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TITLE: Novel High Resolution, Low Dose Flat Panel Mammography Detector Technology

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This project is to apply a thin coating of thallium bromide (TIBr) to the surface of readout CMOS integrated circuits incorporated into a slot-scan mammography system. Such an approach would reduce the cost of sensor fabrication and provide a near 50% increase in quantum efficiency compared with silicon PIN diode detectors hybridized with CMOS readouts. To realize the technology, an evaporator and a sputtering system needed for this project have been assembled, modified, and tested for detector fabrication. A stripping process to remove indium bumps from CMOS readout integrated circuits has been developed. Two thallium-bromide-coated nonfunctional sensor arrays were fabricated and studied by scanning electron microscopy (SEM). Our initial findings indicate that no chemical attack was observed at the interface between thallium bromide and substrate. We, however, may have the adhesion problem of thallium bromide to the substrate material. We have been making efforts to search for a buffer material deposited prior to the growth of thallium bromide to resolve the issue. We expect to deliver two thallium-bromide-coated functional sensor arrays for the evaluation of their X-ray imaging properties in February, March, and April, respectively. A prototype system will then be fully characterized in May.
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Annual Progress Report
Grant No. DAMD17-97-1-7016

A Novel High Resolution, Low Dose Flat Panel Mammography Detector Technology

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1. Introduction

This annual project progress report describes work carried out from October 1, 1998 to December 31, 1999 of a two-year mammography sensor development program. The project, as agreed to in Modification P90003, was originally proposed to deposit the thallium bromide photoconducting material onto an addressable array of cold cathode field emitters (FEs) to fabricate into a Field Emission X-ray Imaging System (FEXIS) specifically optimized for mammography applications. The grant period was from June 1, 1997 through January 1, 2000. After we made 15-month research and development efforts including some progress on TIBr deposition, computer tip emission trajectory simulation, and preliminary device testing and measurement, one of our subcontractors, FED, Inc., however, was unable to manufacture functional field emission arrays suitable for our application. The technical hurdles to be overcome include a smaller pixel size of FE and a higher beam current to discharge the target pixel required for mammography. Therefore, a 3-month, no cost extension was initially requested and approved.

After the first annual report was submitted to Army in September 1998, we began to look for alternative flat panel readout options. We then identified a more promising approach to achieving the stated goals of the grant and proposed a revision to the project on March 22, 1999. The proposed plan, which was approved by Department of the Army on April 19, 1999, is to deposit thallium bromide onto silicon CMOS integrated circuit devices that are currently employed in slot scan imaging. X-rays absorbed in the photoconductor generate photocurrent that will be collected by the array of electrodes and integrated at each pixel during the frame integration time. As such, PrimeX General Imaging (PGI) replaces FED, Inc. as a subcontractor in the present project. The project completion date has been changed to July 1, 2000, with research to be completed by May 31, 2000. Accordingly, all the tasks and experimental results discussed here are based on our revised proposal.

2. Program Description

The present program is to apply a thin (about 50-µm thick) coating of thallium bromide (TIBr) to the surface of readout CMOS integrated circuits (see Fig. 1) incorporated into a slot-scan mammography system. This approach would provide a near 50% increase in quantum efficiency compared with silicon PIN diode detectors hybridized with CMOS readouts. In addition, it also reduces the cost of sensor fabrication by eliminating the need to manufacture PIN diode arrays
and the hybridization process required to attach the diode array to the readout array. As such, the main goal of this program is to determine appropriate materials and develop a deposition process for coating thallium bromide onto silicon integrated circuits. There are three tasks being proposed to carry out the project:

- **Task 1.** Establish the viability of the straight-line evaporation technique employed to deposit thallium bromide films and study the compatibility between thallium bromide and its adjacent materials (e.g., bond pads, intermetallic contacts, and passivation layers used in the silicon CMOS integrated circuits).

- **Task 2.** Determine the best materials to use for intermetallic contacts between thallium bromide and the bond pads on the integrated circuits and for making a bond pad on the top surface of thallium bromide.

- **Task 3.** Test thallium-bromide-coated CMOS detectors in a prototype slot-scanning mammography system developed by PGI.

