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Advanced X-ray Detectors for Industrial and Environmental Applications
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page | ii
\[ \frac{d}{du} (u^2 + 2as) \]
A wise man once said nothing.

It was a night in winter.
It was as dark as it gets in the north, like the eyes of a virgin.
It was cold, cold as the fading shadows which were once loved.
With a glass full of whiskey, a heart full of joy and air full of swimming memories.....
A wise man once sat down wondering about life, he said nothing.

He could hear the falling snow,
stories of a thousand souls he once met, but never again.

All the faces,
all the warm glances,
all the moments of happiness in his life.
Touched him.
Like she did.
Oh! How she did it! He smiled.....

He looked at the flakes stuck to the window.
People who came and never left.
Some left, gold and gems all the way.
Some gave, hopes and inspirations.
Oh! How they did it! He said nothing, he just smiled.....
ABSTRACT

The new generation of X-ray free electron laser sources are capable of producing light beams with billion times higher peak brilliance than that of the best conventional X-ray sources. This advancement motivates the scientific community to push forward the detector technology to its limit, in order to design photon detectors which can cope with the extreme flux generated by the free electron laser sources. Sophisticated experiments like deciphering the atomic details of viruses, filming chemical reactions or investigating the extreme states of matter require detectors with high frame rate, good spatial resolution, high dynamic range and large active sensor area. The PERCIVAL monolithic active pixel sensor is being developed by an international group of scientists in collaboration to meet the aforementioned detector requirements within the energy range of 250 eV to 1 keV, with a quantum efficiency above 90%.

In this doctoral research work, Monte Carlo algorithm based Geant4 and finite element method based Synopsys Sentaurus TCAD toolkits have been used to simulate, respectively, the X-ray energy deposition and the charge sharing in PERCIVAL. Energy deposition per pixel and charge sharing between adjacent pixels at different energies have been investigated and presented.

Novel methods for industrial and environmental applications of some commercially available X-ray detectors have been demonstrated. Quality inspection of paperboards by resolving the layer thicknesses and by investigating orientation of the cellulose fibres have been performed using spectroscopic and phase-contrast X-ray imaging. It was found that, using phase-contrast imaging it is possible to set burn-out like quality index on paperboards non-destructively. X-ray fluoroscopic measurements have been conducted in order to detect Cr in water. This method can be used to detect Cr and other toxic elements in leachate in landfills and other waste dumping sites.
SAMMANFATTNING

Acceleratorbaserade röntgenkällor utvecklas ständigt, dessa kan producera röntgenstrålning med miljarder gånger så hög effekttäthet som de starkaste konventionella röntgenkällorna. Därför finns en vetenskaplig utmaning att utveckla röntgendetektorer som inte förstörs i de extrema flöden som genereras av dessa röntgenkällor. De visioner som finns för de nya källorna är t.ex.; att avbilda detaljer av virus ner på atomnivå, att film kemiska reaktioner eller att undersöka extrema tillstånd hos material. Dessa typer av experiment kräver röntgendetektorer med hög bildhastighet, hög spatial upplösning och stort intensitetsomfång och stor aktiv sensoryt. Detektorsystemet PERCIVAL som bygger på aktiva pixlar med energiupplösning utvecklas inom ett internationellt vetenskapligt samarbetsprojekt. Målet är att uppfylla detektorspecificationerna för de nämnda experimenten inom energiområdet 250 eV till 1 keV, med en kvantverkningsgrad över 90 %.

I föreliggande vetenskapliga avhandlingsarbete har simuleringar av energideponering i PERCIVAL-detektorn genomörts baserat på Monte Carlo-algoritmer och simuleringar av laddningsdelning mellan pixlar har simulerats med hjälp av finita elementmetoden. Därmed har energideponeringen per pixel och laddningsdelningen mellan närliggande pixlar vid olika energier kunnat utredas och presenteras.

I avhandlingen demonstreras nya lovande metoder för industriella applikationer och miljöövervakning, där kommersiellt tillgängliga röntgendetektorer kan användas. Kvalitetsövervakning av kartong tillverkan genom att mäta bestrykningstjocklek och fiberorientering kan realiseras med energiupplösta röntgenbilder eller faskontrastbilder i röntgenområdet. Det kan konstateras att med icke-förstörande provning, genom faskontrastbilder, kan kvalitetsindexvärlden erhållas på samma sätt som kvalitetsindex kan erhållas från ”burn-out”-mätningar. Spektroskopiska mätningar av röntgenflourescens har genomförts för att detektera krom (Cr) i vatten. Metodik för att detektera krom och andra giftiga metaller i lakvatten från deponi och annan lagring för giftigt avfall har utarbetats.
ACRONYMS

FEM  Finite Element Method
HEP  High Energy Physics
XFEL  X-ray Free-Electron Laser
MAPS  Monolithic Active-Pixel Sensor
Geant4  GEometry ANd Tracking, Version 4
TCAD  Technology Computer Aided Design
keV  Kilo Electronvolt
MeV  Mega Electronvolt
CERN  Conseil Européen pour la Recherche Nucléaire
CMOS  Complementary Metal Oxide Semiconductor
CONTENTS

Abstract vii

Sammanfattning ix

Acronyms xi

Contents xiii

List of Figures xv

List of Tables xvii

List of Publications xix

1 Introduction 1
  1.1 Motivation ........................................... 2
  1.2 Social, Ethical and Environmental Aspects .......... 4

2 X-ray Imaging 5
  2.1 X-rays ............................................. 5
  2.2 Interaction of X-rays with Matters ................. 5
  2.3 Imaging with X-ray .................................. 6
  2.4 X-ray Detectors .................................... 14

3 Geant4 Simulation 23
  3.1 Simulation of PERCIVAL ............................. 23

4 TCAD Simulation 31
  4.1 Sentaurus TCAD .................................... 31
  4.2 Simulation of PERCIVAL ............................ 32

5 Detector Applications 37
  5.1 Resolving Paperboard Layer Thicknesses .......... 37
  5.2 Uniformity in the Cellulose Layer in Paperboards 40
  5.3 Replacing Burn-out Methods ........................ 43
  5.4 Detecting Cr in Water ................................ 45

page | xiii
Contents

6 Conclusions 47

Bibliography 49

Summary of the Publications 57

Article I 61

Article II 75

Article III 83

Article IV 97

Article V 113

Article VI 127

Article VII 133

Article VIII 139

Article IX 149

Article X 159
## LIST OF FIGURES

2.1 Wavelengths of soft and hard X-rays. .......................... 5
2.2 X-ray image of human fingers presented in W. C. Röntgen’s article in 1896. One finger with a ring. .......................... 7
2.3 Optical image of lichen. .......................................... 8
2.4 Three different types of X-ray images of lichen. .................. 9
2.5 X-ray images of a bug obtained using PCXI technique. ............. 9
2.6 Grating interferometer. ........................................... 10
2.7 Self-imaging through the Talbot effect. ........................... 10
2.8 Intensities at a single pixel at different g2 positions. ............... 12
2.9 Two sets of intensities at a single pixel. .......................... 13
2.10 One pixel cell in a hybrid detector[54]. ............................ 15
2.11 PERCIVAL illumination modes. ................................. 17
2.12 PERCIVAL schematic. .......................................... 17
2.13 Noise in all pixels within one sub-matrix in PERCIVAL prototype sensor. ............................ 18
2.14 Response under uniform illumination. ............................ 19
2.15 Airy disk patterns generated by a 5 µm pinhole. .................. 20
2.16 Charge Collection Efficiency. ................................... 21

