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Instrumentation for Theory-Inspired Photocathode Development within the Large Area Picosecond Photodetector (LAPPD) Project

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Large Area Picosecond Photodetector Collaboration

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Abstract

We have designed and are commissioning a laboratory for the growth and characterization of photocathodes at Argonne National Laboratory. Two growth facilities, a versatile ultra high vacuum growth chamber and an industrial photomultiplier production facility, allow the investigation of fundamental aspects of the cathode growth and the development, modification and implementation of recipes in an industrial production environment. The instrumentation allows the study of optical properties, electrical behaviors and spectral response of the cathode.

Keywords: photodetector, photocathode, instrumentation, infrastructure, spectral response, quantum efficiency

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1. Introduction

Large area photo detectors have attracted considerable interest for their wide applications in astrophysics, nuclear and particle physics [1, 2]. At Argonne National Laboratory (ANL), the large area picosecond photo detector (LAPPD) project is developing detector modules with 8”x8” active area, a spatial resolution of 1-3mm, and ultimately a time resolution of 1-10ps. Key to increasing detection efficiency and significantly reducing detector system costs is the establishment of a cathode growth process that results in high quantum efficiency (QE), good production yield, and compatibility with the sealing process of the vacuum tube.

Over the years, much effort has been devoted to searching for suitable cathode materials and structures. Cathodes with large variations in wavelength sensitivity, peak QE, dark current, growth properties and robustness are grown or activated by standard, heuristically developed recipes. For example, the QE peak is very low (<1%) when the cathode was invented in the 1930s by an RCA group [3]. It was soon improved to a few percent [4] and then to 25% in the mid 1960s. Recently, Mirzoyan et.al reported PMTs with QE as high as 40% [5]. A survey of the literature shows that the properties, especially the QE, significantly vary even if the cathodes are produced by the same group or vendor using the identical recipe. Selected cathodes may be found that are 2-3 times more efficient than the average. It is our goal to understand the microscopic differences in the structural and chemical composition of these cathodes and correlate them to the macroscopic properties to provide guidelines for the development of new cathode recipes.

Our program encompasses bi-alkali (Na_2KSb and Cs_2KSb), multi-alkali ($Na_2KSb(Cs)$) and doped III-V (GaAs, GaN type) photo-cathodes, covering the detection wavelength from the UV to the IR-range. Our main focus is on the bi- and multi-alkali systems by applying cost-efficient thin film growth techniques.

2. The Requirements

A set of growth and characterization tools is needed to establish a theory-inspired program, i.e. an approach that engineers the microscopic structure of the cathode to optimize its functionality. A specially designed growth and characterization chamber with integrated characterization tools was built to study the *in situ* growth process to correlate the results with its functionality. Samples can be transferred under vacuum or ultrapure gas-atmosphere between this chamber and a high purity inert atmosphere glove box providing high flexibility to model various recipes. The evaporators and the process unit are miniaturized and equipped with minimal *in situ* characterization capabilities so that they can be duplicated and implemented in other chamber systems equipped with surface sciences tools or at synchrotron setups allowing microscopic measurements.

The correlation between “macroscopic” and “microscopic” measurements can be obtained by studying the cathode growth process using different characterization methods. There are “macroscopic” measurements such as optical spectroscopy and electrical measurements. The user may easily measure the absorption, reflection and transmission properties using a custom-built optical station or a fast, but fixed, wavelength fiber-based system. Electrical measurement such as current-voltage (I-V), QE, dark current and conductivity may be obtained *in situ* and in operandi. Also, there are the “microscopic” measurements like X-ray scattering techniques, atomic force microscopy (AFM) and scanning tunneling microscopy (STM) available at user facilities. These macroscopic and microscopic measurements interplay to monitor and analyze the cathode growth process.

An example of *in situ* monitoring of the microscopic structure of films using a “macroscopic” measurement has been reported elsewhere [6]. The variation of electrical resistance, a macroscopic property, with the antimony film thickness on carbon substrates was studied and an instantaneous

decrease of electrical resistance was observed at the amorphous-crystalline transition. This phase transition was also confirmed by transmission electron diffraction and dark-field electron microscopy. The fast and easy performance of "macroscopic" measurements makes it suitable for *in situ* monitoring microscopic growth process of our alkali cathodes.

