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Scintillator Detectors for Scanning Transmission X-ray Microscopes at the Advanced Light Source

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Abstract. The choice of x-ray detectors presents a challenge for scanning transmission x-ray microscopes (STXM). The ultimate detector for the ALS STXMs should have a very high efficiency over a wide energy range, single photon counting capabilities up to 1GHz, high rejection of red laser light, fast response, and work in both vacuum and at atmospheric pressure. The results of an investigation using a photomultiplier tube (PMT) with a number of different scintillators are presented. The PMT is used in photon counting and analog modes. Scintillators used for this study include commercially available phosphor powders and various single crystals.

INTRODUCTION

Scanning transmission x-ray microscopes working at soft x-ray energies present a challenge for the choice of x-ray detectors. At the Advanced Light Source, home of the MES 11.0.2 and Polymer 5.3.2 STXMs [1], there is an ongoing quest for better detectors. Higher detection efficiency is crucial as it allows faster data acquisition or to work at higher spectral resolution. It also reduces radiation damage often observed on organic samples. On the Polymer 5.3.2 STXM, radiation damage on polymers and organic samples and lower flux (bending magnet beamline) requires efficient detection. On the MES 11.0.2 STXM, preventing damage on biological and environmental samples due to the very high flux also requires fast and efficient detection.

The ultimate detector for the ALS STXMs should have a very high efficiency over a wide energy range, typically from 150 eV up to 2000 eV, single photon counting up to 1GHz, high rejection of parasitic visible light (scattered laser light from the interferometer), fast response, and work in both vacuum and atmospheric pressure.

EXPERIMENTAL DETAIL

The working principle for scanning transmission x-ray microscopy is relatively simple. X-rays are focused onto a sample by a zone plate lens to about 40 nm, the sample is raster-scanned in the focal plane and the transmitted photons are detected by a stationary detector. An order sorting aperture (OSA) reduces the diffracted beam to the desired diffracted order (usually 1st).

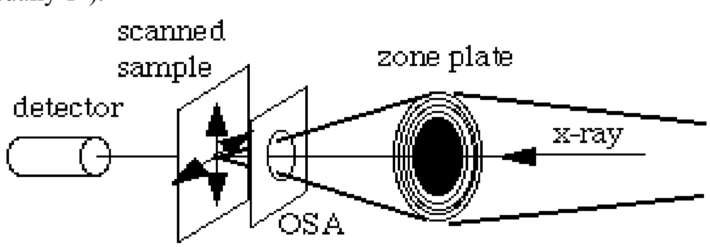

Figure.1. Schematic diagram of a scanning transmission x-ray microscope

The main type of detectors used at both ALS scanning microscopes is a scintillator - light pipe - photomultiplier tube assembly. The inorganic scintillator presently used is a phosphor that converts x-rays into visible light. A lucite

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light-pipe conveys visible photons onto the PMT (Fig.2.). The visible photons are counted using a photon counting unit with count rate capability in the tens of MHz. The PMT is also used in analog mode. The relatively long light pipe is used for two reasons. First, the detector has to fit in the piezo scanning stage frame to allow large scanning range. Second, the PMT has to be far away from the sample in order to reduce the influence of the magnetic field created by a magnet used for sample magnetization measurements. The x-ray beam size is about 0.2 mm to 0.4 mm in diameter at the detector position. Two different light pipe tip shapes are used; flat tip for use with single crystal scintillators and a conical tip with a 1 mm flat top when a phosphor powder is used.

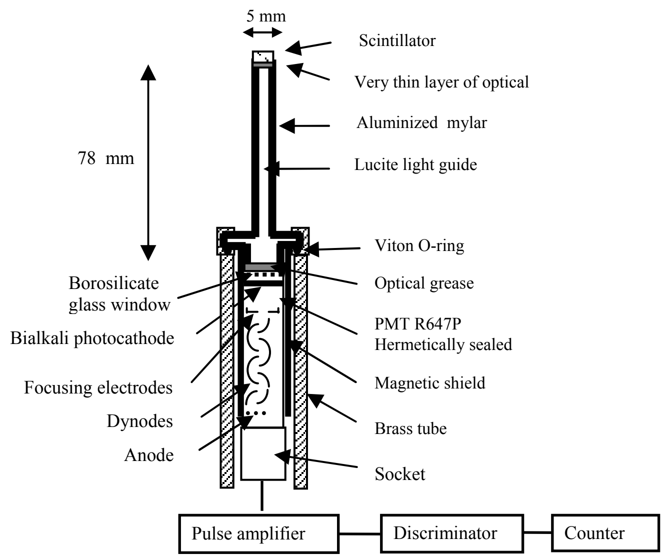

Figure 2. STXM photomultiplier tube detector configuration.

