Quantum Dots Microstructured Optical Fiber for X-ray Detection

S.L. DeHaven \(^{1a}\), P.A. Williams \(^{1b}\), and E.R. Burke \(^{1c}\)

\(^{1}\)NASA Langley Research Center, Hampton, VA 23681

\(^{a}\)Corresponding author: stanton.l.dehaven@nasa.gov

\(^{b}\)phillip.a.williams@nasa.gov

\(^{c}\)eric.r.burke@nasa.gov

Abstract. A novel concept for the detection of x-rays with microstructured optical fibers containing quantum dots scintillation material comprised of zinc sulfide nanocrystals doped with magnesium sulfide is presented. These quantum dots are applied inside the microstructured optical fibers using capillary action. The x-ray photon counts of these fibers are compared to the output of a collimated CdTe solid state detector over an energy range from 10 to 40 keV. The results of the fiber light output and associated effects of an acrylate coating and the quantum dots application technique are discussed.

INTRODUCTION

The development and improvement of x-ray detectors for various applications continues with the introduction of quantum dots. The quantum dots, comprised of nanophosphors, offer several benefits due to their synthesis and materials. These include custom nanophosphors and dopants for specific applications, the ability to be dispersed due to their small size, and unique physical properties [1]. Additionally, quantum dots acting as radio luminescent phosphors can provide biological and medical uses associated with scintillation wavelength shifts from the variation of dopants [2].

The quantum dots in this work act as scintillators or radioluminescent phosphors. Scintillators are generally comprised of two types, organic and inorganic. The quantum dots used are an inorganic scintillator, due to their material composition of ZnS(MgS). The typical inorganic scintillators are NaI(Tl) and CsI(Tl), with NaI(Tl) as the standard scintillator.

Another primary type of x-ray detector is a semiconductor or solid state electronic type. Semiconductor detectors generate current when x-rays interact with the material. The solid state detectors have several benefits compared to scintillators but need an evacuated space behind a beryllium window and require cooling for dark noise considerations [3].

A conventional fiber for x-ray detection is plastic PMMA scintillating fiber. The plastic fiber uses an organic scintillator mixed with a polymer as the core. The plastic has relatively high absorption losses for visible light which requires the core to be 1000 microns in diameter [4].

More recent work in scintillating fibers is the introduction of organic and inorganic scintillators into quartz microstructured optical fiber [5-8]. These fibers operate on total internal reflection. Other fibers have been used which contain scintillation (radioluminescent) core glass as part of scintillating glass fiber optical faceplates [9].
The novel approach presented in this paper is combining a quantum dots scintillator with microstructured optical fiber to form an x-ray detector by placing quantum dots inside the microstructured fibers quartz matrix. The light guidance in the fiber is described through total internal reflection theory. This theory is confirmed by observation of the visible light transmission in the microstructured optical fiber from the quantum dots.

A solid state CdTe x-ray detector is used to characterize the x-ray tube photon counts vs energy over an energy range from 10-40 keV and compared with the optical fibers output. The geometry of the microstructured optical fiber is shown in Figure 1. Each inclusion, or hole, is 2.5 microns in diameter with 138 inclusions in the fiber. The outside diameter of the fiber is 125 microns, which allows connection to standard fiber optics.

**FIGURE 1.** Microstructured optical fiber end SEM image of 138 inclusions, or holes, which extend the length of the fiber taken at 30 keV. Quantum dots scintillation materials are placed in these inclusions to form an x-ray detecting optical fiber.

An experimental technique measures the photon counts from the quantum dots optical fiber when exposed to x-rays. These photon counts are compared with the x-ray counts of a CdTe detector. This measurement allows calculating an x-ray detection efficiency of the quantum dots microstructured optical fiber (MOF). The results of this measurement and experimental technique are discussed.

**THEORY**

The scintillating quantum dots used are comprised of zinc sulfide nanocrystals doped with magnesium sulfide, ZnS(MgS). The scintillation phenomenon occurs when an electron is temporarily placed in a higher energy level due to interaction with an x-ray and subsequently returns to a lower energy level with the release of visible light photons. Many scintillators emit the same wavelength of light when exposed to ultraviolet (UV) light; the ZnS(MgS) quantum dots behave as such and fluoresce when exposed to UV light. This allows viewing the fluorescence emission light from excitation by a UV source [5]. Figure 2 shows the UV photon absorption between 350-440 nm with an emission peak of 580-610 nm. This spectrophotometry analysis was performed for a UV light excitation wavelength of 385 nm with an emission wavelength of 585 nm for the ZnS(MgS) quantum dots in toluene.
using a Hitachi F4500 florescence spectrophotometer. The fluorescence with UV allowed photography and microscope observation of the quantum dots in the fiber to aid in understanding the light transmission for comparison with theory.