In order to transfer the model recipes to a large format, industrial-like cathode facility, we also integrate a commercial phototube production unit. This system will be used to develop the evaporator insets for a large production unit. "Macroscopic" measurements will be employed as process control parameters.

3. Instrumentation

A series of different growth and characterization facilities were designed to study and understand the microscopic structure of cathodes to optimize their functionalities. An ultra high vacuum (UHV) growth and characterization chamber was specifically designed to simulate a wide range of known growth recipes, develop new ones and probe the intermediates *in situ* by various optical and electrical measurements. New recipes developed from the UHV chamber will be tested in a commercial photomultiplier tube (PMT) set up [7] and an evaporator system will subsequently be developed for the 8''x8'' cathode growth unit. A movable optical station was built with options for optical and electrical measurements. The station can be shared with different growth facilities within the lab. Portable evaporation systems are currently designed which allow using sophisticated electron and X-ray scattering and spectroscopic *in situ* tools available at user facilities like the Advanced Photo Source (APS) in ANL or at Brookhaven National Laboratory (BNL) [8].

3.1. Growth and Handling Facilities

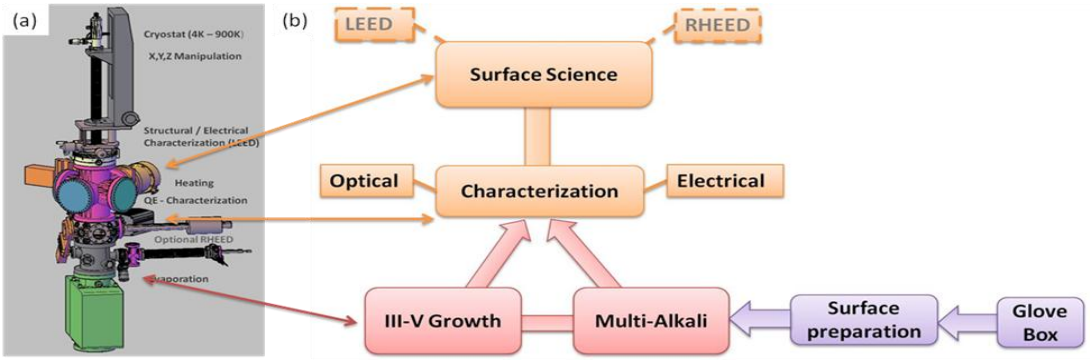

Fig. 1. (a) The UHV growth and characterization module with integrated multiple characterization tools. With the manipulators, samples can be moved around to any position in the chamber for growth and characterization. (b) Diagram of growth and characterization process flow.

3.1.1 UHV Growth and Characterization Facility

A dedicated growth and characterization facility, as shown in Fig. 1 (a), has been designed for the cathode process study. The chamber consists of three major parts: a growth chamber, an optical and electrical characterization chamber and a surface science chamber. The pumping system of the UHV chamber set up consists of a scroll, turbo and ion pump, which can reach a vacuum of a few 10^{-10} Torr.

The vertical design of the system has two major advantages. First, fast transfer between the growth and characterization chambers is possible enabling intermediate characterization points during a growth cycle. Secondly, by using cryo-traps between the three major chambers, cross-contamination is minimized, especially between the surface science and growth chambers.

The growth chamber is mounted directly on top of the ion pump, ensuring good vacuum. Six ports allow large flexibility in equipping the system with various sputter, evaporation and ion sources. Currently, we use either Sb or Sb/Pt alloy beads melted on evaporation wires, and Alkali dispenser sources from SAES for the cathode growth. For future recipe development, the port design allows the use of a Sb sputter source.

To study the correlations between macroscopic properties and microscopic growth a set of characterization tools are integrated in the UHV chamber. These tools allow optical spectroscopy, spectroscopic ellipsometry, measurements of quantum efficiency, photo- and temperature dependent conductivity and reflectivity. The setup also allows a modulation spectroscopic measurement to determine carrier density and mobility. Key components to this approach are a temperature controlled sample stage spanning the temperature range from liquid nitrogen to 700 ^{o}C, the integration of a photo detection system in the UHV chamber, a set of optical ports, and independent biasing of the sample. A quadruple mass spectrometer in combination with a gas inlet system allows study of the influence of cathode poisoning gases.

