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REVIEW OF MERCURIC IODIDE DEVELOPMENT PROGRAM IN SANTA BARBARA*

by

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ABSTRACT

Crystal growth and device fabrication methods used at EG&G's Santa Barbara Operations are reviewed. Different types of detectors are described and areas of application are discussed.

1. INTRODUCTION

The purpose of the detector research and development program at EG&G's Santa Barbara Operations is to develop detector systems that weigh less and are more sensitive and compact than presently available systems. This implies that the new detectors can operate at ambient temperatures, require only a small power supply, and do not need extensive auxiliary opto-electronic equipment. The systems should be able to operate in both the counting and spectrometry modes.

Mercuric Iodide (HgI_2) is one of the candidate materials because of its large bandgap and high absorption for nuclear radiation. Possible other materials are zinc telluride (ZnTe), cadimum telluride (CdTe), and cadimum selenide (CdSe).

2. MATERIAL PURIFICATION AND CRYSTAL GROWTH

2.1 Purification

The raw material used is commercially available, reagent grade HgI_2 (98%) pure). We obtain our supply mostly from Baker, but materials from

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other manufacturers (Malinchrodt, BDH, Allied Chemical) have also been used.

The first steps in the purification sequence are four consecutive sublimations under vacuum (Figure 1). The charge is kept at a temperature of approximately 120°C, and the pumping is continuous. In the next step the product of the vacuum sublimation is melted in batches of approximately 100 g each at 280°C for 24 hours, and then cooled slowly (Figure 2). Finally, the melt product is sublimed in an evacuated, sealed tube through a glass frit, so that impurities segregated out during the melt procedure are separated from the main charge (Figure 3).

2.2 Crystal Growth

The single crystals are grown by the well-known method initially used by Scholz and further developed and refined at EG&G, Santa Barbara. The purified powder is filled into growth ampoules in amounts of 200 to 500 g (Figure 4). The ampoule is placed in a furnace (Figure 5), and the temperature profile is adjusted in such a way that the charge collects on the upper part of the vertical walls of the growth ampoule.

Nucleation is performed by cooling of the center of the pedestal in the ampoule bottom. A proper seed is selected by selective etching, which may include tilting of the seed for proper orientation. Once a good seed is obtained, growth is started by judicious adjustment of the temperatures of the ampoule base and the source material and of the cooling supply to to the pedestal center. The growth sequence is largely automatic, and in-house built control units are used (Figure 6).

At present approximately 20 furnaces are in operation (Figure 7). Ten of these use a simple, vertical source heating coil, and the ampoule is continuously rotated. The others have a static ampoule surrounded by a helically wound vertical coil (Figure 8). The pedestal center can be cooled by air or by the use of a brass rod with a Peltier cooler at the end (Figure 9).

The average time to grow a 200-g crystal is approximately six weeks. When considering this time, it should be kept in mind that many runs are still of the experimental research type, using charge powders which have been prepared in different ways. The minimum time for a complete growth sequence (including nucleation) of a 200-g crystal is three weeks.

2.3 Characterization

The purification and crystal growth procedures are evaluated by several independent methods:

Total Impurity Content – This has been determined by several techniques. The mass spectrometric analysis method seems to be the most consistent; results indicate a total metal impurity content of approximately 100 ppm.

Stoichiometry Measurements — Several laboratories have developed methods to determine the mercury/iodine balance in the material by chemical analytical methods.

Luminescence — Experiments have shown that the luminescence spectrum at low temperatures varies with the quality of the material.

These methods and details of the results obtained have been described extensively in the literature, and will also be discussed in presentations at this meeting.

3. DETECTOR FABRICATION AND TESTING

The crystals (Figure 10) are shaped into the desired form by solution cutting (Figure 11), followed by solution polishing (Figure 12) of the surface on which the contacts will be deposited. These methods of material processing are preferred over cleaving, which causes more extensive damage to the material as shown by gamma-ray diffraction measurements. Cleaving is also restricted to relatively small samples, so that large crystals have to be cut into smaller sections before the cleaving can be done.

