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A new method for vacuum sealing of flat-panel photosensors

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Abstract

Photon detectors are indispensable in many areas of fundamental physics research, particularly in the emerging field of particle astrophysics, as well as in medical imaging and nuclear non-proliferation. For a significant progress in all these areas a new, inexpensive industrial mass-production photosensor technology is needed. In order to reach that goal we have introduced several innovations. This paper focuses on one of them, the ReFerence flat-panel photon detector. We report on the prototype development, and in particular on the development of a new oxidation-free method for vacuum sealing of glass panel surfaces with liquid indium, at processing temperatures below 360 °C.

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1. Introduction

Many future experiments within the broader field of particle astrophysics will study very energetic or extremely rare phenomena.³ These elusive phenomena require special means of observation, with detectors whose sizes should greatly exceed the dimensions of the largest current experiments. In the construction of detectors on such a large scale, no other option remains than to use natural media—the atmosphere, deep packs of ice, and water. In these transparent media, charged particles that originate in impacts or decays of primary particles radiate Cherenkov or fluorescence light. Photosensors are the only well proven active detector component⁴ to convert photons into

electrical signals. Consequently, photon detectors are the single most important detector elements in this field. Photosensors play equally important role in other similar application areas, particularly in medical imaging and nuclear proliferation control. These areas present a large potential market for new, inexpensive, high-quality, industrially mass-produced photosensors. For various reasons [1] neither the current vacuum photosensor technology, nor the modern semiconductor photosensor technologies may be suitable for large-area mass applications.

The proposed ReFerence flat-panel concept [1] essentially combines three fully established and well-understood technologies: the flat-panel plasma and field-emission TV-screen assembly, the large-area photocathode deposition, and the semiconductor particle sensor production (CMOS technology). The recent success of hi-tech vacuum flat-panel TV technologies, which truly exemplify modern industrial mass production, indicates that an equivalent breakthrough might be possible also in the field of vacuum photosensors.

After verifying the basic functionality of the ReFerence concept in a series of single-pixel vacuum-unsealed prototype tests, we started the development of fully functional vacuum-sealed flat-panel ReFerence prototypes. The

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³Proton decay, neutrinoless double beta decay, neutrino oscillations, supernova explosions, cosmic neutrinos, geoneutrinos, cosmic gamma rays, ultra-high energy cosmic rays, gamma-ray bursts, active galactic nuclei, dark matter, etc.

⁴There are ongoing activities to explore radio-wave detection, or acoustic detection of energetic showers, but these technologies have not yet reached maturity.

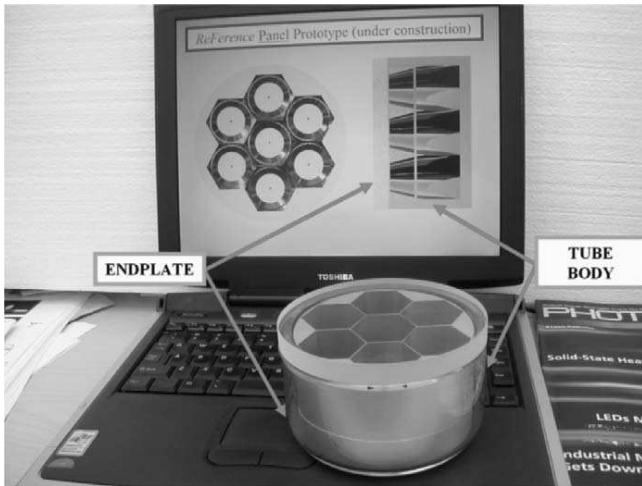


Fig. 1. Components of the ReReference panel prototype: design and reality. See text for details.

ultimate goal of that effort is to demonstrate the feasibility of an industrial mass-production technology. Our first flat-panel prototype comprises seven pixels hosted by a 5-in. diameter vacuum enclosure, see Fig. 1.