The conventional surface science chamber provides flexibility in applying standard surface science characterization tools to the process, which will be implemented in the future. The setup is equipped with a motorized manipulator at the top. Macroscopic properties and microscopic growth properties can thus be measured automatically at any step during the process. The transfer vessel allows cathode samples and activation materials to be transferred to other modules under vacuum or to a high purity inert atmosphere. Fig. 1(b) shows the growth and characterization process flow.

3.1.2 Glove Box

An HE-43-6 DRI LAB glove box from vacuum atmospheres company (VAC) is used for storage and assembly of alkali metal dispensers and for the chemical etching of glass substrates. The hermetically sealed glove box is fully equipped with a gas delivery system, a side-mounted antechamber, an oxygen monitor and a moisture monitor. An HE-493 DRI-TRAIN purification system is used to produce a moisture and oxygen-free atmosphere by recirculating the inert gas inside the glove box. This permits handling of materials sensitive to moisture and oxygen contamination such as alkali metals. The oxygen and moisture levels are closely monitored and the glove box is regularly regenerated to ensure low level impurities since the impurities in the glove box would affect the cathode growth process.

3.2. Characterization Tools

To characterize functionality and the microscopic structure we developed a set of easy to perform characterization tools based on various optical spectroscopic techniques and conductivity measurements. Whereas light source and beam conditioning unit are shared between the individual growth facilities we equipped each growth facility with individual sample handling and detection units. In the following we focus on the description of the basic QE-measurements.

3.2.1 Optical Station

The movable optical station, as shown in Fig. 2(a), is a fully automated optical and electrical measurement system designed and assembled at ANL, which can be shared with other growth and characterization facilities in the lab for multiple applications. A schematic drawing of the optical lay-out of this instrument is shown in Fig. 2 (b). Spectral response and quantum efficiency can be fast and easily performed by measuring the related beam intensity and electrical parameters.

The light source used in the optical station is a Newport 30-W continuum deuterium lamp, which covers the spectral range of 190-1600 nm and is controlled by a 0.1% regulated power supply. The lamp, power supply, motorized filter wheel and optics are fully integrated in a single enclosure, the Newport

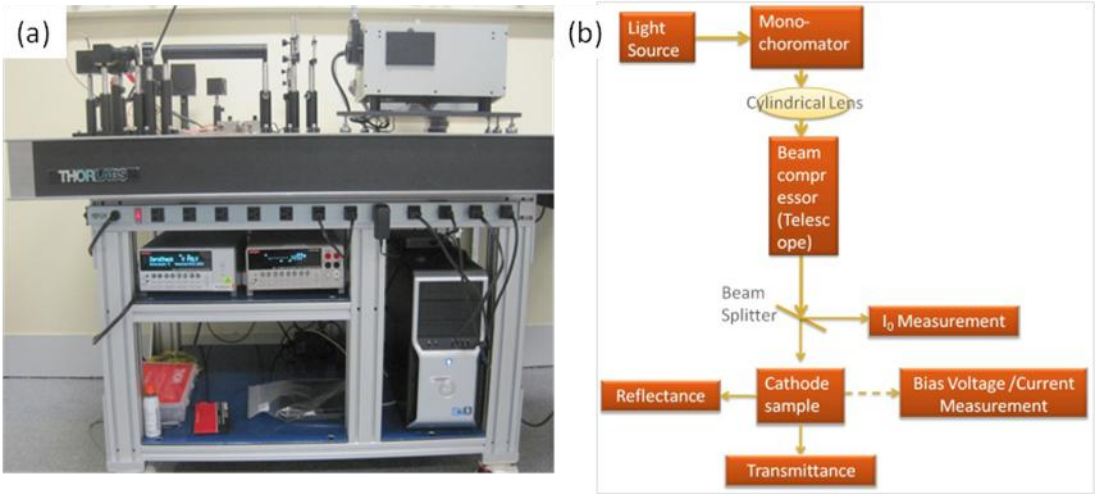

Fig. 2. (a) The custom built optical station at ANL. It consists of a 30-W deuterium lamp, fully automatic monochromator, beam collimation lenses and Keithley readout devices. Data acquisition is performed through LabVIEW software. (b) Schematic drawing of the optical set up layout.

Apex monochromater illuminator. A Newport Oriel Cornerstone 260 1/4 m monochromator was used to get a beam with 0.1 nm wavelength resolution.

