of photons absorbed}} \quad \dots\dots 2.35$$

Excitation produces a free electron and a hole rapidly trapped. The free electron moves through the crystal, under the influence of the field, to the positive electrode, and hence out of the crystal. At the time that an electron moves out of the crystal at the positive electrode, another electron enters the crystal from the negative electrode. Charge continues to flow the crystal until recombination occurs between the trapped hole and a free electron. Gain much larger than unity have been observed by this mechanism.

The gain may be expressed in terms of the life time of a free electron, τ_n , and the transit time of an electron between electrodes, T :

$$\text{Gain} = \frac{\tau_n}{T} \quad \dots\dots\dots 2.36$$

If T is expressed in terms of mobility, μ , the applied voltage, V , and the distance between the electrodes, L , equation (2.36) for the gain, G , may be converted to :

$$\text{Gain} = \frac{\tau_n \mu_n V}{L^2} \dots\dots\dots 2.37$$

2.2B.5 Photocurrent :

Photocurrent I_L is derived from equation (2.31) and is given by the relation.

$$I_L = \frac{ne}{\tau} = eF \frac{\tau_n}{T} \dots\dots\dots 2.38$$

The transit time T , (time required for the carrier to move between the electrodes) is given by the relation,

$$T = \frac{L}{V_d} = \frac{L^2}{V\mu_n} \dots\dots\dots 2.39$$

Where, V_d = drift velocity

V = the applied voltage, and

L = spacing between the electrodes.

Substituting equation (2.39) in (2.38), we get the photocurrent (20) as :

$$I_L = \frac{eF \tau_n \mu_n V}{L^2} \dots\dots\dots 2.40$$

From equation (2.40), the photocurrent should increase linearly with applied voltage assuming that neither life time nor the mobility is voltage dependent.

2.2B.6 Spectral Response :

The photoresponse of the material depends on the

energy of the light falling on it. The maximum photoresponse occurs at the wavelength λ_g , corresponding to the absorption edge of the material. Characteristically as one proceeds from lower energy side (long wavelength) to the higher energy side (short wavelength), the photocurrent starts from zero, rises rapidly as λ approaches to λ_g , reaches maximum, and falls again to some asymptotic value greater than zero. This is shown in Figure 2.2. The decrease in photocurrent with more energetic photons is a surface effect. For the radiation of $\lambda < \lambda_g$, the solid becomes highly absorbing giving rise to the high density of carriers at the surface. This enhances the recombination of electrons and holes, removing carriers more rapidly than the bulk of the sample.

The photoconductivity may be produced by wavelengths λ , greater than that of the absorption edge, λ_g as shown in figure 2.2. This has been originated due to the electrons promoted from impurity levels existing in the band.

2.2B.7 Time-constant :

The time constant is given by the relation given by Bhar and Linder (21).

$$\text{Time constant} = \frac{1}{RM} \quad \dots\dots\dots 2.41$$

Where R is the recombination coefficient.

2.2B.8 Rise and decay time constant :

The rise and decay time constants, t_r and t_d defined as the time required for the photocurrent to rise to 63% of the stationary value or to decay to the same percentage of the preliminary value (22).

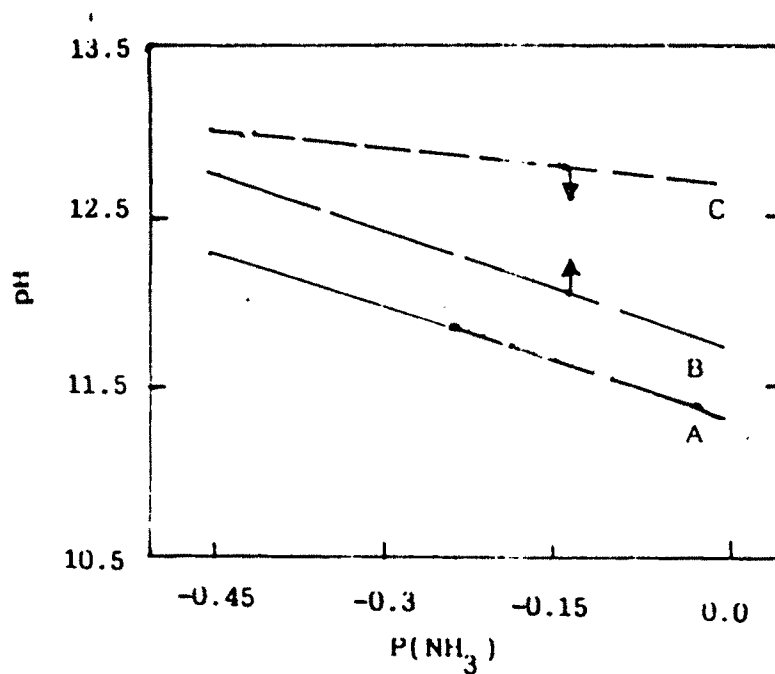


Fig.2.1 : A plot of $P(\text{NH}_3)$ using equation 2.19

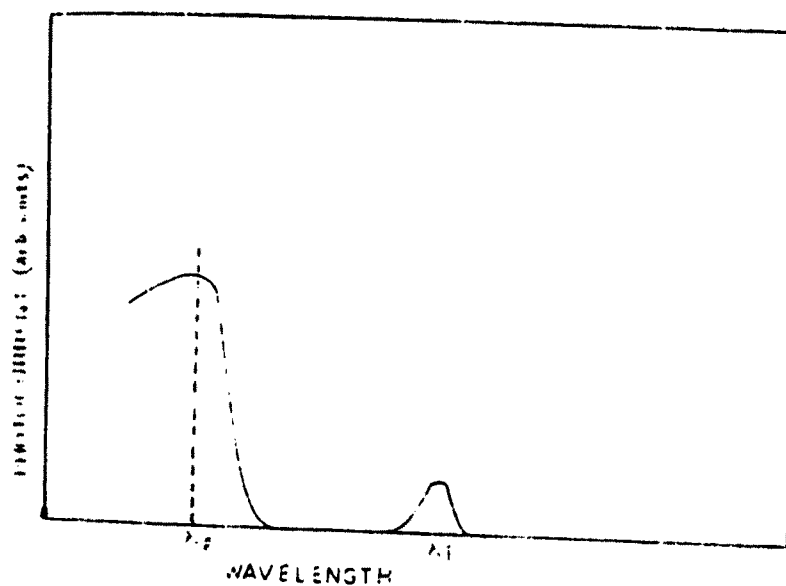


Fig. 2.2 : The variation of photocurrent with wavelength doe a typical photoconductor.

-: R E F E R E N C E S :-

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