3.1 Geant4 framework. ............................................. 23
3.2 A small segment of the simulated detector, together with the thicknesses of the layers. ............................... 24
3.3 Simulated set-up. ............................................... 25
3.4 All interactions inside the Si layer. ................................ 26
3.5 Spectra of energy deposition per pixel at different energies, 10M photons from the source. ............................. 27
3.6 Collected charges. .............................................. 28

4.1 Simulated PERCIVAL structure. ................................ 33
4.2 One pixel with charge generation point in its middle. ............. 33
4.3 Built-in potential. ............................................... 34
4.4 Dark current. .................................................. 34
4.5 Charge transport inside the detector over times (5900 eV). ....... 35
4.6 Charge sharing between pixels. .................................. 36
List of Figures

5.1 Optical image of paperboard (P), only coating (Q) and only cellulose layer (R) ................................. 39
5.2 Thickness profiles of the separated (a) coating and (b) cellulose layer ................................. 40
5.3 The logarithm of the dark-field signal as a function of the sample orientation ................................. 41
5.4 The isotropic scattering coefficients of the samples in relation to the burn-out indices ................................. 42
5.5 Photo of burnt paperboard ............................................. 43
5.6 (a) Phase-retrieved X-ray image of the sample. Red contour line is selected based on color difference. (b) Photo of the same paperboard with contour lines from the X-ray image ................................. 44
5.7 Measured spectra from water with different Cr concentrations (background subtracted) ................................. 45
5.8 Cr $K\alpha$ photon counts from water with different Cr concentrations ................................. 46
## LIST OF TABLES

2.1 PERCIVAL design goals. ........................................... 16

5.1 Isotropic (A) and anisotropic (B) scattering coefficients of the samples. ........................................... 42
LIST OF PUBLICATIONS

This thesis is based on the following publications:

Article I  
Readout cross-talk for alpha-particle measurements in a pixelated sensor system  
B. Norlin, S. Reza, D. Krapohl, E. Fröjd, and G. Thungström  
*Journal of Instrumentation, JINST 10 C05025, May 2015.*

Article II  
Measurement of the sensitive profile in a solid state silicon detector, irradiated by X-rays  
G. Thungström, L. Herrnsdorf, B. Norlin, S. Reza, D. Krapohl, S. Mattsson and M. Gunnarsson  
*Journal of Instrumentation, JINST 8 C04004, April 2013.*

Article III  
Spectral resolution in pixel detectors with single photon processing  
C. Fröjd, D. Krapohl, S. Reza, E. Fröjd, G. Thungström, B. Norlin  
*Proc. of SPIE Vol. 8852 88520O-1.*

Article IV  
Grating-based phase-contrast X-ray imaging technique  
Salim Reza  
*Radiation Detectors for Medical Imaging, Chapter 10, CRC Press, 2016.*

Article V  
Characterisation of a PERCIVAL monolithic active pixel prototype using synchrotron radiation
List of Publications

*Journal of Instrumentation, JINST 11 C02090, February 2016.*

Article VI

**Experimental characterization of the PERCIVAL soft X-ray detector**
*IEEE Nuclear Science Symposium Conference Record 2015.*

Article VII

**Non-destructive method to resolve the core and the coating on paperboard by spectroscopic x-ray imaging**
**Salim Reza**, Börje Norlin, Jan Thim and Christer Fröjdh

Article VIII

**Investigation on the directional dark-field signals from paperboards using a grating interferometer**
*Journal of Instrumentation, JINST 9 C04032, April 2014.*

Article IX

**Phase-contrast X-ray Imaging for Non-destructive Quality Inspections of Paperboards**
**Salim Reza**, Tunhe Zhou, Anna Burvall, Johan Lindgren, Christer Fröjdh, Hans M. Hertz and Börje Norlin
*Manuscript.*
List of Publications

Article X  Detecting Cr Contamination In Water Using X-Ray Fluorescence
Salim Reza, Haosi Chang, Börje Norlin, Christer Fröjdh and Göran Thungström
IEEE Nuclear Science Symposium Conference Record 2015.

Related publications, not included in this thesis:

Smart dosimetry by pattern recognition using a single photon counting detector system in time over threshold mode
S. Reza, W. S. Wong, E. Fröjdh, B. Norlin, C. Fröjdh, G. Thungström and J. Thim
Journal of Instrumentation, JINST 7 C01027, January 2012.

Suitable post processing algorithms for X-ray imaging using oversampled displaced multiple images
J. Thim, S. Reza, K. Nawaz, B. Norlin, M. O’Nils and B. Oelmann
Journal of Instrumentation, JINST 6 C02001, February 2011.

X-ray imaging of high velocity moving objects by scanning summation using a single photon processing system
Jan Thim, Salim Reza, Mattias O’Nils and Börje Norlin
Journal of Instrumentation, JINST 10 C04023, April 2015.

Detector Developments at DESY
List of Publications

Report on Control/DAQ Software Design and Current State of Implementation for the Percival Detector
INTRODUCTION

*Men of science in this city are awaiting with the utmost impatience the arrival of English technical journals which will give them the full particulars of Professor Röntgen’s discovery of a method of photographing opaque bodies.*


In November 1895, Rector Wilhelm Conrad Röntgen was working with a Crookes tube to study electrical discharge through gases in his laboratory in the University of Würzburg. He noticed a fluorescing barium platinocyanide screen, a little far from the Crookes tube, when he generated cathode rays in it[2]. He realised, the reason must be an invisible ray coming out of the tube and irradiating the screen. Later, he also noticed that this mysterious ray can penetrate wood, paper and flesh. He immediately understood the significance of his discovery and devoted all his time to characterize the new ray, later to be known as X-ray.

After the discovery of X-ray was reported to the Würzburg Physico-Medical Society and later to the Berlin Physical Society, the news started to spread around the world. The scientific communities all over became curious to know about the ray. Some scientists started working on characterizing X-ray and developing X-ray detectors, while others were exploring fascinating applications of it.

In the beginning, photographic plates were being used to detect X-ray due to their availability. Then chemical colouration became popular because film development could be avoided using these techniques. Several scientists including Ernest Rutherford pioneered the usage of gas chambers for X-ray detection. After World War II, solid-state detectors were developed together with necessary electronics and have been used widely to detect X-rays. Today, a solid-state detector coupled to a computer is the standard X-ray detection instrument.

Since the revolutionary discovery of X-rays in 1895 by W. C. Röntgen[3], X-rays have been used in medical imaging, material inspections and quality controls, security applications and in research within numerous academic
disciplines in order to investigate the insides of objects. X-ray imaging has become a standard tool for in-vivo observation because of its non-destructive properties.

In this thesis, the technology of modern advanced X-ray detector systems is discussed together with their applications. Energy deposition, electrical properties and charge transport inside the sensor material of such a detector are investigated using Monte-Carlo and FEM based simulation tools. Applications of X-ray detectors in industrial quality control and environmental measurements have been demonstrated.

1.1 Motivation

1.1.1 Detector Technology

The radiation detector is a key element in applications such as medical imaging, industrial inspection and HEP research since it determines the signal or image quality. The rapid development in microelectronics has opened up for new type of detectors where each photon or particle can be treated individually. New detector concepts with improved spectral resolution, spatial resolution and speed can then be developed.

During the late 1990s, direct conversion X-ray detectors dedicated for synchrotron applications started to be developed[4]. Now, highly brilliant synchrotron sources, such as, the 3rd generation storage rings and Free-Electron Lasers[5] are being built. In Hamburg, the XFEL[6] is currently under construction, which will generate 27000 ultra-short X-ray flashes per second, with billion times higher brilliancy than that of the best X-ray sources currently available.