We present a new vacuum sealing technique developed for the manufacture of ReReference panel prototypes. This technique is distinct for three important features: (i) glass-to-glass vacuum sealing, (ii) processing temperature is limited⁵ to less than 250 °C, and (iii) the entire vacuum sealing process (including the sealing surface preparation) is conducted in an ultrahigh vacuum (UHV) environment. Our method utilizes vacuum deposition of precisely dosed layers of chromium, gold, and indium on the glass surfaces to be sealed. We will explain the physical foundation for this technique, report on small-scale experiments used to test and optimize the method, describe the accordingly designed UHV transfer system, and report on the first full-scale sealing tests.

2. Vacuum sealing of glass panels

The ReReference panel prototype (Fig. 1) comprises the following components: (i) a cylindrical glass tube (2.5 in. long, 5 in. diameter, 6.4 mm wall thickness), (ii) a 7-pixel honeycomb plate structure, (iii) a front window plate, and (iv) an endplate that covers the back opening of the tube, forming the final vacuum seal.

The formation of a vacuum seal is in general one of the most difficult steps in the panel manufacture. We have been studying two different sealing techniques: (i) glass-frit sealing, and (ii) sealing based on melted indium. Flat-panel TV industry has developed and applied various techniques

⁵The processing temperature is limited to 250 °C for the following reasons: (i) the photoelectron detector (the avalanche photodiode) should not be exposed to more than 250°, and (ii) in general, one of the goals of the ReReference flat panel concept has been to work at low, cost-effective temperatures, like the flat-panel TV industry, where efficient plasma cleaning has largely replaced the high-temperature bakeout.

based on frit sealing. We have even acquired an ingenious frit sealing system based on infrared lasers (that briefly melt the glass frit), from a leading field-emission panel developer. However, significant dedicated funding would be necessary to start with the related R&D. We have mainly focused on the somewhat simpler indium sealing technique that has been widely used in the image intensifier industry. In order to gain deeper insight into that technology, we also acquired a complete industrial transfer system, the EPIC system developed by Litton-Varo for the production of second-generation night-vision image intensifiers.

With a low melting point $T = 156.67^\circ\text{C}$, very low vapor pressure $p = 1.42\text{E}-17\text{ Pa}$ (at its melting point), and extreme softness and ductility, indium presents an ideal sealing medium. In order to create a vacuum seal, liquid indium needs to wet the sealing surfaces. Pure indium does not wet glass,⁶ and therefore it may not adhere directly to glass. However, indium strongly interacts with gold [2–4] and creates an intermetallic compound, AuIn_2 , which enables efficient surface wetting [3]. The solution is therefore, to pre-coat the glass surfaces with gold, and then deposit indium on top.

If deposited in air, the indium layer would oxidize. Since indium oxide prevents vacuum sealing, it must be removed from the surface in a vacuum. As demonstrated in Refs. [5,6], indium oxide completely evaporates in vacuum from the liquid indium surface, but only above the threshold temperature of 360 °C. The physical process responsible for this effect is the reduction of the solid oxide $\text{In}_2\text{O}_3(\text{s})$ by liquid indium $\text{In}(\text{l})$ above 360 °C, which leads to the formation of gas $\text{In}_2\text{O}(\text{g})$: $\text{In}_2\text{O}_3(\text{s}) + 4 \text{In}(\text{l}) = 3\text{In}_2\text{O}(\text{g})$. Consequently, while in the image intensifier industry (which operates at $T \sim 400^\circ\text{C}$) indium oxide evaporates spontaneously, in our case ($T < 250^\circ\text{C}$) it will not evaporate at all. Our solution to this problem is to deposit an oxide-free layer of indium in UHV, and prevent subsequent oxidation. More specifically, our strategy is to

- a. deposit gold on at least one of the sealing surfaces, in order to enable surface wetting;
- b. deposit indium on the sealing surface, in UHV;
- c. in addition, deposit chromium as a primary layer on all the sealing surfaces [3], in order to establish strong chemical bond with glass, and to prevent gold from flaking off the glass surfaces;
- d. deposit a thin layer of Gold on top of the freshly deposited indium layer, in order to protect indium from oxidation, and

⁶Contrary to many claims in the literature, in our experiments we have found that pure indium does not wet clean glass in vacuum, or in inert gas atmospheres; it rather forms spherical droplets like mercury. However, we also found that indium may wet glass in air, with the help of mechanical rubbing. We believe that the wetting in air is due to the presence of indium oxide, and/or alloys created in contact with the tools used for rubbing (typically nickel brushes).

