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“IMPROVEMENTS IN OR RELATING TO CHEMICAL DEPOSITION OF THIN FILMS OF LEAD SELENIDE LAYERS”

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INDIA, AN INDIAN REGISTERED BODY INCORPORATED UNDER THE REGISTRATION  
OF SOCIETIES ACT (ACT XXI OF 1860).

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PROVISIONAL

*The following Specification describes the nature of this invention.*

This invention relates to improvements in or relating to chemical deposition of thin films of lead selenide layers which when processed can be used as infra-red detectors in the range 3-7 microns.

Hitherto it has been proposed to deposit these thin films either by vacuum deposition or by chemical deposition. This is open to the objection that conditions of deposition are governed by several critical factors and apart from involving costly vacuum coating apparatus reproducibility is difficult. The hitherto known chemical deposition is open to the objection that seleno-urea, by the interaction of which with lead acetate solution in alkaline or hydrazine medium produced the chemical deposits, is prohibitively costly and has to be imported.

The object of this invention is to obviate these disadvantages by simplifying the method by using a comparatively very inexpensive chemical method wherein no sophisticated equipment is necessary at all.

To these ends, the invention broadly consists in taking a mixture of lead acetate and selenium dissolved in sodium sulphite and then adjusting the pH to be on the alkaline side in the range of 8-10 to get the desired film thickness of 1-2 microns of lead selenide on the glass substrate. Ammonia and hydrazine have also been found to be useful in getting good uniform and adherent deposits.

The following typical examples are given to illustrate the invention:

**Example 1**

Deposition bath consists of about 50 c.c. of approximately 40% lead acetate, an equal volume of 1% selenium in sodium sulphite solution of about 20% strength. The ground glass

plates affixed to a perspex holder are immersed in the bath and then the contents are stirred. Fairly concentrated ammonia or hydrazine are added to the solution containing selenium in sodium sulphite solution. The plates are allowed to remain inside the solution for about two hours. Good adherent deposits are obtained. On heating to a temperature around 300°C, these thin films show photoconductivity in the infrared region.

**Example 2**

Deposition bath consists of about 50 c.c. of approximately 5% lead acetate, an equal volume of 1% selenium in sodium sulphite solution of about 20% strength. The ground glass plates affixed to a perspex holder are immersed in the bath and then contents are stirred. The pH of the solution is adjusted to be in the range of 8-10 by adding sodium hydroxide. The plates are allowed to remain inside the solution for about 2 hours. Good adherent deposits are obtained. On heating to a temperature around 300°C, these thin films show photoconductivity in the infra-red region.

The following are the main advantages of the invention:

- (1) No costly equipment is needed.
- (2) Method of deposition is chemical and avoids seleno-urea, a prohibitively costly chemical to be imported from abroad used so far in the only known method of chemical deposition in which seleno-urea and lead acetate are reacted in alkaline medium.
- (3) Method of deposition is very easy and reproducible and can be done at laboratory temperature.
- (4) Control of the bath for introducing dopants etc., will be easy.

COMPLETE

*The following Specification particularly describes and ascertains the nature of this invention and the manner in which it is to be performed.*

This invention relates to improvements in or relating to chemical deposition of thin films of lead selenide layers.

It has particular reference to chemical deposition of infrared sensitive lead selenide layers useful as a solid state device for detecting infrared sources in the wave length range of 3-7 microns.

Prior knowledge:

Hitherto these sensitive layers could be obtained by vacuum deposition or by chemical deposition using seleno-urea.

Drawbacks connected with hitherto known processes/devices:

In the vacuum deposition technique, conditions are governed by several critical factors (the degree of vacuum, the nature of the heating element, the current to be passed through the heating elements which in turn depends upon the length of the heating element-wire, the disposition of the glass plates on which the materials is to be evaporated etc.) which could be achieved only by use of very costly sophisticated equipments. The chemical deposition technique

Price : TWO RUPEES.

involves the only known method using seleno-urea which is prohibitively costly (about Rs. 16,000/- per 200 gms) and this involves a lot of foreign exchange.

The object of this invention is to substitute the costly chemical seleno-urea and make the new process cheaper.