In order to cope with the flux and the planned sophisticated experiments at the XFEL, advanced large detectors with high frame rates, good spatial resolution, high dynamic range and single photon discrimination ability have to be designed[7]. The PERCIVAL detector with such specifications is currently being developed by an international collaboration of scientists from RAL/STFC, Elettra Sincotrone Trieste, Diamond Light Source and Pohang Accelerator Laboratory[8], coordinated by DESY. PERCIVAL is a MAPS-based back thinned detector to access the energy range of 250 $eV$ to 1 $keV$ with an efficiency of minimum 90%.

In MAPS, the substrate of the readout electronics acts as the sensitive element[9]. This architecture offers several advantages, such as, small pixel sizes, low noise floor and low power consumption[10]. The active part or the sensitive element in PERCIVAL is the epitaxial layer, which is also
The X-ray interactions and energy deposition in the PERCIVAL epitaxial layer have been simulated using Monte-Carlo based Geant4 simulation toolkit[12]. Geant4 has been chosen because the electromagnetic models for the low target energies for PERCIVAL are available in this toolkit[13].

The charge collection efficiency and the charge sharing between pixels in PERCIVAL have been investigated using the FEM based Sentaurus TCAD package[14]. This widely used package solves Poisson’s equation and the continuity equations for both electrons and holes. It also allows both steady-state and transient simulations.

1.1.2 Detector Applications

Modern X-ray detectors with both high spectral and spatial resolution are used in many applications. The most common imaging modality is transmission imaging where the absorption of the X-rays in the object is measured. The spectral absorption properties can be used to distinguish between different materials. X-ray diffraction and X-ray fluorescence are other methods to study the composition of objects. Phase-Contrast X-ray Imaging (PCXI)[15] is a method to reveal tiny structures within soft objects or objects with homogeneous density distributions[16].

In conventional attenuation-based X-ray imaging, the image of an object is constructed based on the absorption of the X-ray beam while passing through it. In PCXI, it’s the phase shift of the wave that generates the contrast in the image[17]. For this reason, PCXI is suitable for imaging soft biological objects which have low or no X-ray absorption. The enhanced edge effect is also a significant advantage in cases for which the observation of the inner structures of objects is of interest.

Paperboard is made of biological materials, so absorption-based X-ray imaging is not optimal for imaging. A typical sheet of paperboard contains a core of cellulose fibres \([C_6H_{10}O_5]\), coated on one or both sides with layers of calcium carbonate \([CaCO_3]\) or Kaolin \([Al_2Si_2O_5(OH)_4]\)[18]. The end-use performance of paperboard, such as the printing quality and the stiffness, depends on the uniformity and the thickness of these layers. In this thesis, results from paperboard quality inspection using spectroscopic imaging and PCXI have been presented[19, 20].

With the rapid growth in population and the overwhelming demand of
Chapter 1. Introduction

industrial consumer products around the world, the amount of generated wastes is also increasing. The waste disposal sites may produce toxic and green house gases and also a substantial amount of leachate, which can affect the environment[21]. Leachate may contain toxic and harmful substances, such as Chromium (Cr), Arsenic, Lead, Mercury, Benzene, Chloroform and Methylene Chloride, and can contaminate surface water and aquifers. While consuming small amount of Cr(III) is considered as beneficial for health, Cr(VI) is highly toxic. It is a known carcinogen, which can cause lung cancer[22, 23].

Measurements using X-ray fluorescence have been performed to detect Cr in water[24]. The same method can be used in landfills or at waste dumping stations in order to monitor Cr concentration in leachate. X-ray fluorescence measurement has been tested as an alternative to most of the currently used methods of detecting Cr in water, which involve chemical experiment and analysis[25, 26]. These experiments are accurate, but they are slow processes and require a significant amount of preparation time.

1.2 Social, Ethical and Environmental Aspects

During the doctoral studies; social, ethical and environmental values for education and research have been considered seriously and respected accordingly. The PERCIVAL detector is intended to be used in experiments related to fundamental research, which will create new knowledge and allow the scientific community to verify existing hypotheses. PCXI has the potential to revolutionize medical imaging by allowing the doctors to diagnose patients with diseases at their early stages. The paperboard industry could benefit from the quality inspection methods demonstrated in this thesis. It will allow them to improve the quality of their products and get an edge in a very competitive market. The possibilities of economic and operational growth of the paper industry favour the regional developments. The presented method of Cr detection can also be used in detecting other toxic elements in the water in waste dumping sites. This way municipalities will be able to supply cleaner, safer and healthier water to the citizens; which may improve the public health. Radiation safety regulations were followed and all recommended means of radiation safety were practised during all measurements in this thesis. No animals were harmed.
Our imagination is stretched to the utmost, not, as in fiction, to imagine things which are not really there, but just to comprehend those things which are there.
– Richard Phillips Feynman.

2.1 X-rays

X-rays are electromagnetic waves with 10 nanometre as the longest wavelength[27]. X-rays are divided into two groups; soft X-ray and hard X-ray (figure 2.1). The soft X-rays have an energy lower than ~12.4 keV and a wavelength longer than 1 Å. The hard X-rays have wavelengths which are shorter than 1 Å and with a photon energy larger than ~12.4 keV[15].

Figure 2.1: Wavelengths of soft and hard X-rays.

X-ray photons are created by two atomic processes after electrons hit a metal target[28], namely, bremsstrahlung and X-ray fluorescence. In the bremsstrahlung process, the incident electrons scatter due to the electric field near the nuclei of the atoms of the target metal and lose energy as photons. In the latter case, the incident electrons knock out electrons from the inner shells of the metal atoms, then, electrons from the upper shells can take the empty spaces by emitting photons or Auger electrons.

2.2 Interaction of X-rays with Matters

X-ray photons interact with matter in three main ways; photoelectric absorption, Compton scattering and pair production[29].
Chapter 2. X-ray Imaging

In the process of photoelectric absorption, a photon interacts with an atom in the material. The photon is completely absorbed by the atom and an electron is knocked out, generally from the K-shell of the atom. The removed electron has an energy which can be calculated by subtracting the binding energy of the electron from the energy of the photon. The vacancy left behind by the electron may be filled up by capturing a free electron from the interacting material or by obtaining an electron from the other shells of the atom. The excess energy is emitted as characteristic X-ray photons or as Auger electrons.

The Compton scattering or the Compton effect, was first observed and explained by A. H. Compton in 1923[30]. When a photon with a particular wavelength collides with an electron in an atom, it scatters away from its original direction with a longer wavelength. This is an inelastic process because the scattered photon loses some energy. The change in the wavelength can be obtained by

\[ \lambda_s - \lambda_p = \frac{2h}{mc} \sin^2 \frac{1}{2} \theta \]  

where, \( \lambda_s \) and \( \lambda_p \) are the wavelengths of the scattered and the primary photons, respectively, \( h \) is the Planck constant, the mass of the electron is \( m \), \( c \) is the velocity of light and \( \theta \) is the angle between the directions of the scattered and the primary photons.

The pair production or the gamma conversion process occurs when the energy of an incident X-ray photon is more than twice the rest-mass energy of an electron, which is 0.511 MeV. In this process, the incident photon is annihilated and an electron-positron pair is produced. All the extra energy of the photon above 1.02 MeV is shared by the electron and the positron as their kinetic energy.

In addition to the interaction processes discussed above, X-ray photons may interact with all the electrons in an atom through coherent or Rayleigh scattering, leaving the atom unaffected.