A collimated beam is essential for our measurements since the optical set up will be shared among different facilities and the testing samples are at different distances from the beam exit. Moreover, a beam with low emittance enables measurements with defined angle between beam and sample surface. Cylindrical lenses are applied to collimate the beam since the light from the monochromator exit slit has different parallel and vertical focuses. After the beam is collimated, it is compressed to a small size (5 mm in diameter) using a telescope. The iris diaphragms are used to reduce scattering background in the system and define the beam position. The beam is split by a 90:10 (transmission: reflection) beam splitter so that the lamp emission can be monitored by measuring the current induced by the reflected beam from an NIST traceable silicon photodiode.

Cathode optical spectroscopy (reflection, transmission and absorption) as function of wavelengths (λ) is performed by measuring the different light intensities using the reference silicon photodiodes. The incident angle of the beam with respect to the sample normal is controlled with a 2-circle/goniometer, which also allows positioning of the photodetector. The reflected and transmitted beams are measured using a Keithley 2701 multimeter and a low noise Femto DLPCA-200 current amplifier by measuring the photoelectron current from the calibrated silicon photodiodes. The photoelectron current is amplified through a low noise amplifier with a gain setting at 10^8 or 10^9 V/A depending on the beam intensity. The

output voltage is then measured with the Keithley 2701 multimeter and the number of photons in the beam can be calculated easily.

For the quantum efficiency measurement, the bias voltage is provided by a Keithley 6517B electrometer and the photoelectrons generated from the cathode, which is held at ground potential, are directly measured through a Keithley 6517B electrometer with a detection limitation as low as 100 aA (1 aA = 10^{-18} A). Dark currents down to 500 electrons/s can be measured. All the instruments are connected through a LabVIEW program.

3.2.2 In situ Optical Characterization Based on Fiber Optics

A fiber optic system is used for *in situ* monitoring of the optical and electrical properties of the cathodes during the deposition process in a commercial PMT set up [7] as well as in the UHV growth facility. Since the substrate will be baked at 350 ^{o}C in the commercial PMT set up, we designed and fabricated a fiber optic system with a temperature tolerance as high as 400 ^{o}C. For the optical fiber, we chose a silica core with a diameter of 600 μm and a numerical aperture (NA) of 0.22. The outside jacket is made of polyimide with specification range from -190^{o}C to 400^{o}C. Three micro light emitting diodes (Mic-LED) with wavelengths at 365 nm, 405 nm and 470 nm are integrated with the optical fibers as light sources. Each Mic-LED provides a collimated beam that is combined into a single collinear beam by using a beam combiner optiblock. On the exit port of the beam combiner optiblock, a barrel and a focusing aspheric condenser lens are mounted to couple the beam into the optical fiber. Upon reaching the end of the optical fiber, the light is separated into three beams. The three beams are collimated with lens attached at the ends of the optical fibers. The ends of the optical fibers are mounted onto a cover for *in situ* optical and electrical characterizations (Fig. 3).

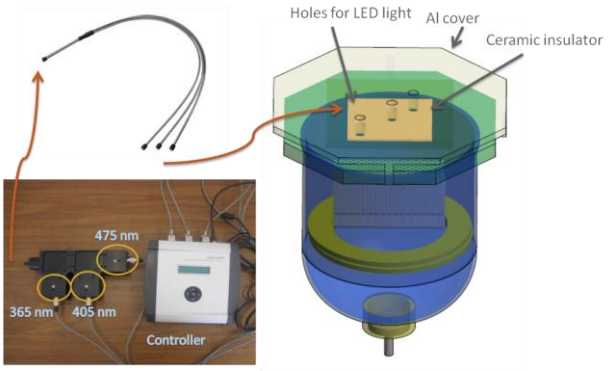

Fig. 3. In-situ optical characterization set up based on fiber optics for Burle facility. Three 365 nm, 405 nm and 470 nm Mic-LEDs are used as light sources. The beams are introduced into Burle growth facility through the high temperature optical fiber and illuminate three different positions to obtain average measurement results.

In the UHV growth and characterization facility, the fiber optic system also provides an easy way for *in situ* optical and electrical characterizations during the growth process. The collimated beam is introduced into the facility through glass windows and the electrical signals generated from the cathodes are collected through the electrical feedthroughs.