Presently, the most commonly used electrode materials are palladium and germanium, although colloidal carbon solutions are sometimes still used for quick evaluations of the material. The metals are evaporated by means of thermal heating or, preferably, by electron bombardment in a system at a relatively low vacuum (10^{-5} Torr) (Figure 13). The piece of mercuric iodide to be contacted is masked off by a photoresist technique which allows for exact definition of the metal deposition area. Using these techniques, it is possible to evaporate several 1-cm^2-area detector structures in one operation on a large slice of material (Figure 14). The individual detectors can then be cut out of the slice.

The presently used contacting procedure is far from ideal. The mechanical (adhesion) and electrical properties of the contacts needs further improvement. New materials being considered are tin oxide, tin, and lead.

Detector quality is evaluated by several methods. The standard procedures developed use a 1-cm^2 detector, nominally 0.5 mm thick at a bias of 1500 V.

The leakage current measured when the detector is brought up to the bias required for charge collection is used as an indication of the background noise which can be expected during data acquisition. Especially for low-energy x-ray detectors, it is critical to keep the leakage current as low as possible.

The quality of the ^{241}Am and ^{137}Cs spectra is used to evaluate the charge collection properties of the detectors (Figures 15 and 16). The americium spectrum provides information primarily about the detector stability, and the quality of the cesium spectrum (FWHM, peak to valley ratio) indicates the amount of hole collection.

One detector from each crystal is normally used to determine the $\mu\tau$ product. Recently the values obtained are of the order of 10^{-5} cm^2/V for holes and 10^{-3} cm^2/V for electrons. These values, combined with optical

microscopic observations of the detector slices before contacting, are valuable feedback information for the material preparation and crystal growing group.

4. APPLICATIONS

1. Thin (0.5 mm) Gamma-Ray Spectrometers (Figure 17): Area 1 cm^2; Range 10 to 500 keV

   These spectrometers can be used singly or in an array arrangement to detect and identify gamma rays. This type of detector has several existing applications, for example the MIT balloon experiment flown in 1981 to measure gamma-ray bursts from Cygnus I, for which a 12-element array was used.

2. Thin X-Ray Spectrometers: Area 10 mm^2; Range 1.0 to 20.0 keV

   Applications of this type of detector are in a variety of x-ray fluorescence instrumentation. Use of these detectors has been developed mainly by Dr. Huth's group at USC under EG&G's sponsorship and the leadership of Dr. A. Dabrowski, and will be discussed in a separate presentation.

3. Thick Gamma-Ray Counters (1 cm): Volume 15 cm^3; Range 10 to 1000 keV

   Use of thick detectors as sensitive gamma-ray counters has been developed by John Warren in EG&G SBO laboratory. The hand-held systems (Figures 18, 19, and 20), including the required electronics, will be described in more detail in proceedings of this workshop.

4. Thick Gamma-Ray Spectrometers (1 cm): Volume 6 cm^3; Range 100 to 1000 keV

   Recently, we have been able to obtain spectra of high energy radiation with thick detectors at moderate biases (2000 V)(Figures 21, 22, and 23). The work in this area will be discussed by Drs. A. Beyerle and K. Hull in the workshop proceedings.

5. Single Detector Arrays (Figures 24, 25, 26, and 27): Total Area 2×2 cm; Range 10 to 150 keV; Electrodes — 1.8 mm; gap — 0.2 mm typically; Resolution — 2×2 mm

   These position-sensitive arrays are being developed primarily for gamma-ray imaging systems which have medical and other applications. C. Ortale will discuss this work in the workshop proceedings.

6. Multi-Element Arrays: 64 (8×8) Single 1-cm^2 Detectors; Range 10 to 600 keV

   These arrays (Figure 28) are being developed for applications in large gamma-ray cameras. The signals from individual elements will be processed by appropriate electronics, so that the intensity and the energy of the incident radiation can be mapped.

7. Space Experiments

Plans are in an advanced stage to grow a relatively small (20 g) single crystal of mercuric iodide on board Spacelab III. A special furnace (Figure 29) has been designed for this purpose. The rationale behind this experiment and the equipment details will be discussed in the proceedings by W.F. Schnepple.

5. CONCLUSION

This presentation has reviewed the highlights of the mercuric iodide development program at EG&G Santa Barbara Operations. Results obtained during the past year indicate that the presently available detector systems can be used effectively in many applications, and that other applications can be expected as the quality of the detectors improves.