2.3 Imaging with X-ray

X-rays or any other electromagnetic waves are affected by the complex refractive index of a material while passing through it[31]. The complex refractive index is a mathematical expression regarding the propagation of electromagnetic waves in a material. It can be written as

\[ n = 1 - \delta + i\beta \]  

(2.2)
2.3.1. Attenuation-based X-ray Imaging

where, \( n \) is the complex refractive index, \( \delta \) represents a phase shift and \( \beta \) represents absorption. The real part of this index, \( \delta \) can be expressed as

\[
\delta = \frac{2\pi \rho_a Z r_0}{k^2} \tag{2.3}
\]

where, \( \rho_a \) is the atomic number density, \( Z \) is the atomic number, \( r_0 \) is the classical electron radius and \( k \) is the magnitude of the wave vector. The imaginary part of the index, \( \beta \) can calculated as

\[
\beta = \frac{\rho_a \sigma_a}{2k} \tag{2.4}
\]

where, \( \sigma_a \) is the absorption cross-section.

When an X-ray photon passes through a material, it becomes refracted due to a decrease in the real part of the refractive index, \( \delta \). \( \beta \) attenuates the incident beam\[32\]. The real part of the refractive index, \( \delta \) and the imaginary part, \( \beta \) depend on the wavelength of an incoming photon in a different manner.

2.3.1 Attenuation-based X-ray Imaging

![X-ray image of human fingers presented in W. C. Röntgen’s article in 1896. One finger with a ring.]

This conventional X-ray imaging technique is based on the concept of X-ray transmission\[33\]. In this technique, X-rays are generated and emerge from a source, pass through an object to be imaged and are then detected. The differences in the X-ray attenuation in different areas within the object form the image. The difference in thickness and density within the volume
Chapter 2. X-ray Imaging

of the object causes a variation in the intensity of the passing X-ray beam. X-ray attenuation is explained by the Beer-Lambert law

\[ I = I_0 e^{-\mu t} \]  

(2.5)

where, \( I \) and \( I_0 \) are the intensities of the X-ray beam, after and before passing the object, respectively, \( t \) is the thickness of the material and \( \mu \) is the linear attenuation coefficient. \( \mu \) depends on the type of material and the energy of the passing X-ray beam. Figure 2.2 shows an image of human fingers, acquired using attenuation-based X-ray imaging. The bones inside the fingers are visible.

K-edge imaging is an advanced attenuation based imaging technique. In this method, two X-ray images are recorded with energies just above and below the K-edge of an element of interest in an object. Then, these two images can be subtracted to produce an image containing only the element.

2.3.2 Phase-Contrast X-ray Imaging (PCXI)

The distribution of the decrement in \( \delta \) (Eqn. 2.3) within an object produces a contrast in the phase-contrast images of that object[34]. For high energy hard X-rays, the cross section for elastic scattering is much larger than that for absorption[35]. Elastic scattering causes the phase shift. PCXI of an object at a certain wavelength can have some orders of magnitude higher sensitivity than attenuation based imaging at that wavelength[36]. One example of such objects can be biological tissue. Soft tissues with a 50 \( \mu m \) thickness can hardly attenuate a 17.5 keV X-ray, but the phase shift can be near to \( \pi \)[37].

Lichen (Usnea Filipendula, figure 2.3) is a fungus-like organism containing soft biological elements. For this reason, the phase-contrast and the dark-field image, which is also produced using PCXI technique, have more contrast and details than the absorption image (figure 2.4) of the lichen.

![Figure 2.3: Optical image of lichen.](image)
2.3.2. Phase-Contrast X-ray Imaging (PCXI)

As in the case for lichen, the phase-contrast and dark-field image of a bug revealed much more information about the bug than its absorption image (figure 2.5). Both objects were investigated using a Medipix3 detector.

Several techniques have been developed over the years to produce an X-ray image of an object based on phase shift; namely, crystal interferometer[38], propagation based imaging[39], analyser based imaging[40] and grating interferometry. The grating interferometry-based PCXI is discussed in details below.

**Grating-based Phase-Contrast Imaging**

*Theory*

The grating interferometer consists of a source grating $g_0$, a phase grating $g_1$ and an analyser/absorption grating $g_2$ (Article IV). However, with a microfocus X-ray source, for which the spatial coherency in the beam is sufficient for PCXI, the interferometry can work without $g_0$[41]. Figure 2.6 shows a grating interferometer using a microfocus X-ray source.
Chapter 2. X-ray Imaging

In a set-up, which consists of a conventional X-ray source with a large focal spot, the source grating $g_0$ is placed immediately after the source. The grating is an arrangement of transmission slits, which creates an array of periodically repeating line sources[42]. The phase grating $g_1$ splits the incoming beam and divides it into $+1^{st}$ and $-1^{st}$ diffraction orders[43]. This causes a phase shift of $\pi$ to the passing beam and attenuates it by a negligible amount. The periodic phase modulation in the incident beam caused by $g_1$, is transformed into an intensity modulation on the plane of $g_2$, through the Talbot effect[44, 45]. The Talbot effect is caused by near-field diffraction. Through the Talbot effect, self-image of a periodic diffraction grating (figure 2.7) is produced at certain distances away from the grating, when a plane wave passes through it.

Figure 2.6: Grating interferometer.

Figure 2.7: Self-imaging through the Talbot effect.\(^\dagger\)

\(^\dagger\)Image is partly adopted from W. B. Case et al.[46].
2.3.2. Phase-Contrast X-ray Imaging (PCXI)

$g_2$ has the same periodicity as the fringes produced by $g_1$ and is placed just before the detector. $g_2$ transforms the position of the fringe into an intensity variation. The detector requires the assistance of the transmission properties of $g_2$, because, generally, the detector resolution is not sufficient to resolve the few microns spacing of the interference fringes. In the future, a high resolution detector with a smaller pixel size will enable the construction of a grating interferometry without $g_2$. $g_2$ is scanned along the direction $x_t$, so that the intensity in each pixel in the detector oscillates in relation to $x_t$.

The distance between $g_1$ and $g_2$ for a plane wave, $d_m$ can be calculated by

$$d_m = m \frac{p_1^2}{8\lambda} \tag{2.6}$$

where $p_1$ is the period of $g_1$, $\lambda$ is the wavelength and $m$ is an odd integer representing the order of the fractional Talbot distance. For a set-up using a spherical wave this distance will be

$$d_m^* = \frac{l}{l - d_m} d_m \tag{2.7}$$

where $l$ is the distance between the source and $g_1$. The period of $g_2$, $p_2$ should be the same as the periodicity of the fringes produced by $g_1$ on the plane of $g_2$. The periodicity of the fringes, for the plane wave[47] is

$$p_2 = \frac{p_1}{2} \tag{2.8}$$

For a spherical wave, the period of $g_2$, $p_2^*$ can be obtained by

$$p_2^* = \frac{l + d}{l} p_2 \tag{2.9}$$

where $d$ is a general term for the distance between $g_1$ and $g_2$ for any beam geometry. For a spherical wave, $d = d_m^*$. So, $p_2^*$ can be re-written as

$$p_2^* = \frac{l}{l - d_m^*} p_2 \tag{2.10}$$

**Phase Stepping**

The grating interferometry is designed to detect the deflection in the beam after passing through an object. When an object is placed in front of the phase grating $g_1$, it attenuates the intensity of the beam due to its absorption properties and, also deflects the beam by its refractivity[48]. The deflection angle is given by[49]
Chapter 2. X-ray Imaging

\[ \alpha(x_t) = \frac{\lambda}{2\pi} \frac{\partial \Phi(x_t)}{\partial x_t} \]  (2.11)

where \( \Phi(x_t) \) is the phase profile in the wave-front in relation to the transverse direction \( x_t \).