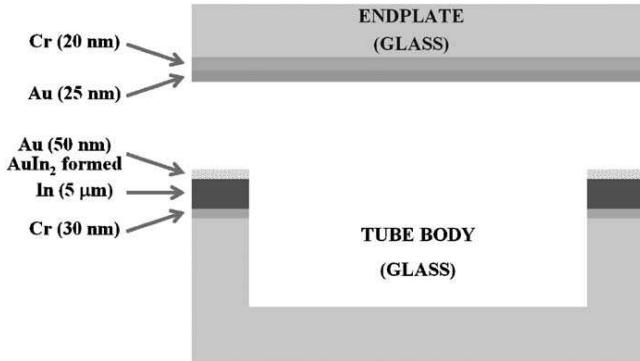


Fig. 2. The multi-layer structure deposited on the sealing surfaces. See text for details. (Sizes not to scale.)

e. keep the prepared prototype components exclusively in UHV.

To test and optimize this technique, we have carried out a series of small-scale experiments using pairs of microscope glass slides. One of the slides in a pair had a 10 mm hole for vacuum leak testing. We deposited Cr, Au, and In layers on the slides in UHV, and sealed them in another UHV system, which means that the deposited layers were in the meantime exposed to air. Gold capping of the indium surface (point (d) above) apparently worked well in preventing oxidation, but we still observed some solid residue in the indium melt. We concluded that it would be safer to keep the components in UHV during the entire process (i.e. we appended point (e) to the list). More important, if the prototype components were exposed to air, they would need another bakeout in UHV. Such a bakeout would prematurely melt the deposited indium seal and expose it to oxidation. Consequently, complete processing in UHV seems to be mandatory.

Our experiments have confirmed the feasibility of the proposed method. They also confirmed the importance of very precise dosage of the deposited materials. In some experiments, we have overdosed the amount of gold, which resulted in the formation of a brittle, microporous and non-sealable layer [2–4]. Precise dosage of indium and gold is therefore of paramount importance. We converged to the deposition scheme displayed in Fig. 2, which is very similar to the method developed for power laser sealing, and for self-assembly of MEMS devices [3]. Through our small-scale experiments, we also understood and solved numerous problems related to thermal evaporation, high-current power transmission, and excessive heating. Based on all the derived processing requirements, we have constructed a new transfer system for full-size prototype processing and vacuum sealing, described in the following section.

3. UHV transfer system

The UHV transfer system, Fig. 3, comprises four individually pumped subsystems connected by gate valves.

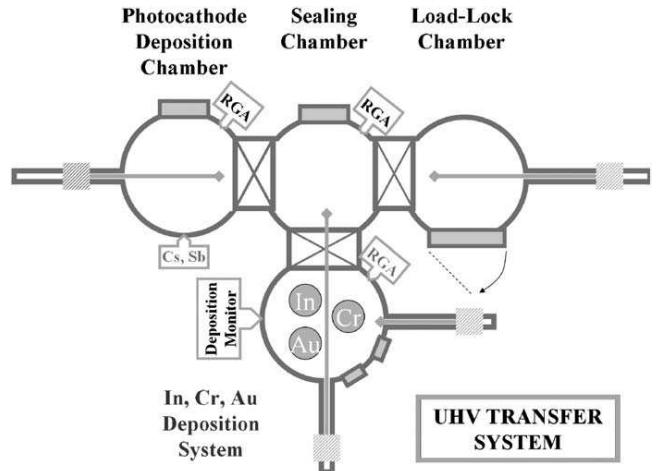


Fig. 3. The UHV transfer system comprises four subsystems connected by gate valves. Four magnetic arms manipulate prototype components throughout the system. See text for details.

