Imaging performance of a CaWO₄/CMOS sensor

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ABSTRACT. The aim of this study was to investigate the modulation transfer function (MTF) and the effective gain transfer function (eGTF) of a non-destructive testing (NDT)/industrial inspection complementary metal oxide semiconductor (CMOS) sensor in conjunction with a thin calcium tungstate (CaWO₄) screen. Thin screen samples, with dimensions of 2.7x3.6 cm² and thickness of 118.9 μm, estimated from scanning electron microscopy-SEM images, were extracted from an Agfa Curix universal screen and coupled to the active area of an active pixel (APS) CMOS sensor. MTF was assessed using the slanted-edge method, following the IEC 62220-1-1:2015 method. MTF values were found high across the examined spatial frequency range. eGTF was found maximum when CaWO₄ was combined with charge-coupled devices (CCD) of broadband anti-reflection (AR) coating (17.52 at 0 cycles/mm). The combination of the thin CaWO₄ screen with the CMOS sensor provided very promising image resolution and adequate efficiency properties, thus could be also considered for use in CMOS based X-ray imaging devices, for various applications.

KEYWORDS. CaWO₄; Phosphors; Medical imaging; APS; CMOS sensors; MTF.

INTRODUCTION

Phosphor materials made of calcium tungstate (CaWO₄) [1,2] and zinc sulfide (ZnS) were used as radiation detectors, in the form of thin screens, for almost a century [3,4]. CaWO₄ is a low cost, very stable material, but with a decay time not ideal for applications that require high counting rate measurements (Table 1) [5]. CaWO₄ emits light in
the blue region of the spectrum which provides excellent compatibility with photocathodes, incorporated in various types of photomultipliers (PMTs), charge-coupled devices (CCD), as well as, with non-passivated amorphous hydrogenated silicon photodiode (a-Si:H), employed in thin film transistors of active matrix flat panel detectors [5]. When rare earth phosphors came into the spotlight, CaWO4 was dis-continued for medical imaging applications. Thereafter, terbium-doped gadolinium oxysulfide (Gd2O2S:Tb) rare earth phosphor, and later cesium iodide (CsI) were established for digital imaging applications (medical, industrial radiography, etc.) including CCDs and complementary metal oxide semiconductors (CMOS) [3,4,6-11]. In industrial radiography, non-destructive testing (NDT) is used; it consists of a variety of non-invasive inspection techniques that is used to evaluate material properties, components, or entire process units. Radiographic testing is one of the most frequently used NDT techniques that involve the use of X-rays and digital detector systems, such as amorphous silicon, CCDs and CMOS sensors [12-14]. However, beyond the dominance of terbium-doped gadolinium oxysulfide, the interest for CaWO4 has been renewed in applications such as, particle astrophysics in the quest for dark matter in the universe [15-17], for WIMP-nucleon elastic scattering interactions [18,19], as well as, for customs and border control [15,20]. The resolution properties of this material [21,22], along with the adequate luminescence emission efficiency, at specific X-ray energies [5] could be also considered for dual energy applications [23-28].

All the aforementioned applications require efficient detectors, of high resolution, with good spectral matching between the phosphor’s emission light and the sensitivity of the sensor, thus the aim of this study is to investigate further the properties of a thin layered calcium tungstate screen, coupled to a state-of-the-art NDT active pixel sensor (APS) CMOS sensor, in order to enhance the imaging capabilities of the integrated detector. Measurements were conducted, following standardized methodologies for medical imaging configurations (sensors and scintillator material combinations) [11]. Standardized protocols were used for both resolution and efficiency measurements [5, 29-31]. The latest IEC 62220-1-1:2015 protocol from the International Electrotechnical Commission (IEC) 62220 series was used [32,33].

**MATERIALS AND METHODS**

**Phosphor screens**

Samples of CaWO4 were extracted from an Agfa Curix universal screen. For the resolution measurements samples with dimensions 2.7x3.6 cm² were used. The phosphor is used in the intensifying screens employed in X-ray imaging [31,34,35]. The internal properties of the samples were examined via scanning electron microscopy (SEM) [36].

| Property                        | Value                        |
|---------------------------------|------------------------------|
| X-ray absorption (@50 keV)      | 35%                          |
| Light conversion efficiency     | 4-5%                         |
| Melting point                   | 1570-1670 °C                 |
| Molar mass                      | 287.9156 g/mol               |
| Atomic number                   | 74                           |
| Density                         | 6.06-6.1 g/cm³               |
| Afterglow                        | From 5x10⁻⁶ sec up to a few sec |
| Refractive index                 | 1.94                         |
| K-edge                           | 69.5 keV                     |
| Decay time                      | 6-8x10³ ns                   |

Table 1: CaWO4 properties [3-5,15,17,37,38].