The deflection angle \( \alpha \) causes a local displacement of \( \alpha \cdot d_m^* \) in the interference pattern at a distance of \( d_m^* \) from \( g_1 \). In this manner, the phase shift \( \Phi(x_t) \) caused by the object’s refractive index, transforms into an oscillated intensity pattern on the plane of \( g_2 \). The absorption grating \( g_2 \) assists in resolving the pattern, since, the limited spatial resolution of the detector makes it difficult to detect the pattern directly.

In order to record the intensity pattern, \( g_2 \) is scanned over its minimum one period along the direction \( x_t \). \( g_2 \) is stepped toward \( x_t \), and at every position an image is taken. The intensity in every pixel in the detector oscillates as a function of the position of \( g_2 \).

Figure 2.8 shows the oscillated intensity data at a single pixel in the detector for different \( g_2 \) positions. The minimum scanning distance from the first data point to the last one is deemed to be one period of \( g_2 \). The number of scan points may vary, but, since the phase retrieval process requires a fully established mathematical system, the minimum number should be three[50]. It is common practice among researchers to use eight scan points per period. In order to reduce the statistical errors, it is better to increase the number of scan points. However, statistical errors also depend on the photon counts in each pixel of the detector in all the scan images.

In order to obtain a phase-contrast image of an object, two sets of scan images are necessary. One scanning should be conducted with the object in front of the phase grating \( g_1 \) and one without the object in order to record...
reference images. All the scan positions for the images with the object should be exactly the same as those for the reference images. Thus, in this manner, two intensity data sets are obtained for every pixel. Two sine curves can be fitted to those data and using the properties of the curves in all pixels, the phase-contrast image can be constructed.

**Image Reconstructions**

There are several methods for constructing a phase-contrast X-ray image of an object using the scan images obtained by grating interferometry. In this chapter, the sine-fitting method is discussed.

![Figure 2.9: Two sets of intensities at a single pixel.](image)

Two sine signals are fitted (figure 2.9) to the two sets of intensity data at a single pixel. The curve at the top shows the intensity data for the reference images without an object and the second curve shows the intensities from the images with an object.

Both curves have an amplitude $A$, phase $\Phi$ and offset $O$. The absorption image $ABS$ is the ratio of the offsets of the two curves[51]

$$ABS = -\log\left(\frac{O_{OBJ}}{O_{REF}}\right)$$  \hspace{1cm} (2.12)

The differential phase image $PHS$ can be obtained by

$$PHS = \Phi_{REF} - \Phi_{OBJ}$$  \hspace{1cm} (2.13)
Chapter 2. X-ray Imaging

One advantage of grating interferometry is that, the dark-field image\[52, 53\] of the object can also be obtained using the same set-up and the same image reconstruction method, with no additional equipment being required. The dark-field image of an object is constructed, based on only those X-ray photons which are scattered while passing through a target object. It is a very useful and efficient method for contrast enhancing. A dark-field signal is very sensitive to the micro-structures and the granularity within objects. The dark-field images of two objects with the same absorption but with different inner structures are significantly different, while their absorption images are almost identical. The dark-field image $DKF$ can be calculated as

$$DKF = \frac{V_{OBJ}}{V_{REF}}$$ \hspace{1cm} (2.14)

where $V_{OBJ} = A_{OBJ}/O_{OBJ}$ and $V_{REF} = A_{REF}/O_{REF}$ are the visibilities of the two signals.

The whole process of calculating the absorption image, the differential phase image and the dark-field image has to be repeated for every pixel in the detector in order to obtain the complete matrices of the three images.

2.4 X-ray Detectors

In the traditional method of X-ray imaging for medical and industrial applications, X-rays are detected on a film after passing the object to be imaged. The film is coated with a light sensitive silver halide, typically, silver bromide. The film can be exposed directly in the beam, but in that case, a long exposure time is required due to the low sensitivity of the film. In order to reduce the exposure time and, thus, to reduce the radiation dose on the objects, an intensifying scintillator screen is often placed before the film to convert the X-rays to visible light.

X-ray images can also be acquired, stored and displayed digitally using X-ray detectors and computers, following the direct conversion method. X-rays produce electron-hole pairs inside the detector material by interacting with the material in the ways discussed earlier in this chapter. The electron-hole pairs can be detected and then processed as an electrical signal[54]. Electronic detectors, together with a photomultiplier and scintillator, can detect X-rays indirectly.

The results presented in this thesis are obtained using the Timepix detector[55], the Medipix3 detector[56] and flat panel detectors, where a semiconductor detector is coupled with a scintillator screen. Both the Timepix and the Medipix3 detectors belong to the semiconductor based Medipix
2.4.1. Medipix Detector

detector family, with different pixel read-out electronics architecture and functionalities. The simulation works discussed in the thesis are based on the PERCIVAL detector.

2.4.1 Medipix Detector

The Medipix collaboration, hosted at CERN has developed a number of hybrid pixel detectors (figure 2.10). All detectors are based on CMOS readout chips with single photon processing. The MEDIPIX collaboration has been active in developing different configurations for more than 15 years. So far all the readout chips developed by the MEDIPIX collaboration share the same geometry with a pixel size of 55 x 55 $\mu m^2$ and a pixel matrix of 256 x 256 pixels.

Figure 2.10: One pixel cell in a hybrid detector[54].

However, the functionality has changed between the different members of the family. MEDIPIX2[57] has two energy thresholds and can thus operate in window mode. Charge sharing between neighboring pixels[58] was detected as a major problem in MEDIPIX2. A key development in the successor MEDIPIX3 was to implement on chip charge summing to improve spectral
resolution. The TIMEPIX chip was derived from MEDIPIX2 by modifying the pixel to either operate in Time-over-Threshold (ToT)\cite{59} or Time-of-Arrival (ToA) mode. The main advantage was that the energy deposited in each pixel could be determined more precisely than by using thresholds and that a spectrum could be obtained in one measurement. Charge sharing can also be corrected by off-line reconstruction. The disadvantage is that the chip only operates in low fluxes since the pixel occupancy has to be kept low to separate individual hits. The TIMEPIX3 chip handles ToT and ToA simultaneously. The chip is also event driven which means that each individual hit is read out immediately. Each chip can handle up to 40 Mhits/s. Multi-pixel events can easily be reconstructed by using the ToA information.

2.4.2 Flat Panel Detectors

A typical flat panel detector consists of a matrix of pixel circuits made in amorphous silicon. A scintillator screen converts the incoming X-rays into visible light photons which are then detected by a photodiode in the pixel, which also stores the signal in a capacitance. Readout is done by addressing the pixels and drifting the stored charge to the readout circuit.

2.4.3 PERCIVAL

The PERCIVAL(Pixelated Energy Resolving CMOS Imager, Versatile and Large) is a MAPS-based soft X-ray detector, intended to be used with highly luminous synchrotron and FEL sources. The requirements include high false-positive rejection rate, low noise, high dynamic range and fast read-out system. The primary design goals are stated in table 2.1.

<table>
<thead>
<tr>
<th>Table 2.1: PERCIVAL design goals.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Energy Range</td>
</tr>
<tr>
<td>Quantum Efficiency (QE)</td>
</tr>
<tr>
<td>Frame Rate</td>
</tr>
<tr>
<td>Pixel Size</td>
</tr>
<tr>
<td>Sensor Sizes</td>
</tr>
<tr>
<td>Noise RMS</td>
</tr>
<tr>
<td>Dynamic Range</td>
</tr>
</tbody>
</table>
2.4.3. PERCIVAL

Figure 2.11 shows two different operation modes of PERCIVAL: the Front Side Illumination (FSI) and the Back Side Illumination (BSI) modes. Back thinning and back illumination are needed in order to achieve the target QE at the aimed photon energy range.

