



Improved direct detection of low-energy ions using a multipixel photon counter coupled with a novel scintillator



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ABSTRACT

Recently, we described a direct low-energy ion detector for time-of-flight applications based on a single-photon avalanche diode (SPAD) array optically coupled to an LYSO (cerium-doped lutetium yttrium orthosilicate) scintillator. Here, we present a greatly improved version of the detector, developed through testing of a number of different scintillator and phosphor materials in combination with a commercially available SPAD array. The various scintillator materials have been characterized in terms of the achievable detection sensitivity and time response when used in conjunction with the SPAD array. Organic parapolyphenylene dyes, a relatively new class of scintillator for particle detection, were found to exhibit markedly improved performance relative to well-established scintillators such as LYSO crystal scintillators and P47 phosphors, both in terms of brightness and time response. The optimized detector has a time resolution of ~ 2 ns, an improvement of more than an order of magnitude over the original version, at the same time as achieving a five- to ten-fold improvement in detection sensitivity. Our approach points the way towards the development of a generic silicon-based ion detector technology to complement or replace the microchannel-plate (MCP) ion detectors in widespread use at present. Such a detector would remove the need for high voltages and high vacuum, and at the same time greatly reduce detector cost relative to MCP-based detectors.

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1. Introduction

The ability to measure the arrival time of a charged particle with high sensitivity and high precision is a key experimental requirement in many areas of chemistry and physics. Detectors with such capabilities are required in areas ranging from gas-phase reaction dynamics [1,2] and particle physics [3] to medical imaging [4]. However, perhaps the largest area of application is time-of-flight (ToF) mass spectrometry, in which precise and accurate arrival time measurements must be made for ions accelerated to energies of up to 20 keV.

In addition to exhibiting high sensitivity and time resolution, an ideal ToF detection system should have a high saturation threshold, a mass-independent response, and be sufficiently robust to resist damage under a range of operating conditions. Low maintenance requirements and/or replacement costs are also desirable.

The simplest ToF detectors rely on direct charge detection at a Faraday cup or plate. While offering universal detection for particles of any mass, the ion currents recorded are generally at the sub-femtoampere to picoampere level, yielding low sensitivity. This is true even with substantial amplification, which is often achieved at the expense of time resolution. More commonly, the detector is comprised of a particle multiplier or one or more microchannel plates (MCPs) [5,6]. An ion incident on such a detector elicits a cascade of electrons, yielding a signal amplification of several orders of magnitude. For example, one of the most commonly used detector configurations comprises a pair of microchannel plates mounted in a chevron arrangement, yielding a charge amplification of around 10^6 . The readout system for an MCP-based detector typically consists either of a metal anode coupled to a fast current amplifier, or (particularly for imaging experiments) a phosphor screen coupled either to a photomultiplier and associated amplification electronics or to a CCD or CMOS camera. The intrinsic response speed of a microchannel plate is on the order of 100 ps or less [7], and this has been realized in a number of cases; but the overall time response of an MCP detector is often limited either by the time response of the amplification electronics or by the decay lifetime of the phosphor, if one is present [8].

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Despite the fact that MCP-based detectors have become the standard in ToF mass spectrometry, they suffer from a number of shortcomings. They are both expensive and extremely fragile, a less than ideal combination. Reliable operation is only possible at pressures below 10^{-5} mbar or so, with operation at higher pressures resulting in electrical arcing that can cause catastrophic damage to the plates. They also have a maximum output current [9], which sets a limit on data acquisition rates at the level of around 10^8 – 10^9 counts $\text{cm}^{-2} \text{s}^{-1}$. Finally, the secondary electron emission from the MCPs does not scale linearly with the kinetic energy of the detected particles, but instead scales with their velocity [10]. In ToF mass spectrometry, this has the consequence that because the ion velocity scales as the square root of the mass, the detection efficiency decreases with increasing ion mass. This effect can be mitigated to a reasonable extent by the use of suitable discrimination and pulse-counting electronics, so that the recorded signal depends only on the number of recorded ion events rather than on the intensity of the signals they generate. However, at some point, the intensities of the signal peaks generated by the MCPs at very high ion masses will fall below the discriminator threshold, and detection will therefore fail.

High-energy charged particles (MeV or higher) are readily detected using a combination of a scintillator and a photomultiplier tube. In the past, this has not been a practical approach to the detection of much lower energy (0–10 keV) particles, due to the very low conversion efficiency of scintillators in this energy range. Low-energy ions are stopped within the first few nanometers of the scintillator surface, generating only a handful of photons per keV, and yielding signals that are too small to be useful. However, the relatively recent development of silicon detectors capable of single-photon detection has made solid-state direct ion detectors an avenue worthy of further exploration. In particular, the development of in-pixel single-photon avalanche diodes (SPADs) opens up the intriguing possibility of single-photon-sensitive imaging detectors.