**Scanning electron microscopy (SEM)**

Parameters such as particle size and thickness of the CaWO4 compound were verified via SEM micrographs using the Jeol JSM 5310 scanning electron microscope and the INCA software. Within this system, gold can be used to image a site of interest of the sample. For the elementary particle analysis, a carbon thread evaporation process was used. Carbon was flash evaporated under vacuum conditions to produce a film suited for the CaWO4 SEM specimen in a BAL-TEC CED 030 carbon evaporator (~10² mbar) [36].
CMOS sensor

The CaWO₄ scintillating screen was manually coupled to an optical readout device including a CMOS Remote RadEye HR photodiode pixel array [39]. The CMOS photodiode array consists of 1200x1600 pixels with 22.5 μm pixel spacing. This sensor was initially manufactured for non-destructive testing (NDT)/industrial inspection applications especially for tight or difficult to reach spaces. However, due to its unique resolution properties can be also used in medical imaging applications. Thus, it would be of interest to integrate a scintillating material that came to the spotlight once again, with this state-of-the-art sensor in order to further exploit their imaging characteristics. The CaWO₄ screen was directly overlaid onto the active area of the CMOS and irradiated with a BMI General Medical Merate tube having rotating Tungsten anode and inherent filtration equivalent to 2 mm Al at 70 kV (RQA-5) X-ray beam quality and source to detector distance of 156 cm [21].

Modulation Transfer Function (MTF)

The modulation transfer function (MTF) was measured by irradiating a PTW Freiburg tungsten edge test device, following the procedures described in the IEC standard [11,32,33]. The updated IEC 62220-1-1:2015 [11,33,40] standard describes certain modifications, such as the method for the determination of the modulation transfer function (MTF) in which the final MTF can be now obtained only through averaging of the oversampled edge spread function (ESF) [33,41-43]. The average of all oversampled ESFs was then fitted with a modified Fermi-Dirac distribution function as follows [11,21]:

$$F_{\text{Fermi}}(x) = \frac{c}{e^{(x-a)/b} + 1} + d$$ (1)

The values of fitting parameters are a=7200, b=15, c=65500 and d=0. The fitted ESF was differentiated to obtain the line spread function (LSF) and Fourier transformed to finally obtain the MTF [21,44].

Luminescence efficiency measurements

The efficiency (output signal) of a scintillator to emit light, upon X-ray irradiation is experimentally determined by measuring the emitted light energy flux $\Psi_\lambda$ and the exposure rate ($X$) using a calibrated dosimeter. In this study, the Piranha P100B RTI was used. The light flux measurements were performed using a light integration sphere (Oriel 70451), coupled to a photomultiplier (PMT) (EMI 9798B) and connected to a Cary 401 vibrating reed electrometer [5,9]. The circular CaWO₄ sample was also exposed to X-rays on the BMI General Medical Merate tube, with energies ranging from 50 to 125 kVp. An additional 20 mm Al filtration was introduced in the beam to simulate beam quality alternation by a human body [45,46].

X-ray luminescence efficiency (XLE)

The X-ray luminescence efficiency (XLE) is a unitless measure of the fraction of incident energy converted into emitted light energy, i.e. the ratio of the emitted light energy flux over the incident X-ray energy flux ($\eta_{\Psi_\lambda} = \Psi_\lambda/\Psi_0$). XLE was determined [9] by converting the measured X-ray exposure data into X-ray energy flux ($\Psi_0$) [9], as follows: $\Psi_0 = X\hat{\Psi}$ where $\hat{\Psi}$ is defined as the X-ray energy flux per exposure rate, estimated according to Eq.(2) [5,36]:

$$\hat{\Psi} = \int \frac{\Psi_0(E) dE}{\Psi_0(E)} \frac{dE}{X/\Psi_0(E)}$$ (2)

where

$$X/\Psi_0(E) = (\mu_{en}(E)/\rho)_\text{air} \cdot (W_A/\rho)^{-1}$$ (3)

is the factor converting energy flux into exposure rate, ($\mu_{en}/\rho)_\text{air}$ the X-ray mass energy absorption coefficient of air, at energy $E$, and ($W_A/\rho$) is the average energy per unit of charge required to produce an electron-ion pair in air. ($W_A/\rho$) and ($\mu_{en}/\rho)_\text{air}$ were obtained from tabulated data [47].