4. Optical Station Commissioning

It is essential to commission and benchmark the characterization facility. The results of two different methods will be described: 1) beam splitter ratio; and 2) comparison of QE measurements from a commercial PMT (Hamamatsu) with known QE characteristics.

4.1. Beam splitter ratio commissioning

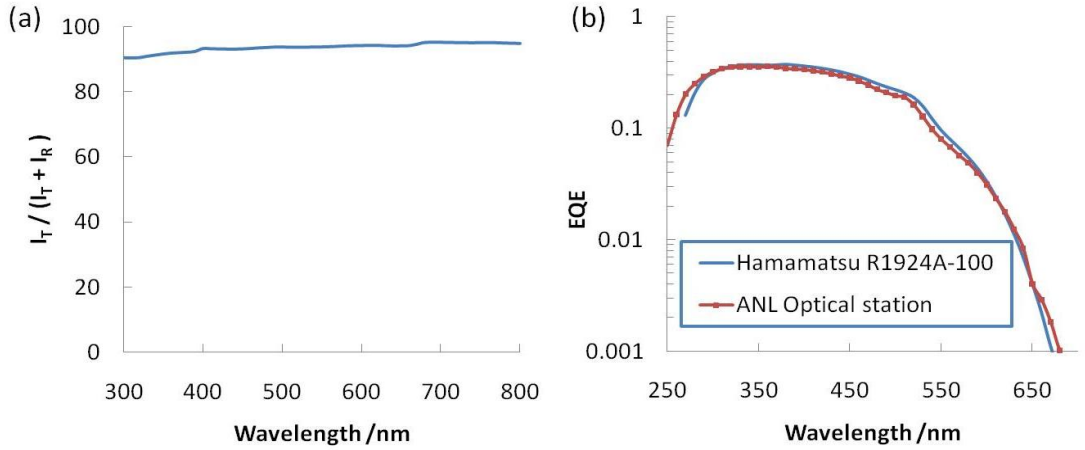

Fig. 4. (a) Percentage of incident (transmitted) beam out of the sum of incident beam and reference (reflected) beam intensity as function of wavelength. The percentage is between 90% - 95%, agreeing well with the expected percentage (90%) considering the variation at different wavelengths. I_T, incident (transmitted) beam intensity, I_R, reference (reflected) beam intensity. (b) Comparison of QE spectra of three PMTs measured by optical station and by Hamamatsu. They agree well with each other.

A beam splitter is used to separate the original collimated beam into two portions with a 90:10 Transmission: Reflection ratio so that the incident beam intensity which illuminates the photocathode can be monitored as the measurement goes on. This enables monitoring of the beam intensity during measurement. Two NIST traceable silicon photodiodes are placed in front of the transmitted and reflected beams. The beams are centered on the photodiodes and the beam intensities are measured. The ratio of incident beam intensity to reference beam intensity (Fig. 4(a)) is nearly 90:10, which agrees well with the expected ratio.

4.2. Benchmarking with the Hamamatsu commercial photomultiplier tubes:

A Hamamatsu commercially available photomultiplier tube (R1924A-100) with QE 35% at 380nm was purchased and used for the QE reference measurement. The QE spectrum of the PMT cathode as a function of wavelength is measured with the custom built optical station and compared with the QE spectrum obtained from Hamamatsu. The results show good agreement between the optical station measurement and the Hamamatsu measurement as shown in Fig. 4 (b).

5. First Results of PMT Cathodes

In many cases, the most important property of a PMT is its quantum efficiency as a function of wavelength. Thus, a measurement of the QE becomes our first goal. However, a lot of other optical parameters can also be obtained easily, such as reflection and transmission properties. Initial optical and electrical measurements of some commercial PMTs were performed at ANL as a test of the optical station.

5.1. Current-Voltage Behavior

Current-voltage (I-V) behavior of three PMTs successfully fabricated with the commercial PMT set up was measured using the optical station. The dark currents of the photocathodes are at the pA (10^{-12}) level.

At a fixed wavelength (400 nm), the photocurrent increases with increasing bias voltage and flattens above 10 V. Fig. 5 (a) shows a typical I-V characteristic curve for three PMTs deposited using the commercial PMT set up.