![Figure 2.11: PERCIVAL illumination modes.](image)

In order to minimize the capacitance, the charge collection junction is designed as a Partially-Pinned Photodiode (PPD) [60]. The peripheral circuits (figure 2.12) perform Correlated Double Sampling for noise reduction, gain modulation, Analogue-to-Digital conversion and fast data read-out.

![Figure 2.12: PERCIVAL schematic.](image)
Chapter 2. X-ray Imaging

In order to achieve large dynamic range, PERCIVAL modulate the individual pixel gain according to the incoming photon flux in real time[61]. This is done by adopting the 3T-Active Pixel Sensor(APS) architecture featuring lateral overflow. Under low flux condition, the transistors between the photodiode and the capacitors are biased to disconnect the capacitors, ensuring minimum capacitance, and thus, also low noise. When the incoming flux is high, the capacitance is increased. This reduces the charge-to-voltage conversion factor and therefore, increases the dynamic range.

PERCIVAL Prototypes

In order to investigate the performance of PERCIVAL, two versions of small test sensors have been manufactured and characterized, namely TS1 and TS2, with different collection junction designs (Article V, Article VI). The sensor size is 25 X 25 \( \mu m^2 \), containing 210 X 160 pixels. The TS1 sensor is divided into six sub-matrices following six different photodiode sizes.

Noise

Measurements have been conducted in dark condition in order to record the noise in the TS1 sensor. The mean noise value 14.07 e\(^-\) is smaller than the target value 15 e\(^-\) (figure 2.13). This confirms the usability of PERCIVAL system for single photon discrimination at low energies.

![Figure 2.13: Noise in all pixels within one sub-matrix in PERCIVAL prototype sensor.](image)
2.4.3. PERCIVAL

Uniformity

Figure 2.14 shows response under a uniform illumination provided by a red LED. The temperature of the sensor was at -40°C.

Sensitivity

In order to verify the sensitivity of PERCIVAL to low energy photons, tests were performed at the I-10 beamline in the Diamond Light Source. A beam of 400 eV photons was used to create an airy disk diffraction pattern (figure 2.15) through a 5 μm pinhole, placed 2.4 m away from the sensor. The patterns have been recorded by PERCIVAL. A careful comparison of the distribution of maxima and minima[62] in the recorded image to the expected pattern reveals that the detector response is dominated by the main harmonic (400 eV) and also, that the contributions from higher harmonics are negligible.
Chapter 2. X-ray Imaging

Figure 2.15: Airy disk patterns generated by a 5 µm pinhole.

Charge Collection Efficiency (CCE)
The 400 eV beam at I-10 was used to estimate the CCE of PERCIVAL, because of the absence of the higher harmonics. A combination of slits kept the beam area smaller than the detector area. A calibrated Hamamatsu 1127-04 GaAsP photodiode has been inserted in front of the detector, in order to calculate the incoming flux by measuring the photodiode current. It was made sure that the entire beam after the slits hits the photodiode.

Then the photodiode was moved away and the detector was exposed to the same beam. A series of images were acquired, averaged and the total charge collection by the detector have been estimated. The total collected charge has been compared to the maximum charge corresponding to the incoming photon flux. This procedure was repeated several times in order to average out random errors in the measured values, and the CCE has been estimated to be 84 - 85 %. Figure 2.16 shows the CCE of the PERCIVAL detector at 400 eV.
Figure 2.16: Charge Collection Efficiency.
Chapter 3

GEANT4 SIMULATION

*If I have seen further, it is by standing on the shoulders of giants.*
– Isaac Newton.

Geant4 is a Monte-Carlo based object-oriented simulation platform and is equipped with set of libraries necessary to perform simulations of particle interactions inside matter[63, 64, 12]. It is scripted in the C++ programming language. When provided with a primary beam, physical structure of a detector and relevant physics definitions for interactions, Geant4 kernel can perform the simulation and generates outputs (figure 3.1).

![Geant4 framework](image)

Figure 3.1: Geant4 framework.

3.1 Simulation of PERCIVAL

The energy depositions inside the PERCIVAL (BSI) epitaxial layers by photons with different energies have been simulated.
Chapter 3. Geant4 Simulation

3.1.1 Detector Construction

The detector has been constructed with three layers; namely, a sensitive absorber layer (epitaxial), a metal layer and a bulk layer (figure 3.2).

![Diagram of detector layers](image)

Figure 3.2: A small segment of the simulated detector, together with the thicknesses of the layers.

The epitaxial layer has a pixelated structure and contains 70 X 80 pixels (same as a PERCIVAL sub-matrix). The pixel pitch is 25 \( \mu m \). Each pixel is defined as a sensitive volume.

The metal layer contains a mixture of 40% Al and 60% SiO\(_2\). The idea behind this simplistic adoption is to replicate the layer with circuitry in the actual detector.

3.1.2 Physics Processes

Geant4 was initially intended for HEP simulations. The Geant4 electromagnetic (EM) physics models contain definitions of physics processes in order to simulate electromagnetic interactions of particles with matters. The Geant4 standard EM physics model is valid between the energy range of 1 keV - 10 PeV[65].

The primary target energy range of PERCIVAL is 250 eV - 1 keV, which is below the energy range of the Geant4 standard EM physics model. For this reason, special low energy physics model is needed, such as Livermore
3.1.3 Set-up

model, Penelope model, Geant4-DNA processes, etc[66]. In this simulation Livermore model has been used with the following physics model classes:

- G4LivermorePhotoElectricModel
- G4LivermoreComptonModel
- G4LivermoreRayleighModel
- G4LivermoreGammaConversionModel
- G4LivermoreIonisationModel
- G4LivermoreBremsstrahlungModel

3.1.3 Set-up

In the simulated set-up the detector and the particle source are placed within a "world" volume (figure 3.3). The world volume has a dimension of .8m X .01m X .01m and has vacuum inside. The distance between the source and the detector surface is 76 cm. The source is modelled in a way that the whole detector area is under illumination. The half opening angle of the source[67] is $0.2^\circ$.

Figure 3.3: Simulated set-up.
Chapter 3. Geant4 Simulation

3.1.4 Results

Figure 3.4 shows energy depositions after all interactions by photons at 4 keV inside the Si epitaxial layer. This was done in order to verify the functionalities of the physics processes. The peak at 1.82 keV is a Si fluorescence peak and the peak at 2.17 keV is the corresponding escape peak. From the source 10000 photons were generated. The area of the beamfront, ABF at 76 cm is,

\[
ABF = \pi \times (0.76 \times \tan(0.2))^2 m^2
\]

\[
\Rightarrow ABF = 0.00022 m^2
\]

The area of the detector, DA is,

\[
DA = 70 \times 25 \times 25 \times 25 \mu m^2 = 0.000035 m^2
\]

The number of photons, N, that hit the detector surface can be calculated as,

\[
N = \frac{DA}{ABF} \times 10000 \approx 1590
\]

![Figure 3.4: All interactions inside the Si layer.](image)

The absorption of a 4 keV photon beam inside 12 \( \mu m \) thick Si is around 72\%[68]. So, the number of photons absorbed inside the Si layers, \( N_{\text{abs}} \) is,

\[
N_{\text{abs}} = 0.72 \times 1590 \approx 1145
\]
3.1.5. Comparison with Measurements

In order to construct the pixelated sensitive layer, first one pixel has been defined. Then that pixel volume has been replicated to create a single column and then, the column to create the whole structure. The G4PVReplica class has been used for the replication.