Detector quantum optical gain (DQG)

Detector quantum optical gain (DQG) is the ratio of the light photon flux ($\Phi_\lambda$) over the X-ray photon flux ($\Phi_\lambda$) and expresses the emission efficiency in terms of quantum gain (number of emitted light photons per incident X-ray). Using this quantity, the emitted light photon fluence can be expressed in terms of experimentally determined quantities (absolute efficiency, exposure, mean light wavelength), by using Eq.(4) [5]:

$$\Phi_\lambda = \frac{\Psi_0}{\eta_{\Psi_\lambda}}$$ (4)
Φₐ = Ψₐ / k₀X⁻¹ \hspace{1cm} (4)

Φₓ was determined by using Eqs.(2,3), replacing Ψ₀ by Φ₀ and dividing Eq.(3) by the X-ray energy [9]. Eqs(2,4) may be expressed in the spatial frequency domain. Within this framework the detector quantum gain may be expressed by a gain transfer function (GTF), defined as follows [36,45]:

GTF(E₀,ν,w) = Φₐ(E₀,ν,w) / Φ₀ \hspace{1cm} (5)

where ν denotes spatial frequency, w coating thickness and Φₓ(E₀,ν,w) is the spatial frequency-dependent emitted light photon fluence. Gain transfer function, can be expressed through the MTF [36,45]:

GTF(ν,w) = MTF(ν,w) · DQG \hspace{1cm} (6)

where DQG is the detector quantum optical gain. In medical imaging, where fluorescent screens are used in combination with optical detectors (films in the past, photocathodes, photodiodes), the spectral matching between the emitted phosphor light and the optical detector sensitivity must be considered. This is because the degree of spectral matching affects the amount of light utilized to form the final image. Thus, Eq.(6) is reduced by a factor αₛ, expressing the fraction of emitted light that can be detected by the optical detector, which exhibits a specific spectral distribution of sensitivity. αₛ can be calculated by Eq.(7) [5,45]:

αₛ = \int \frac{S_p(λ)SD(λ)dλ}{S_p(λ)dλ} \hspace{1cm} (7)

where S_p(λ) is the spectrum of the light emitted by the phosphor and SD(λ) is the spectral sensitivity of the optical detector coupled to the phosphor [5]. By considering αₛ, we may define the effective gain transfer function as follows [36,45]:

eGTF(ν,w) = DQG · MTF(ν,w) · αₛ \hspace{1cm} (8)

RESULTS AND DISCUSSION

Fig.1 shows the grain-size deposition per thickness of CaWO₄, estimated from scanning electron microscope images. Qualitatively the mean particle size of CaWO₄ phosphor (6.02 μm) was estimated from the SEM images using the ImageJ analysis software, as shown from the grain-size distribution [48,49]. The calculated screen thickness was equal to 118.9 μm estimated by profile measurements on the area depicted as inset in Fig.1, across the material coating [5,22]. Furthermore, in Fig.1 the energy dispersive X-ray (EDX) analysis of the material is demonstrated. It was found that CaWO₄ was dominantly present in the sample along with carbon (C) due to the carbon thread evaporation process. Normalized stoichiometric results, obtained by the SEM on the region of interest (ROI) of Fig.1, showed the following % weights of the elements in the mixture: Calcium (Ca) 5.77%, oxygen (O) 26.54%, tungsten (W) 29.94 and carbon due to the carbon thread evaporation process 37.75% [22]. The X-ray characteristic curves (output signal versus incident exposure) of CaWO₄ and (for comparison purposes) of a flexible fluorescent Gd₂O₂S:Tb sample (gold standard for imaging applications) of similar coating thickness (30.8 mg/cm² for Gd₂O₂S:Tb versus 36.26 mg/cm² for CaWO₄) are plotted in Fig.2. These coating thicknesses were calculated assuming densities of 7.3 g/cm³ for Gd₂O₂S:Tb and 6.1 g/cm³ for CaWO₄ with packing densities of 50% for both materials. Results for CaWO₄ and Gd₂O₂S:Tb show linear dependence between the output signal and exposure rate in the 4-299 mR/s range. The linear no-threshold fit gave correlation coefficient values R² of 0.9997 for CaWO₄ and 0.9953 for Gd₂O₂S:Tb, which are very close to unity and indicate that the screens have linear response in this energy range. Gd₂O₂S:Tb was found with clearly higher output signal values than those of CaWO₄ due to its higher absolute efficiency values [5].

Fig.3 shows all the oversampled ESFs (Fig.3a) used to create the average ESF and then the Fermi-fitted ESF (Fig.3b), as well as, the resulted LSF (Fig.3c), following the IEC 2015 protocol and the edge phantom. The edge test device consists of a 1 mm thick W edge plate (100×75 mm²) fixed on a 3 mm thick lead plate. Images of the edge, placed at a slight angle in order to avoid aliasing effects, were obtained. Irradiation was performed at 70kVp and 50 mAs for the tube current and
Figure 1: Grain-size distribution obtained from the scanning electron microscope image of the CaWO$_4$ scintillating material (a), obtained from a region of interest (b), along with the energy dispersive X-ray (EDX) analysis (c).