Recently, we reported a first prototype of a direct ion detection system [11], comprising an LYSO ($\text{Lu}_{1.8}\text{Y}_{0.2}\text{SiO}_5(\text{Ce})$) scintillator crystal coupled to a commercially-available SPAD array detector. The detector was used to record ToF mass spectra for butanone and carbon disulphide, and the response of the sensor was characterized as a function of ion kinetic energy, showing a linear increase that reflected the expected photon-emission characteristics of the LYSO scintillator. The work represented a very promising stepping stone on the path towards a generic silicon-based ion detector, which would remove the need for high voltages and high vacuum and at the same time greatly reduce detector cost relative to MCP-based detectors. However, the prototype detector exhibited relatively low detection sensitivity, and the time resolution was limited by the ~ 40 ns decay lifetime of the LYSO scintillator.

In the present work, we test a number of alternative scintillator materials, and show that by taking advantage of recently developed fast scintillator screen materials [12], we are able to improve both the detection sensitivity and the time resolution of the direct ion detector by around an order of magnitude.

2. Materials and methods

The prototype detector and time-of-flight mass spectrometer used in the characterization experiments have been described in detail in earlier publications [11,13]. The sensing element in the detector is a Hamamatsu multipixel photon-counting sensor (MPPC S10362-11-025C), comprising a 1 mm^2 , 1600-pixel array of single-photon avalanche photodiodes (SPADs) arranged on a $25 \mu\text{m}$ pixel pitch. The individual SPADs are connected together in parallel, such that the sensor output is the sum of the individual pixel outputs. At present, individual pixels cannot be addressed independently,

so imaging experiments are not possible and the sensor simply records the total incident photon signal. The sensor is most sensitive to photons in the wavelength range from 390 to 470 nm, with maximum sensitivity at ~ 420 nm. To create the direct ion detector, the MPPC sensor is optically coupled to a scintillator material. In the original experiments [11], this was a $1.5 \text{ mm} \times 1.5 \text{ mm} \times 0.2 \text{ mm}$ LYSO scintillator crystal, mounted onto the front face of the sensor with a small amount of optical grease. In the present work, we have tested a number of different scintillator materials. These allow both the sensitivity and timing precision of the detector to be tuned. The intrinsic timing precision of the SPAD array is on the order of 200 ps, with a reset time for each pixel of around 20 ns, but the time resolution of the complete detector will in general be limited by the decay lifetime of the scintillator material.

In addition to repeating measurements with an LYSO scintillator for comparison with our previous study [11], the detector was tested with Exalite 389, Exalite 404, BBOT (2,5-di-(5-tert-butyl-2-benzoxazolyl)-thiophene), P47, and Coumarin 503 scintillators. The performance of several of these scintillator materials in an MCP/scintillator-based particle imaging detector has been discussed in detail in a previous publication by the present authors [12]. In the MCP-based detector, the Exalite scintillators in particular were found to outperform P47, exhibiting higher brightness and improved time resolution. None of these scintillators are available as single crystals, and instead they were prepared as thin layers on microscope cover slips, which were then attached to the front face of the MPPC sensor using a small amount of optical grease. The cover slips were 0.5 mm in thickness, and were cut to dimensions in line with that of the LYSO crystal. Thin layers were prepared using one of two different approaches [12], depending on the chemical and physical properties of the scintillator material.

Exalite 389, Exalite 404, and BBOT were deposited on the substrate by vacuum sublimation. The substrate was mounted on the cold finger of a commercially available vacuum sublimation apparatus (Ace vacuum sublimation apparatus, inner diameter 60 mm), and 0.1 g of the scintillator material was placed in the base of the apparatus. The cold finger was filled with a water/ice slush; the apparatus was placed on a hot plate, and the scintillator material was then heated to the onset of sublimation at a pressure of 15 mbar until all of the material had sublimed and deposited on the surface of the cold finger. After complete sublimation, a layer of scintillator approximately $200 \mu\text{m}$ ($\pm 10\%$) in thickness, as measured with a micrometer, had formed on the substrate.

P47 (95% of particles $< 7.2 \mu\text{m}$) and Coumarin 503 scintillator screens were prepared via a sedimentation process. The substrates were placed in a beaker tilted at an angle of around 8° . A solution of barium chloride (1.16 mM in deionized water) was added to the beaker until the substrates were submerged beneath 5 cm of solution. 0.15 g of the scintillator was suspended in a 0.37 M potassium silicate solution and slowly poured into the prepared beaker with the substrates. After a sedimentation period of 20 min, the solution was siphoned from the beaker and the coated substrates were dried in an oven at 60°C for 20 min. This method yielded a layer thickness of approximately $350 \mu\text{m}$ ($\pm 10\%$), measured using a micrometer.

To interface with the mass spectrometer, the detector (MPPC with attached scintillator) was mounted on the vacuum side of a DN40 ConFlat flange, and connected to its control module (Hamamatsu C10507-11-025U MPPC control module) outside the vacuum via a six-pin electrical feedthrough. USB interface software provided by the manufacturer was used for data acquisition, with the option of viewing and acquiring either the analogue or digitized (photon counted) signal from the sensor. All experiments reported here utilized the analogue output. This was viewed and recorded using a fast digital oscilloscope (LeCroy DDA-260).

To produce an ion signal with which to characterize the detector, a pulsed molecular beam containing the sample molecule of

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