The usefulness of this detector depends upon the properties of the scintillator chosen for the STXM detector. Three single crystal scintillators and three powder phosphors were compared (Table 1). The large scanning area (up to 25 x 4 mm) of the STXM offered unique possibilities to compare those scintillators. The scintillator were mounted on a very thin glass slide (200 μm thick) and scanned in the focused x-ray beam while the luminescent photons were detected with a PMT detector without a scintillator. The angular acceptance angle for the visual photons was kept constant for all scintillators. The exposure time was 1 ms per pixel unless otherwise noted.

Type	Composition	Wavelength of maximum emission	Decay time
P47	Y_2SiO_5:Ce	400nm (blue)	55 ns
P43	Gd_2O_2S:Tb	545nm (green)	1 ms
YAP:Ce	$YAlO_3$:Ce	370nm (blue)	27 ns
YAG:Ce	$Y_3Al_5O_{12}$:Ce	550 (green)	70 ns
CWO	$CdWO_4$	470nm (blue)	28 us

Table 1. Scintillators used in this work [2].

RESULTS

Figure 3a shows an image of 3 different single crystal scintillators obtained at 700 eV. One can clearly see that the YAG crystal is more efficient than the YAP and CWO crystals. There is also evidence, that the single crystals do not emit homogeneously. The visual light microscope inspection showed that the spots with higher luminescent emission had a higher quality polished surface. Figure 3b shows relative luminescence from those three scintillators as a function of the x-ray energy.

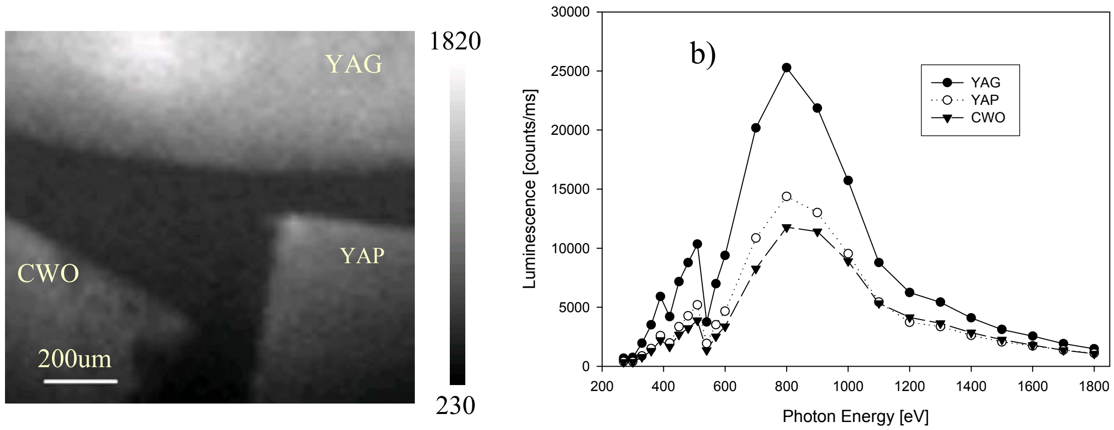

Figure 3. (a) Luminescence image of YAG, YAP and CWO scintillating single crystals recorded at 700 eV. (b) Relative luminescence intensity as a function of x-ray photon energy.

Figure 4a shows luminescent images of different phosphor powders. The P47 has significantly higher luminescence then the others. Quantitative comparison has to account for the fact that the P47 powder grains are relatively sparsely distributed and cover only a small percentage of the area. Using only single grain comparison of the energy dependence of the luminescence (Figure 4b), the significance of this problem was reduced.

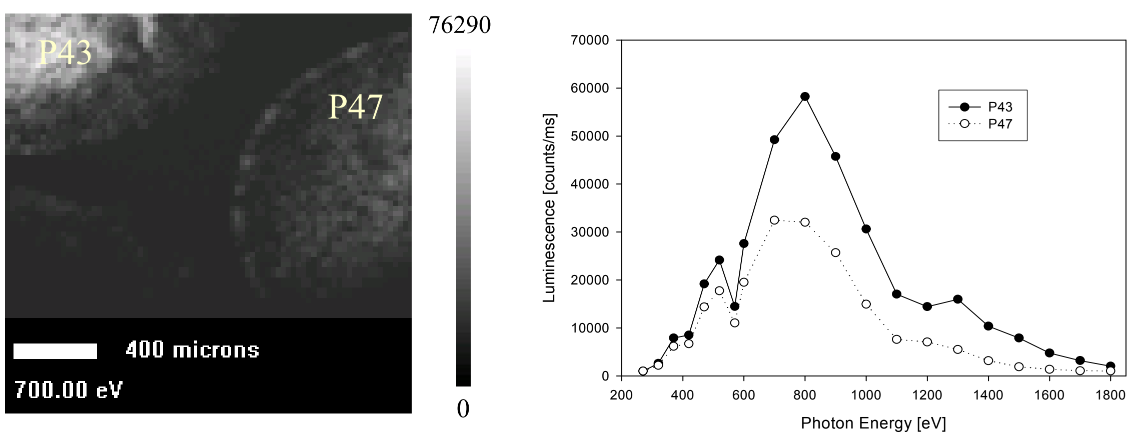

Figure 4. (a) Luminescence image of phosphor powders. (b) Relative luminescence intensity of single grains as function of x-ray photon energy.

