Low background, UHV compatible scintillator detector for the CLS cryo scanning soft X-ray microscope

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Abstract. A new soft X-ray scanning transmission X-ray microscope (STXM) optimized for cryo spectro-tomography was designed and commissioned at the Canadian Light Source (CLS). The instrument was required to achieve ultra high vacuum and be compatible with in-situ plasma cleaning. It also required a scintillator detector, and the design of this detector had to evolve to meet these environmental requirements. The scintillator deposition technique, and the suppression of background by introduction of an edge filter are also presented.

1. Introduction

Soft X-ray (100 – 2000 eV) scanning transmission X-ray microscopy (STXM) is an established technique with many instruments operating around the world and more under construction [1]. One advantage of STXM over complimentary techniques such as scanning electron microscopy (SEM) or electron energy loss in transmission electron microscopes (TEM-EELS) is relatively low dose interrogation [2], which has enabled investigations of radiation sensitive samples such as organics. One of the earliest detectors developed for STXM was a scintillator-photomultiplier tube (PMT) [3-5] placed behind the sample to measure transmitted X-rays. Although many other detectors have been implemented in STXM over the years, the PMT detector remains in wide use because it still has the highest counting efficiency of any transmission detector below about 600 eV, fully covering the water window. The PMT detector is and has been the default transmission detector for the ambient-STXM at the Canadian Light Source (CLS) Spectromicroscopy beamline across the full photon energy range.

A new STXM, optimized for cryo spectro-tomography (cryo-STXM), was recently designed and commissioned at the CLS. Buildup of ice and carbon contamination on the cryo-cooled sample would negatively affect the experiments, so the instrument was designed to achieve ultra high vacuum (UHV), and be compatible with in-situ plasma cleaning. The cryo-STXM required a PMT detector capable of the same environmental requirements. Modification of an existing PMT detector design [5] was necessary to achieve UHV and plasma compatibility, without sacrificing efficiency or signal to noise performance.

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2. Materials selection for UHV and plasma environment

The overall mechanical and optoelectronic design is centered on the proven Hamamatsu R647P PMT, with new emphasis on materials to meet the goal of UHV and plasma compatibility (Figure 1). A Hamamatsu C9744 photon counting unit, C9727 power supply and a National Instruments PCIe-6351 data acquisition card complete the counting circuit.

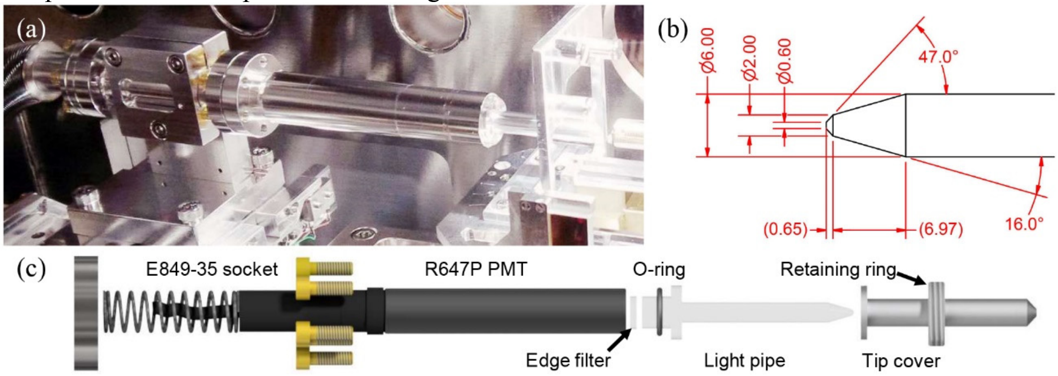

Figure 1. (a) Photograph of the assembled PMT detector installed in the cryo-STXM. Overall length from tip cover to bellows is 221 mm. (b) Dimensions of the light pipe tip in millimeters. (c) Exploded view drawing depicting the internal components.