Figure 2: Output signal of the 36.26 mg/cm$^2$ CaWO$_4$ screen and a 30.8 mg/cm$^2$ Gd$_2$O$_2$S:Tb flexible phosphor in the radiographic range of exposures.

exposure time product. This energy is also within the energy range of 51-84 keV that can be utilized for testing steel thicknesses between 2.5 and 12.5 mm [13]. The edge spread function, of the small CMOS detector, was calculated by the extraction of a 2×2 cm$^2$ ROI, which covers approximately 41% of the active area of the small area CMOS sensor (2.7×3.6 cm$^2$), with the edge roughly at the center. In the IEC protocol a 5×10 cm$^2$ ROI is suggested [33]. A smaller ROI may not be
adequate to observe a possible low-frequency sharpness drop in scintillating screens [39]. The variation in the ESF curves that can be depicted from Figs.5(a,b), can be attributed to a statistical noise component mostly prominent at the edge surface area. Fig.3d shows results for the modulation transfer function of the CMOS sensor combined with the 118.9 μm CaWO₄ screen, under the RQA-5 (70kVp) beam quality. MTF values were found high across the examined spatial frequency range.

![Figure 3: (a) ESFs of a 118.9 μm CaWO₄/RadEyeHR CMOS combination, following the IEC 2015 method, (b) averaged and Fermi fitted ESF, (c) LSF, (d) Modulation transfer function following the IEC 2015 protocol, under the RQA-5 beam quality.](image)

Figs.(4,5) show the variation of GTF and eGTF with spatial frequency for the CaWO₄ phosphor screen measured at 70 kVp. The difference between this curve and the corresponding MTF curve is due to the influence of X-ray absorption and optical emission on GTF, which are more apparent at lower frequencies. As frequency increases, the influence of MTF on GTF is more significant than the corresponding influence of detector quantum gain (DQG: 18.17 at 70 kVp [5]) causing a further decrease in the GTF (Fig.4).

Figs.5(a-d) shows indicative effective GTF results of the CaWO₄ screen with various optical detectors. Results are shown up to 5 cycles/mm, since the measured MTF incorporates the MTF of the CMOS semiconductor which however has been reported to be higher than 0.984, thus the calculation error is minimum [8,50-52]. The best optical detector-screen combination was obtained for a charge-coupled device having broadband anti-reflection (AR) coating (Fig.5c) with an eGTF value of 17.52 at zero spatial frequency. This value reduces GTF only by 3.54% (spectral matching factor: 0.964 [5]). The eGTF values of the CCD, is followed by the Hamamatsu MPPC silicon photomultipliers S10985 (Fig.5b) (eGTF: 17.39 at 0 cycles/mm, matching factor: 0.957), the GaAs photocathode (eGTF: 17.36 at 0 cycles/mm, matching factor: 0.955) (Fig.5a) and the non-passivated amorphous hydrogenated silicon photodiode (a-Si:H) (eGTF: 17.39 at 0 cycles/mm, matching factor: 0.948), employed in thin film transistors in active matrix flat panel detectors.

CaWO₄ also shows very good eGTF values with Sensl’s silicon PMTs, with eGTF value 14.21 at 0 cycles/mm, for the MicroFM-10035 (matching factor: 0.782), with the MicroFB-30035-SMT (eGTF: 15.25 at 0 cycles/mm, matching factor: 0.839) and with the MicroFC-30035 (eGTF: 15.94 at 0 cycles/mm, matching factor: 0.877) (Fig.5b). Furthermore, it showed very good eGTF with Hamamatsu flat panel position sensitive photomultipliers, such as the H8500C-03 (eGTF: 14.60 at 0 cycles/mm, matching factor: 0.80) (Fig.5c). It is of importance to note that eGTF showed good values when CaWO₄ is combined with complementary metal-oxide semiconductors, used in digital radiography and mammography systems, showing maximum when coupled with a hybrid blue CMOS (eGTF: 17.39 at 0 cycles/mm, matching factor: 0.854) [53].
Figure 4: Variation of the GTF with spatial frequency for the thin CaWO₄ phosphor screen.

Figure 5: Variation of the eGTF with spatial frequency for the CaWO₄ phosphor screen combined with the various optical detectors.
CONCLUSIONS

Applications such as, non-destructive testing, medical imaging etc., require efficient detectors, of high resolution. In this study the resolution properties of a non-destructive testing/industrial inspection CMOS sensor, in conjunction with a scintillating material that came to the spotlight once again (CaWO₄) was examined in order to further exploit and enhance the imaging capabilities of an integrated detector, incorporating these two modules. Experiments were carried under X-ray radiography imaging conditions, following the IEC 62220-1-1:2015 protocol. Furthermore, the detector quantum gain of the screen and the spectral compatibility was also examined for various optical sensor combinations. MTF values of the CaWO₄ screen/CMOS combination were found high across the spatial frequency range. As a conclusion, the resolution properties of the thin 118.9 μm CaWO₄ screen/CMOS combination are promising for general radiography medical imaging applications.

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