Four horizontal translation–rotation magnetic motion arms, in conjunction with a motorized vertical xyz stage above the sealing chamber, allow us to introduce prototype components into the system (through the load–lock chamber) and transfer them from one subsystem to another. Each of the three processing subsystems has its own residual gas analyzer (RGA), which allows permanent control of the concentration of gases, particularly of those that would be harmful for the photocathode and indium. An RGA also monitors the outgassing dynamics during the bakeout of the prototype components, indicating thus, when the process has completed. The transfer system is pumped (in total) by four turbo molecular pumps, two ion pumps, and two Titanium sublimation pumps with cryoshrouds. The base pressure at room temperature (when pumped only with ion and sublimation pumps) reaches 6×10^{-11} Torr. The load–lock chamber is the only part of the transfer system that occasionally opens to the atmosphere during regular operation, which should enable seamless serial tube processing with minimum downtime.

The deposition system consists of two sections connected by a gate valve; the lower section hosts the evaporators of Cr, Au and In, the upper hosts the transfer mechanism, the RGA, and the quartz crystal deposition monitor. The deposition monitor provides full control of the deposition rates and thicknesses, with a sub-nanometer precision. During the deposition process, AC currents of ~ 60 A heat the ceramic crucibles containing the sources. In spite of the strong heating (> 500 W), the pressure in the deposition system does not exceed 5×10^{-8} Torr. In order to diminish excessive heating of prototype components during the deposition process, we have constructed an exceptionally robust heat shield around the evaporation crucibles, using multi-layer tantalum foil cups, and a large tantalum shutter that insulates the entire crucible section; only the small

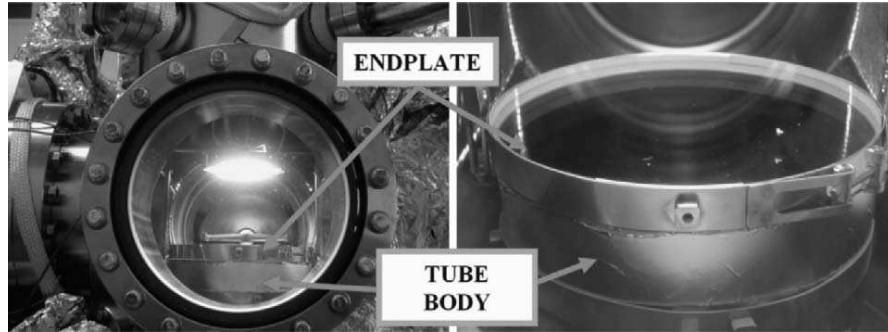


Fig. 4. The sealing process in the sealing chamber: the endplate is united with the tube body (see Figs. 1 and 2) and radiation heated from above (left); indium melts, dissolves gold, and forms the seal. After cooling, the sealed assembly is lifted, and prepared for the removal from the transfer system (right).

surface of the active source is open to the substrate through a hole in the shutter.

Once the Cr, Au, and In layers have been deposited on the sealing surfaces according to the prescription in Fig. 2, the components are transferred to the sealing chamber (see Fig. 4), joined together, slightly compressed, and radiation heated from above until the indium is melted. After the sealed assembly cools down, we take it out from the system through the load-lock chamber. We sealed the first two empty panel enclosures. The transfer system has performed according to the expectations based on the previous small-scale experiments. The sealed surfaces are smooth, and we saw no traces of oxide. We tested the seal for vacuum leaks, locally around the perimeter, using a helium leak tester, and we found no leaks within the sensitivity range of the instrument ($\sim 10^{-9}$ Torr l/s).

4. Summary and outlook

We have developed a new oxide-free method for the vacuum sealing of glass surfaces with indium. We discussed the physical foundation of this technique, reported on the systematical studies used to verify and optimize the method in a series of small-scale experiments, described the accordingly designed UHV transfer system, and reported on the first full-scale sealing tests of empty panels. In the

next step, we will seal closed tubes containing a bialkali photocathode.

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