The PMT is in air, inside a re-entrant housing to protect it from chamber process gasses, especially He which causes premature failure. The housing weldments are electropolished 304L stainless steel, while the tip cover and threaded retaining ring are electropolished 6061 Al. A stainless steel welded bellows is used, and the ConFlat® screws are Au rather than Ag coated, as Ag rapidly develops a black particulate Ag_2O powder in an oxidizing plasma. The light pipe material is borosilicate glass (Hilgenberg GmbH) with all optically active surfaces fire polished. Compared to poly(methyl methacrylate) (PMMA) which was used in the past, glass has superior vacuum properties. In addition, PMMA is etched by oxidizing [6] and reducing plasmas [7], whereas glass is practically inert. A small amount of DuPont Krytox® LVP lubricant was applied to the baked out (120°C for 48 h) brown Viton® o-ring before setting it in the vented groove. A thin coat of Dow Corning High Vacuum Grease was applied to both surfaces of the optical components, which are out of vacuum.

In this configuration, following CLS UHV cleaning and handling procedures, the cryo-STXM instrument with the assembled PMT detector installed has achieved pressures in the 10^{-9} Torr range without baking. It has also endured several 10 minute exposures to hydrogen plasma (GV10x, ibss Group) without any noticeable effects or changes to PMT performance. This PMT detector cannot be baked in the current configuration as the R647P tube cannot exceed 50°C. However, we plan to add air cooling of the tube housing weldment to permit baking. A glass-to-metal seal for the light pipe could further improve vacuum performance.

3. Scintillator deposition

P43 phosphor powder (2.1 μm median particle diameter, NP-1043-01, Nichia) was selected as the scintillator material as it exhibits good all-around performance and high efficiency [5,8], and has proven reliable in the ambient-STXM over a decade. To deposit the loose white P43 powder, a vanishingly thin layer of Dow Corning High Vacuum Grease is transferred to the light pipe tip. The tip is then plunged into a vial of desiccated P43. Puffs of compressed air are used to remove excess powder while periodically inspecting the uniformity and thickness of the layer using an optical microscope. The layers produced by this method are inconsistent. The quality of the scintillator layer has a very significant impact on the detector efficiency, so we explored many other methods including dipping, settlement and evaporation from suspension [9], and thermal evaporation. After review, we find the best method is still plunging the greased tip into desiccated scintillator powder. We compared optical micrographs to raster scanned detector images to determine the characteristics of a well performing layer (Figure 2).

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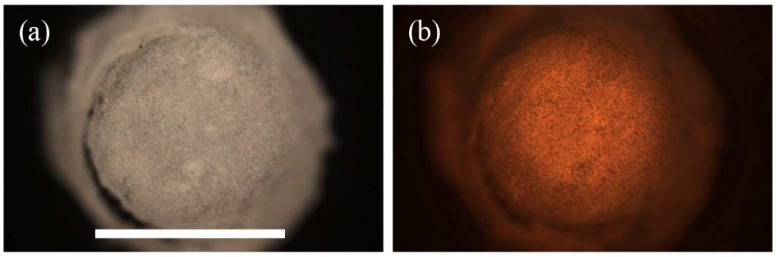

Figure 2. Optical micrographs of a well performing P43 layer in (a) reflection and (b) transmission, 200× magnification, scale bar = 600 µm. A uniform layer a few grains thick was found to be optimal.

After optimizing the deposition method, we compared the efficiency to the ambient-STXM PMT detector. These experiments were performed in the ambient-STXM using the same counting circuit and settings for both detectors. The efficiency of the cryo-STXM PMT detector relative to the ambient-STXM PMT detector was measured to be 77% at 320 eV (absolute efficiency (AE) ~30%), 85% at 395 eV (AE ~50%), and 104% at 700 eV (AE ~60%). A further study of the absolute efficiency is planned.

4. Suppressing background with an edge filter

The sensitivity curve of the R647P PMT spans 300 – 650 nm. It is sensitive to the visible light from the scintillator (signal), as well as any other light present inside the cryo-STXM chamber (background), including scattered laser light from the interferometer. This background has a negative effect on the limit of detection for optical density measurements and spectroscopy. The tip cover partially intercepts background light, and the sample and order sorting aperture also act as light baffles. Purpose-made light baffles are implemented in other STXMs. It is not practical to add a visible light filter over the tip cover aperture (*i.e.*, in the X-ray path) because the soft X-rays would be strongly attenuated and the signal would suffer. However, we hypothesized that it would be possible to suppress the laser light portion of the background by inserting an edge filter between the light pipe and the PMT (Figure 1).