The total energy deposition inside a single pixel can be calculated by summing-up all energies, deposited by all interactions within that pixel. If this calculation is repeated for all pixels, the spectrum of energy deposition per pixel can be produced (figure 3.5).

![Energy deposition spectra](image)

Figure 3.5: Spectra of energy deposition per pixel at different energies, 10M photons from the source.

3.1.5 Comparison with Measurements

Measurement has been conducted at DESY with an Fe55 source. The source produces K_{\alpha 1}, K_{\alpha 2} and K_{\beta} X-rays at 5.89875, 5.88765 and 6.49045 keV, respectively. These X-ray energies are very close to each other, and often mentioned as mono-energetic 5.9 keV X-rays all together. Fe55 source was chosen, because it is a commonly available low energy near mono-chromatic source, which allows measurements in a lab, avoiding waiting time for a synchrotron beam line. At the time of this measurement, 34 photons (per image in a PERCIVAL sub-matrix containing 70 X 80 pixels) were estimated
to interact inside the sensor. A total of 25000 images have been recorded, each having 500 ms of exposure time. The distance between the source and the sensor (76 cm) was same as in the simulated set-up.

The simulation has been repeated at 5.9 keV photon energy. For 25000 images, total number photons that interact inside the sensor, $N_I$ should be,

$$N_I = 34 \times 25000 = 850000$$

The absorption of 5.9 keV photon beam inside 12 $\mu m$ Si is about 35%. Which indicates that the total flux at the simulated detector surface, $N_T$ should be,

$$N_T = \frac{100}{35} \times N_I \approx 2428571$$

Finally, the number of photons ($N_P$) from the primary source in order to perform the simulation was calculated to be,

$$N_P = \frac{N_T}{DA} \times ABF \approx 15265306$$

Figure 3.6: Collected charges.

Figure 3.6 shows the collected charges per pixel from the measurement and the simulation. Deposited energies in the simulation were divided by 3.6 (minimum 3.6 eV needed to create an e-h pair in Si[69]) to estimate the
3.1.5. Comparison with Measurements

charge collections. In the measured plot, the peaks at 0 and at around -1100 correspond to noise and false positives, respectively. The primary photon peak at \( \sim 1638 \, e^- \) and the escape peak at \( \sim 1138 \, e^- \) are visible, both in the measurement and the simulation. However, the fluorescence peak at \( \sim 500 \, e^- \) is not visible in the measured plot.

The measured charges are lower than those in the simulation. The prototype PERCIVAL chip used for the measurement, was later found to be highly contaminated. That highly contaminated chip showed unusually high recombination rate, which might be the main reason for low charge collections.
Chapter 4

TCAD SIMULATION

Reality is merely an illusion, albeit a very persistent one.
– Albert Einstein.

It is of great interest to perform physics simulation of semiconductor devices, since, the conventional "fabricate and test"-based development methods are time consuming and expensive[70]. In order to quantify the charge collection and the charge sharing between neighbouring pixels in PERCIVAL, simulations have been performed using the Synopsys Sentaurus TCAD tools. Charge sharing between pixels degrades spectral and spatial resolution of detectors (Article I, Article III).

4.1 Sentaurus TCAD

Sentaurus TCAD is a Finite Element Method[71]-based simulation platform. In this platform, the whole detector is divided into a finite number of small regions. Then, the partial differential equations for carrier behaviour in an external electric field are solved within each of those small regions.

Three partial differential equations are solved in this charge transport simulation, namely, the Poisson equation and the continuity equations for electrons and holes[72]. The Poisson equation calculates the electrostatic potential based on the local charge densities and can be written as,

$$\varepsilon_{Si} \nabla^2 \Psi = -q(p - n + n^+_D - n^+_A)$$  \hspace{1cm} (4.1)

where,

$$\varepsilon_{Si} = \text{Permittivity of Si}$$

$$\Psi = \text{Electrostatic potential}$$

$$q = \text{Charge of an electron}$$
The continuity equations for electrons and holes describe the drift-diffusion transport model for the carriers and are as follows,

\[
\nabla \vec{J}_n = qR + \frac{\delta n}{\delta t}
\]

\[
\nabla \vec{J}_p = qR + \frac{\delta p}{\delta t}
\]

where,

\[ R = \text{Recombination rate} \]

\[ \vec{J}_n = \text{Electron current density} = -nq\mu_n \nabla \Phi_n \]

\[ \text{and} \vec{J}_p = \text{Hole current density} = pq\mu_p \nabla \Phi_p \]

where \( \mu_n, \mu_p, \Phi_n, \) and \( \Phi_p \) are the electron mobility, the hole mobility, the electron quasi-Fermi potential and the hole quasi-Fermi potential, respectively.

4.2 Simulation of PERCIVAL

A 2D structure of a PERCIVAL segment has been created and simulated. The equations 4.1, 4.2 and 4.3 have been solved within each element inside the volume of the segment, created by meshing.

4.2.1 Detector Structure

Using the Sentaurus Structure Editor (SDE) tool, a 2D structure of PERCIVAL (epitaxial layer) containing 10 pixels has been constructed (figure 4.1). The command file to create the structure is written in the Scheme programming language. The pixel pitch is 25 \( \mu m \). The thickness of the simulated sensitive layer is 12 \( \mu m \) and made of Si.
4.2.2. Device Simulation

Figure 4.1: Simulated PERCIVAL structure.

Every pixel in PERCIVAL has a nearly rectangular diode on top of it, which appears as two separate diodes in a 2D cross-sectional view (figure 4.2).

Figure 4.2: One pixel with charge generation point in its middle.

Charge can be generated anywhere inside the detector volume. The location of charge generation with its surroundings and the regions with high doping concentration have finer meshing than other regions. The mesh is created using the Sentaurus Mesh (SNMESH)[75] tool. The anodes are set on top of the diodes and the cathodes are between the pixels.

4.2.2 Device Simulation

The device simulations were performed in two steps. At first, a quasi-stationary simulation has been conducted to set right bias to the contacts. Then, a transient simulation was run. During the transient simulation, charge was generated using the built-in Heavy Ion model. Both of these
simulations were performed by running command files in the Sentaurus Device (SDEVICE) tool.

4.2.3 Results

In order to verify the physics models and the definitions used in the simulation, a simple pn-diode has been created and simulated. The pn-diode has uniform doping concentrations at the p and the n regions. Two contacts were set to the p-surface and the n-surface. When both contacts were grounded, the built-in potential was simulated to be 0.692 V (figure 4.3) at 300 K. It is almost similar to the theoretically calculated value[77], which is 0.715 V at the same temperature.

Figure 4.3: Built-in potential.
Figure 4.4: Dark current.

After the verification of the physics in the simulation, next step was to check the dark current in the detector. Dark current is the current that flows through the detector without any radiation and produced by thermal excitation[78]. The detector was biased with 1 V. The current in each anode was collected[79, 80] and then integrated, in order to calculate the charge[81]. Charges in two anodes which belong to a single pixel were added together, and the sum was divided by electron charge to calculate the total collected electrons in that pixel. The dark current in all pixels in the simulated detector (figure 4.4) can be considered as negligible.