The x-ray detecting MOF contains quantum dots adhering to the 138 inclusions of the fibers quartz matrix. For light guidance, total internal reflection is the predominant mechanism where the center solid quartz element acts as a core and the surrounding quartz and air matrix acts as a cladding. Total internal reflection is light guidance in two dimensions and relies on the difference in refractive index between the solid quartz capturing the emitted light from the quantum dots and the air filled inclusions surrounding the core providing a lower index of refraction to guide the light. This is the same mechanism used to describe the x-ray output of liquid filled and radio luminescent core fiber but with different materials [5-9].

The quantum dots MOF presented in this work has scintillation light that is generated by the quantum dots and then captured by the microstructured quartz. The quantum dots emitted light is on the inside surface of the inclusions for ~ 2.5 cm of the fiber length. The surrounding of the core with scintillating quantum dots allows capturing scintillation light by the MOF core. This capture allows transmittance of the light along the fiber length. Figure 3 shows emission from the cross section of the quantum dots MOF when exposed to 365 nm UV light. Light is emitted from the end of the fiber containing only quartz and air.

**FIGURE 2.** Excitation-Emission spectra of ZnS(MgS) quantum dots in toluene from spectrophotometry. The peak emission is between 580-610 nm.

The transmission of light along the fiber length is from total internal reflection theory, based on the materials and geometry. Figure 4 shows the fiber end positioned for viewing using an optical microscope at 500x when the opposite end containing quantum dots is illuminated with a 365 nm UV light. The resulting image confirms that the
primary light transmission mode is through the fiber core in accordance with total internal reflection theory, due to the visible light intensity; the visible light shown is emitted from the end of the fiber containing only quartz and air.

**FIGURE 3.** (a). Microstructured optical fiber (MOF) containing quantum dots on a microscope stage. The 585 nm light is emitted from the fiber end by illuminating the quantum dots in the MOF with a 365 nm UV lamp source; (b). Side view of microstructured fiber inclusions containing quantum dots viewed at 500x magnification when using a 365 nm UV lamp source.

**FIGURE 4.** End view of microstructured optical fiber containing quantum dots at 500x magnification showing light transmission. The 585 nm light is transmitted along the fiber length primarily through the core in accordance with total internal reflection theory with the air/quartz cladding forming an effective index of refraction lower than the solid quartz core.
The energy range used in this work is 10-40 keV. Because of this lower energy range, all of the x-ray interaction with the quantum dots scintillator is considered absorption from the photoelectric effect. Absorption of the x-rays is based on Beer’s law along the axial fiber direction for a length of 2.5 cm. A fraction of the emitted photons are captured in the quartz and subsequently transmitted via the fiber.

**EXPERIMENT**

The quantum dots MOF’s were prepared by placing quantum dots in the fiber microstructure. Quantum dots suspended in a toluene solution were drawn into the microstructured inclusions of the fiber via capillary action. The toluene evaporated from the fiber end leaving quantum dots adhered to the quartz walls of the inclusions. A 365 nm UV light allowed quickly viewing the presence of quantum dots in the fiber. Figure 3 shows the quantum dots MOF illuminated by UV light to verify quantum dots placement before being exposed to x-rays. The fiber length was chosen as 12 cm with 2.5 cm of the fiber filled with quantum dots after the toluene was allowed to evaporate for 72 hours.

An experiment was constructed, similar to previous work, to measure the relative photon counts from the MOF with quantum dots [5-8]. The x-ray source was characterized using a CdTe detector and multichannel analyzer to collect counted x-ray photons at varying tube voltages. Measurements were taken between tube voltages of 10 kV to 40 kV at 5 kV intervals. The x-ray source used was a 40 kV, 4 W silver anode x-ray tube.

Figure 5 shows the experimental setup for the x-ray tube characterization and quantum dots MOF measurements. A collimator with 100 micron and 200 micron aperture tungsten disks 5mm thick are placed in front of the CdTe detector assembly to collect x-ray photons. This collimator and the selected aperture sizes are intended to represent the optical fiber for comparison purposes. During fiber measurement, a photon counting module (PCM) having a quantum efficiency of 65% at 585 nm collects light pulses from the optical fiber.