The wavelength of the cryo-STXM interferometer is 632.8 nm (HeNe). The calculated transmission spectrum of the edge filter (SP01-633RU-9.7-D, Semrock) is presented in figure 3a. The cut off wavelength is dependent on the angle of incidence (AOI) and polarization, but the transmission of 632.8 nm light at AOIs from 0 – 28° is practically zero. The X-ray excited optical luminescence (XEOL) spectrum of P43 was measured at the CLS Spherical Grating Monochromator (SGM) beamline and is presented in figure 3b.

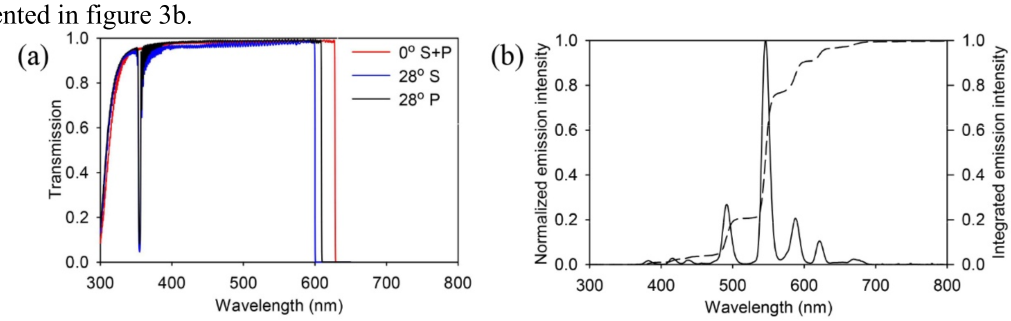

Figure 3. (a) Calculated transmission spectrum of the edge filter for different angles of incidence and polarization: 0° S and P (red), 28° S (blue), 28° P (black). (b) Measured XEOL spectrum of P43 phosphor (solid line), and the integrated spectrum (dashed line).

We measured the XEOL spectrum using excitation energies of 300, 500, 800, 1200, and 1800 eV. All of these spectra were identical. Visually the emission appears bright green with λ_{max} at 546 nm, and 91% of intensity is below 600 nm. Knowing the spectral properties of the edge filter and of P43, the geometry of the light pipe was designed so that 1) light emitted into the light pipe will totally internally reflect down to the PMT, and 2) this light will reach the edge filter with an AOI less than 28°.

Experiments were performed to quantify the effect of the edge filter on the signal and the background (Table 1). First, the PMT power supply voltage and the discriminator were optimized following manufacturers procedures. The counting card accuracy was found to be perfect using a pulse generator. Internal components including the zone plate, order sorting aperture, and sample plate were placed in actual measurement positions. There are no purpose-built light baffles in the instrument.

Table 1. Effect of the edge filter on background and signal measured in the cryo-STXM.

Conditions	Background (Hz, /10)	Signal + background (Hz, /10)
Filter out, laser on	940	2.31×10^5
Filter out, laser off	14	2.31×10^5
Filter in, laser on	60	2.04×10^5
Filter in, laser off	14	2.04×10^5
Filter in, laser off, tip aperture blocked	14	N/A

Some counts were observed when the tip cover aperture was physically blocked by two layers of Al foil. We attribute these to electronic noise (cables, thermal electrons within the PMT, etc.). When the lasers were switched off, the count rate was identical to the physically blocked trial; it appears that the lasers are responsible for all light-based detector background in the cryo-STXM. The edge filter reduced background counts by a factor of 15, but it did not eliminate all laser light. This may be due to fabrication tolerances of the light pipe or the edge filter. The edge filter reduced the real signal by 13%.

The edge filter is a coating which could be directly deposited onto the light pipe or PMT. This would ease assembly and could improve the detector efficiency. It may also be beneficial to employ an interferometer with a laser wavelength outside the sensitivity curve of the PMT in future STXMs.

5. Conclusion

A scintillator-PMT detector was fabricated for the CLS cryo-STXM. The instrument including the detector has achieved pressures in the 10^{-9} Torr range without baking, and endured multiple exposures to hydrogen plasma. The detector efficiency was found to be similar to the ambient-STXM PMT detector. Introduction of an edge filter was found to reduce the detector background by a factor of 15.

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