![Figure 1. A thallium bromide coated CMOS X-ray detector.](image)

### 3. Experimental Details

#### 3.1. Task 1

After the Army office approved the revised experimental plan of the present project, we purchased components that were needed to set up an evaporator and modify our existing dc sputtering system. There was a waiting period to receive all components as needed. A straight-line evaporation system was finally assembled in October 1999 and tested to coat silicon CMOS readout circuits. The system consists of a bell jar (12 inches in diameter and 18 inches in height) evaporation chamber that is pumped by an oil free scroll pump and a turbo pump. The thermal evaporation uses a quartz crucible heated by tungsten wire basket. A quartz lamp and a temperature controller are employed to control the substrate temperature. The deposition rate is controlled and monitored by a Leybold quartz rate monitor.

The CMOS integrated circuits (ICs) used for this project were originally designed for the readout of silicon PIN diodes. Therefore, prior to the deposition of thallium bromide, indium bumps have to be removed from the CMOS integrated circuits. In order to remove the indium bumps, the ICs are soaked in a dilute solution of nitric acid. **PGI** has assembled a facility to store, handle, and properly dispose of the nitric acid solutions used in the stripping process. Care must be given to this process such that all of the indium is removed yet no contaminants are left on the chips during or after processing. Moreover, it is important to ensure that no other variables (e.g., nitric acid residual, and the formation of nitrides) are introduced during this process that could lead to problems during thallium bromide deposition. The readout ICs also have to be carefully handled to avoid static discharge damage. They are inspected visually before, during, and after processing to ensure proper quality control of the process.
The stripping process, which has been developed initially using non-functional integrated circuits and then applied to working integrated circuits, can be summarized as follows:

Step 1: The wire bondpads of the integrated circuits (ICs) are coated with a photoresist.

Step 2: After the photoresist has dried, the ICs are placed in a 0.1-N nitric acid solution and left to soak for 12 hours.

Step 3: The ICs are removed from the solution and washed with distilled water.

Step 4: The ICs are then visually inspected with an optical microscope to determine all of the indium has successfully been removed and that no degradation of the bondpads or vias has occurred.

Step 5: The photoresist is then removed.

Step 6: The ICs are then given a final visual inspection.

A process to inspect the surface of the CMOS integrated circuits (ICs) after thallium bromide growth has been developed. ICs are examined for failure because thallium bromide attacks a variety of materials. The failure analysis procedure involves cleaving the die and examining the die with a cross-sectional view using a scanning electron microscope. With this process, we are able to examine the structural integrity of the ICs with extremely high resolution. Both functional and non-functional ICs are inspected and compared.

Two non-functional, stripped ICs were coated with 5-μm-thick thallium bromide films. One of them was then over-coated with 50 nm of hastelloy as a metallic contact layer. The coated ICs or sensor arrays were cleaved and examined using a scanning electron microscope (SEM).

Figure 2 shows a view of the sensor array at a 14,000X magnification. As illustrated in the upper portion of the image, the thallium bromide layer has been peeled back from the surface of the sensor array exposing the pixel bond pad via. There is no observable corrosion or degradation of the surface of the sensor array. Figure 3 shows another cross-sectional view at a higher magnification, 25,000X. As shown a layer of titanium-tungsten a few thousand Angstroms thick covers the pixel bond pad via. Beneath the metal layer is a layer of over-glass or passivation.
layer. A layer of glass and a layer of aluminum follow this layer. There is little or no observable corrosion or damage to any of these layers. There seems to be a pocket of missing material in the lower right hand portion of the via. This may have been caused by cleaving the die or from poor fabrication and does not appear to be caused by the thallium bromide. Figure 4 shows another cross-sectional view of a pixel bond pad. In this view, some of the indium bump material is shown. This indicates that the process developed to remove this material does not remove all of the indium. Therefore, we will increase the soak time used in the removal process to make sure that all of indium is removed.

![Figure 3](image3.png)

**Figure 3.** A cross-sectional SEM view (25,000X) of a thallium-bromide-coated sensor array.

![Figure 4](image4.png)

**Figure 4.** A cross-sectional SEM view of a pixel bond pad.

### 3.2. Task 2

We have noticed that after the sensor arrays are cleaved, the thallium bromide layer is easily peeled off of the arrays. This would indicate that we are not getting good adhesion. In order to improve adhesion, a layer of thin conductive oxide will be applied to pixel bond pads. As such,
we have modified our dc sputtering system that is now also equipped with a rf sputtering unit. The system will allow us to sputter both metal and oxide targets.