The main finding (the new principle) underlying the invention:

In this new process the lead selenide is deposited on ground glass substrate, by the reaction on the solution of sodium seleno sulphate ( $\text{Na}_2\text{SeSO}_3$ ) with the lead acetate in alkaline medium in a pH range of 8-10. The preparation of sodium seleno-sulphate is quite easy and consists in dissolving selenium powder in an aqueous solution of sodium sulphite.

The new result flowing from the new finding:

The new process replaces the use of seleno-urea which is prohibitively costly (200 gms costing Rs. 16,000/-) and is at present to be imported. Whereas the preparation of seleno-urea is very much involved, sodium seleno-sulphate is prepared easily by merely dissolving selenium in sodium sulphite solution. The selenium powder (A. R.) though not produced indigenously, is available easily as about Rs. 200/- per 200 gms.

According to the present invention the process consists in the chemical deposition of thin films of lead selenide layers by reacting sodium selenosulphate and lead acetate in alkaline medium in a pH range of 8-10.

In the invented process, seleno-urea has been replaced by sodium seleno-sulphate ( $\text{Na}_2\text{SeSO}_3$ ) which can be prepared easily by the dissolution of selenium powder in an aqueous solution of sodium sulphite.

Addition of ammonia or hydrazine during the deposition would make a deposit more uniform and adherent. The method of deposition is easy and factors like film thickness, uniformity etc. which govern the properties of the film can easily be controlled during the deposition. The deposits are obtained at room temperatures (30-35°C).

A few typical examples:

#### Example 1

Deposition bath consists of about 50 c.c. of approximately 40% lead acetate, an equal volume of 1% selenium in sodium sulphite solution of about 20% strength. The ground glass plates affixed to a perspex holder are immersed in the bath and then the contents are stirred. Fairly concentrated ammonia or hydrazine are added to the solution containing selenium in sodium sulphite solution. The plates are allowed to remain inside the solution for about two hours. Good adherent deposits are obtained. On heating to a temperature around 300°C these thin films show photoconductivity in the infrared region.

#### Example 2

Deposition bath consists of about 50 c.c. of approximately 5% lead acetate, an equal volume of 1% selenium in sodium sulphite solution of about 20% strength. The ground glass plates affixed to a perspex holder are immersed in the bath and then the contents are stirred. The pH of the solution is adjusted to be in the range of 8-10 by adding sodium hydroxide. The plates are allowed to remain inside the solution for about 2 hours. Fairly adherent deposits are obtained even without addition of hydrazine or ammonia.

On heating to a temperature around 300°C., these thin films show photoconductivity in the infrared region.

The main advantages of the invention:

1. No costly equipment is needed.
2. Method of deposition is chemical and avoids the use of seleno-urea, which is a costly material requiring at present foreign exchange.
3. Method of deposition is very easy and reproducible and can be done at laboratory temperature (30-35°C).
4. Addition of ammonia or hydrazine during the deposition would make a deposit more uniform and adherent.
5. Control of the bath for introducing dopants etc. will be easy.

Summary (critical discussion):

Infrared sensitive lead selenide layers have been used in the wave length range 3-7 microns. These layers were previously obtained either by vacuum deposition technique or by the method of chemical deposition. They however involve either costly equipments or costly chemicals like seleno-urea. This invention has evolved a chemical method of deposition which avoids the use of seleno-urea. By the addition of ammonia or hydrazine more uniform adherent deposits could be obtained. The whole process reduces the cost of production of these cells.

Noteworthy features:

1. The present invention of chemical deposition of lead selenide layers by using sodium seleno sulphate avoids the use of the costly imported chemical seleno-urea used in a similar method.
2. By this process the number of lead selenide cells that may be prepared for a gram weight of selenium is about three times in comparison with the seleno-urea method.

We Claim:

1. The chemical deposition of thin films of lead selenide layers by reacting sodium selenosulphate and lead acetate in alkaline medium in a pH range of 8-10.
2. A process as claimed in claim 1 wherein seleno-sulphate is prepared by the dissolution of selenium powder in an aqueous solution of sodium sulphite.
3. A process as claimed in claim 1 or 2 wherein is added ammonia or hydrazine during the deposition.
4. A process as claimed in any of the preceding claims wherein the deposits are obtained at room temperatures (30-35°C)
5. A process for the chemical deposition of lead selenide layers substantially as described in the examples.
6. A process for the chemical deposition of thin films of lead selenide layers substantially as hereinbefore described.

Dated this 4th day of November 1967.

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