Figure 5 shows the P47 and P43 phosphor grains in higher magnification. What is characteristic of the powders, is that the luminescence is not homogeneous. It depends on the shape and size of individual grains as well as on how the grains are joined together. On a microscale, it is probably not possible to make a thin phosphor layer with homogeneous sensitivity to soft x-rays.

An interesting observation was that the P47 phosphor lost luminescence intensity with prolonged exposure to x-rays. With photons of 640 eV energy and flux of about 5 x 10^8 ph/s in a 40 nm size spot, the luminescence decreased about 30% after a 10 min exposure.

Another important characteristic of the scintillators should be considered when making a choice for a STXM detector - decay time. The ALS STXMs can scan as fast as 50 µs dwell time per pixel. A scintillator decay time should be of the same scale or shorter. All of the scintillators studied have shorter decay times except the P43 phosphor, which has a decay time of about 1 ms. To determine how much distortion to expect from this, we have compared luminescent images of P43 and P47 phosphor grains recorded at about 1 ms pixel dwell time to those recorded at much faster scanning speed with 50 µs (P43) and 80 µs (P47) dwell time (Figure 5).

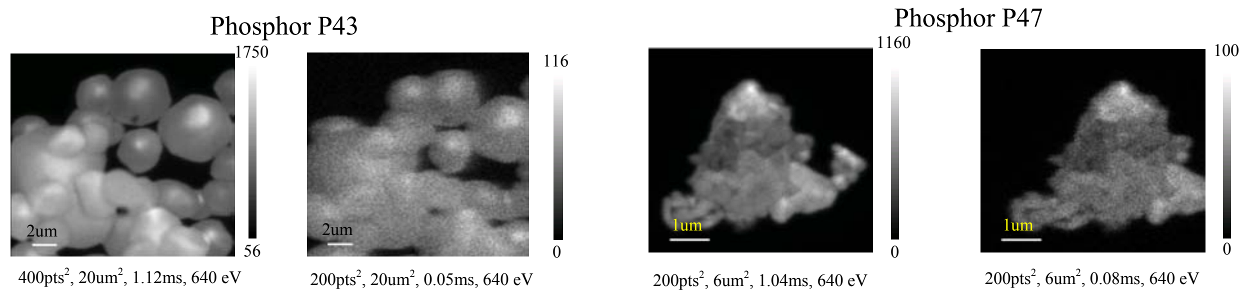

Figure 5. Luminescence images of P43 and P47 phosphor powders recorded at slow and fast scanning speeds. The P43 displays low intensity shadows on the right hand side of individual grains at high scanning speed.

Figure 6 shows scanning profiles of phosphor grains from images shown in Figure 5. Fast scans of the P43 grain exhibit distortions due to slow decay. A "tail" in the profile of grain luminescence is equivalent to the decay time of about 200 μs.

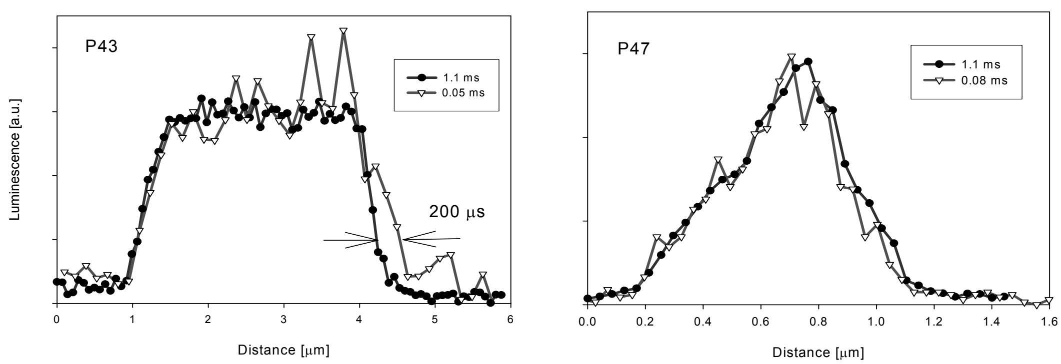

Figure 6. Normalized scanning profiles of P43 and P47 phosphor particles from slow and fast scans.

CONCLUSION

In the scanning transmission x-ray microscope application, the detector efficiency is more important than the homogeneity of its sensitivity. Thus, powder phosphors are a better choice as a scintillator than single crystals. In the case of imaging radiation sensitive samples the P43 phosphor is a preferred choice. When the samples are not so sensitive to radiation damage and there is sufficient photon flux to use very fast scanning speeds, the P47 phosphor should be used.

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