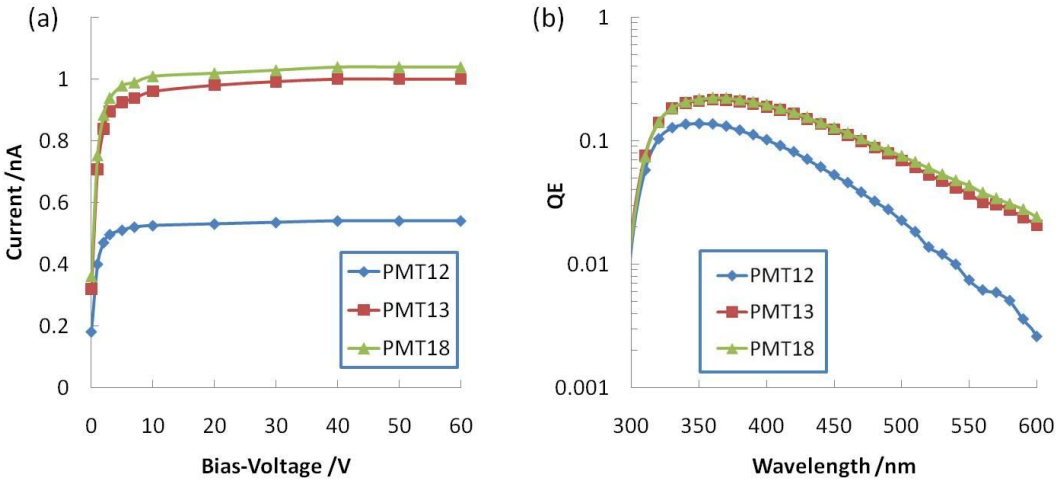

Fig. 5. (a) I-V curve at 400 nm for three PMTs deposited using the Burle facility. The results show typical cathode I-V behavior. (b) QE of three bi-alkali PMT cathodes in the range (300-600 nm), the QE is between 14~23% at 360 nm.

5.2. External Quantum Efficiency Measurement

Since we cannot simultaneously measure the incident beam intensity on the PMT glass surface and the photoelectron current, we calculate the number of photons in the incident beam indirectly. First, we measure the ratio of the incident (transmitted) beam intensity to the reference (reflected) beam intensity as a function of wavelength as shown in Fig. 4 (a). During the QE measurement, for each wavelength, the reference beam intensity and the cathode photoelectron current induced by the incident beam are measured together. Using the curve in Fig 4 (a), we calculate the incident beam intensity and number of photons. The number of photoelectrons is calculated by the directly measured photoelectron current. The external QE of the PMT cathode is then calculated and plotted as a function of wavelength, as shown in Fig. 5 (b). The QE for these three PMTs are between 14% and 23% at 360 nm and decrease at the UV and IR ranges. Here, we must state that no correction has been made for the reflection and absorption losses of the PMT glass window. The fast drop rate in the UV range is partially due to the absorption of the PMT glass window in that range.

6. Conclusion

We have designed and are commissioning a photocathode growth and characterization laboratory. Many different characterization methods are planned for the UHV chamber for *in situ* characterization. A movable optical station can be shared between *in situ* and *ex situ* optical and electrical measurements. It has been commissioned with Hamamatsu standard PMT cathodes. The fiber optics system allows for *in situ* monitoring of the optical and electrical properties during deposition process in both the commercial PMT set up and the UHV growth facility. The first measurements of PMT cathodes grown using the

commercial PMT set up were performed. The results show the expected cathode I-V behavior and the QE of these PMT cathodes is between 14~23% at 360 nm.

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References

[1] Alimonti G. et al. *Astrop. Phys.* 2002;**16**:205-234.

[2] Eguchi K. et al. *Phys. Rev. Lett.* 2003;**90**:021802.

[3] Iams, H., Salzberg, B. *Proceedings of the IRE* 1935;**23**:55-64.

[4] Görlich, P. *Zeitschrift für Physik* 1936;**101**: 335-342.

[5] Mirzoyan R., Laatiaoui M., Teshima M. *Nucl. Instr. and Meth. A* 2006;**567**:230–232.

[6] Ghosh, C., Varma B. P. *Thin Solid Films* 1975; **29**: 1-6

[7] Yusof Z. et al. Development of an alkali transfer photocathode for large area microchannel-plate-based photodetectors; this conference proceedings.

[8] Lee S.W. et al. Revealing the Correllation between Growth Recipe & Microscopic Structure of Multi-alkali Photocathodes; this conference proceedings.