The CdTe detector is considered 100% efficient at detecting x-rays with energies between 10 keV and 50 keV. The multichannel analyzer counts the x-rays at discrete energies and allows creating an x-ray energy spectrum of the tube output. The CdTe detector and associated multichannel analyzer operate with software for data storage on a computer.

The x-ray characterization was performed prior to taking quantum dots MOF measurements. The tube characterization ensured tube stability and measurement repeatability. An americium-241 source was used to calibrate the CdTe detector energy. The collimator and tube were carefully aligned to maximize photon counts with the collimator placed 5mm from the tube output with the tube voltage varied at maximum tube current over five minute intervals.

The quantum dots MOF was placed in the same location as the CdTe collimator with the end containing quantum dots placed 5 mm from the x-ray tube. The x-ray tube voltage was varied with the tube current fixed at maximum output during five minute measurement intervals. The objective of the configuration was to expose the quantum dots MOF to the same x-ray photons and energies as the CdTe detector configuration. The MOF end void of quantum dots was coupled to a photon counting module FC fiber optic connector using a bare fiber adapter. The output from the module was sent to a digital counter to provide photon counts. The counts were recorded at tube voltages between 10 and 40 kV at 5 kV increments.

A black out cloth and a darkened room were used when taking data with the quantum dots MOF. The cloth was placed over a leaded Plexiglas three-sided enclosure having a lead foil covered aluminum back plate. Lead shielding was used over the PCM and in front of the optical fiber to reduce noise. The electronics power was provided with a common ground and additional electronics noise sources were suppressed. Evaluating the PCM counts with the x-ray tube current set to zero provided a baseline for photon counting at varying tube voltages.

A 30 micron thick piece of aluminum foil was placed over the tube outlet to filter out low energy x-rays below 10 keV. The fiber length was selected to approximate the collimator length with both the collimator and fiber positioned 5 mm from the tube during data collection. The fiber was aligned with the tube outlet to maximize the number of counts from the PCM.
FIGURE 5. Experimental configuration for characterizing the x-ray tube (top); the quantum dots x-ray detecting fiber measurements (bottom).

RESULTS AND DISCUSSION

Figure 6 shows the x-ray tube photon energy spectrum collected using the CdTe spectrometer with a 100 micron collimator for tube characterization. Tube characterization and fiber data were taken over five minute intervals. A 30 micron thick sheet of aluminum foil was placed in front of the tube as a low energy filter. These curves were integrated over each energy range to provide a total number of x-ray photons at each tube voltage after importing the data into MATLAB. This total number of x-ray counts was considered that of a 100% efficient detector, based on the CdTe’s 100% efficiency between the energy ranges of 10 keV to 50 keV.

Figure 7 shows the ratio of quantum dots MOF to CdTe counts as a percent efficiency vs tube voltage. The quantum dots fiber output exceeds 5% efficiency for all energies. However, this efficiency is non-linear which has not been observed in previous measurements. Both organic and inorganic scintillating materials have a linear or near linear efficiency vs tube voltage (or energy) relation [3, 5-8].

A significant factor in the quantum dots MOF is the ease of fabrication. Only capillary action is necessary to introduce quantum dots dispersed in a toluene solution into the fiber. Removal of the toluene occurs in a few days from evaporation, leaving behind quantum dots nanocrystals on the MOF inclusion walls.

The fiber has an acrylate coating on the outside of the quartz cladding. This coating protects the fiber and prevents breakage during normal handling. Removing this coating had no significant effect on the efficiency or optics of the quantum dots MOF. Other scintillator containing MOFs showed a variation in efficiency when the coating was removed [7, 8]. The ability to introduce quantum dots into microstructured fiber without damaging or removing the fiber coating while retaining efficiency is a useful feature.

In future work, improvements to the measured efficiency could be made by having a photon detector with a quantum efficiency greater than 65%. The measured efficiency would be increased in proportion to the corresponding detector increased efficiency. The fiber length was selected based on capillary flow and the collimator dimensions. However, a shorter fiber or a fiber containing only quantum dots may improve the x-ray detection efficiency.
FIGURE 6. Characterization for 4W 40kV silver anode x-ray tube showing the number of x-ray photon counts vs energy distribution curves at 5 kV increment tube voltages. The plot is collected using a 100 micron collimator with the CdTe detector x-ray spectrometer over a 300 second time interval.

FIGURE 7. Shown is a plot of x-ray efficiency for quantum dots MOF at tube voltages from 10 to 40 kV. The fiber efficiency is over 5% at all tube voltages and non-linear.
REFERENCES