3.3. Task 3

Nothing was performed on Task 3 during this project reporting period.

4. Future Work

4.1. Task 1

We have received two functional and several non-functional readout integrated circuits from PGI. With the newly assembled straight-line evaporator dedicated to this project, we are now making significant efforts on studying the effect of process parameters (temperature, pressure, power level, the distance of evaporation source to substrate, etc.) on thallium bromide properties. The results will be compared with those of the thallium bromide films fabricated using a hot-wall evaporator. After conducting a systematic study of process development led by Dr. Kung (P.I. to the present project) for a corporate-sponsored R&D project in 1999, we were able to obtain good control over the properties of hot-wall-evaporated thallium bromide films fabricated for vidicon-based medical imaging applications. Those thallium bromide films deposited on aluminum disks in the hot-wall evaporator, in general, show their resistivity values in the range of $10^{12}$ - $10^{13}$ ohm-cm at $-30$ °C and very few or zero blemishes in X-ray images. We hope that we could achieve the same or even better result for thallium bromide films deposited in the straight-line evaporator in two months. Our approach is to coat two functional ICs with thallium bromide in February, March, and April, respectively. These films will be fabricated under the best processing conditions achieved during that month and sent to PGI to test for their X-ray imaging properties. To realize such a goal, we will vary process parameters and deposit thallium bromide onto non-functional ICs, aluminum disks, and silicon wafers. These films will be characterized by X-ray diffractometry (XRD), scanning electron microscopy (SEM), energy-dispersive analysis of X-ray (EDAX), and secondary ion mass spectroscopy (SIMS). They will also be tested for their electrical resistivity and X-ray detection properties as well as the adhesion of thallium bromide to substrate material. Several control samples will be simultaneously produced and tested in a conventional vidicon imaging tube.

4.2. Task 2

We will develop well-controlled sputtering processes to deposit bottom (if needed to improve the adhesion properties) and top electrode layers. At the moment, we consider using copper oxide or cerium oxide for bottom electrodes if it helps improve the adhesion properties. For top electrodes, we will try indium-tin oxide first. All of these electrode layers will be characterized.

4.3. Task 3

PGI has begun to set up its facilities for testing the X-ray imaging properties of thallium-bromide-coated integrated circuit devices. It is expected that the properties including sensitivity,
spatial resolution, noise figure, dynamic range, detector quantum efficiency, and modulation transfer function of a prototype slot-scanning mammography system based on thallium bromide integrated detectors will be measured. The project principal investigator, Pang-Jen Kung, will meet with PGI staff in March to discuss details on measurements. Professor Roehrig of the University of Arizona will provide expertise and help on some of these technical issues.

4.4. **Schedule**

<table>
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<th>Month</th>
<th>Remarks</th>
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| February | - Optimize the deposition process of thallium bromide films  
- Deliver two thallium-bromide-coated functional sensor arrays to PGI for evaluation |
| March | - Optimize the deposition process of top and bottom electrode layers  
- Deliver two thallium-bromide-coated functional sensor arrays to PGI for evaluation  
- Meet with PGI staff to discuss measurement details |
| April | - Finalize the fabrication protocol for thallium bromide X-ray sensor arrays  
- Deliver two thallium-bromide-coated functional sensor arrays to PGI for evaluation |
| May | - Evaluate product commercialization potential  
- Characterize thoroughly a prototype X-ray imaging system  
- Submit papers to technical journals |
| June | - Attend the Era of Hope Meeting  
- Submit the final report |

5. **Conclusions**

The evaporator and the sputtering system needed for this project were assembled, modified, and tested. A stripping process to remove indium bumps from CMOS readout integrated circuits was developed. Two thallium-bromide-coated nonfunctional sensor arrays were fabricated and studied by scanning electron microscopy (SEM). Our initial findings indicate that no chemical attack was observed at the interface between thallium bromide and substrate. We, however, may have the adhesion problem of thallium bromide to the substrate material. We have been making efforts to search for a buffer material deposited prior to the growth of thallium bromide to resolve the issue. We expect to deliver two thallium-bromide-coated functional sensor arrays for the evaluation of their X-ray imaging properties in February, March, and April, respectively. A prototype system will then be fully characterized in May. Commercialization potential to this technology will also be assessed in the near future.

6. **References**

None.

7. **Appendices**

None.
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