Charges equivalent to 500 eV (140 e⁻), 1000 eV (280 e⁻) and 5900 eV (1640 e⁻) were generated at the same location as shown in figure 4.2 and a transient simulation has been performed in each of the cases. After the simulation, the drift-diffusion of the generated charges towards the anodes at several time points can be plotted (figure 4.5).
4.2.3. Results

Figure 4.5: Charge transport inside the detector over time (5000 eV).
Chapter 4. TCAD Simulation

Figure 4.6 shows the collected charges in the pixel where the charges were originally generated and the charges which spread into the neighbouring pixels.

![Figure 4.6: Charge sharing between pixels.](image)

Alternatively:

Figure 4.6: Charge sharing between pixels.
Chapter 5

DETECTOR APPLICATIONS

It was a great step in science when men became convinced that, in order to understand the nature of things, they must begin by asking, not whether a thing is good or bad, noxious or beneficial, but of what kind it is? And how much is there of it? Quality and Quantity were then first recognised as the primary features to be observed in scientific inquiry.

– James Clerk Maxwell.

Short after Röntgen’s announcement, doctors around the world started to use X-rays to locate fractures in bones. Within a year, a whole radiology department was opened in a hospital in Glasgow. The uses of X-ray transformed modern medicine.

Today, the applications of X-rays are not limited to only hospitals. Numerous industrial, security, environmental and academic research applications have been developed over more than a century, because of the non-destructive nature of X-rays.

Improvements in detector technologies and the adoption of computer systems have revolutionised X-ray imaging. Now, advanced X-ray systems are being used in a wide range of industries for quality inspection. In this chapter novel methods for quality inspection in paperboard industries using X-rays are discussed. Also, X-ray fluorescence measurements to detect toxic components in water are presented.

5.1 Resolving Paperboard Layer Thicknesses

Using the X-ray attenuation principle, the thicknesses of the layers in a multi-layered structure with known content, such as paperboard, can be calculated[82] (Article VII).

If \( t_1 \) and \( t_2 \) represent the thicknesses of the layers in a two-layered paperboard, and \( \alpha_{1\gamma} \) and \( \alpha_{2\gamma} \) represent the linear attenuation coefficients (dependant on photon energy and material[83]) of the materials inside the
layers at a certain energy $e_1$, then the X-ray response from the primary interactions can be written as

$$\phi_{e_1} = \phi_{0_{e_1}} e^{-(\alpha_{1_{e_1}} t_1 + \alpha_{2_{e_1}} t_2)}$$  \hspace{1cm} (5.1)$$

$$\Rightarrow \alpha_{1_{e_1}} t_1 + \alpha_{2_{e_1}} t_2 = -\ln(\frac{\phi_{e_1}}{\phi_{0_{e_1}}})$$  \hspace{1cm} (5.2)$$

This equation shows that it is not possible to calculate $t_1$ and $t_2$ with only one energy. If another energy $e_2$ is used, then the response is

$$\phi_{e_2} = \phi_{0_{e_2}} e^{-(\alpha_{1_{e_2}} t_1 + \alpha_{2_{e_2}} t_2)}$$  \hspace{1cm} (5.3)$$

$$\Rightarrow \alpha_{1_{e_2}} t_1 + \alpha_{2_{e_2}} t_2 = -\ln(\frac{\phi_{e_2}}{\phi_{0_{e_2}}})$$  \hspace{1cm} (5.4)$$

where $\alpha_{1_{e_2}}$ and $\alpha_{2_{e_2}}$ represent the linear attenuation coefficients of the materials at energy $e_2$.

Equation 5.4 can be written as

$$t_2 = -\frac{\ln(\frac{\phi_{e_2}}{\phi_{0_{e_2}}}) + \alpha_{1_{e_2}} t_1}{\alpha_{2_{e_2}}}$$  \hspace{1cm} (5.5)$$

By putting this value of $t_2$ into equation 5.2,

$$\ln(\frac{\phi_{e_1}}{\phi_{0_{e_1}}}) = -\alpha_{1_{e_1}} t_1 + \frac{\alpha_{2_{e_1}}}{\alpha_{2_{e_2}}} (\ln(\frac{\phi_{e_2}}{\phi_{0_{e_2}}}) + \alpha_{1_{e_2}} t_1)$$  \hspace{1cm} (5.6)$$

$$\Rightarrow \ln(\frac{\phi_{e_1}}{\phi_{0_{e_1}}}) = t_1(-\alpha_{1_{e_1}} + \frac{\alpha_{2_{e_1}} \alpha_{1_{e_2}}}{\alpha_{2_{e_2}}}) + \frac{\alpha_{2_{e_2}}}{\alpha_{2_{e_2}}} \ln(\frac{\phi_{e_2}}{\phi_{0_{e_2}}})$$  \hspace{1cm} (5.7)$$

$$\Rightarrow t_1 = \frac{\ln(\frac{\phi_{e_1}}{\phi_{0_{e_1}}}) - \frac{\alpha_{2_{e_1}}}{\alpha_{2_{e_2}}} \ln(\frac{\phi_{e_2}}{\phi_{0_{e_2}}})}{-\alpha_{1_{e_1}}(1 - \frac{\alpha_{2_{e_1}} \alpha_{1_{e_2}}}{\alpha_{2_{e_2}} \alpha_{1_{e_1}}})}$$  \hspace{1cm} (5.8)$$

$$\Rightarrow t_1 = \frac{\frac{\alpha_{2_{e_1}}}{\alpha_{2_{e_2}} \alpha_{1_{e_1}}} \ln(\frac{\phi_{e_1}}{\phi_{0_{e_1}}}) - \frac{\alpha_{2_{e_1}}}{\alpha_{2_{e_2}} \alpha_{1_{e_1}}} \ln(\frac{\phi_{e_2}}{\phi_{0_{e_2}}})}{1 - \frac{\alpha_{2_{e_1}} \alpha_{1_{e_2}}}{\alpha_{2_{e_2}} \alpha_{1_{e_1}}}}$$  \hspace{1cm} (5.9)$$

Now, $t_2$ can also be calculated by putting the value of $t_1$ in Eq. 5.4.

The denominator in Eq. 5.9 will be zero if the ratios between the absorption coefficients for the two materials at the energies of interest are similar. In that case, the thickness calculation will give a large error.
5.1. Resolving Paperboard Layer Thicknesses

This method can be applied using a wide spectrum X-ray source and a well calibrated spectroscopic detector system. In order to separate the calcium carbonate layer from the core layer, it is important to use energies which are just above and below the k-edge of Ca at 4 keV[84]. The spectral resolution, offered by the imaging system using a wide spectrum X-ray source at the energy range of interest, was not sufficient. For this reason, CaO and Ti were used as 3.7 keV and 4.5 keV fluorescence X-ray sources. A microfocus X-ray tube with a Tungsten target was used as the primary X-ray source.

The Timepix readout system with Silicon sensor has been used in the measurements. This detection system has a noise floor at 2.34 keV, which is necessary in order to detect the low fluorescence energies of CaO and Ti. The sensitivity profile of Silicon as a detector material was investigated (Article II).

Using a Millitast 1083 instrument, the thicknesses of the investigated paperboard sample in total, the core layer and the coating layer were measured to be 490\(\mu m\), 430\(\mu m\) and 60\(\mu m\), respectively. The sample was prepared in such a way that a single X-ray image frame can contain all the areas to be investigated (figure 5.1).

![Image](image_url)

Figure 5.1: Optical image of paperboard (P), only coating (Q) and only cellulose layer (R).

X-ray images of the prepared sample were taken using the fluorescence sources and, equation 5.9 and 5.5 were applied to calculate the thicknesses (figure 5.2) of the paperboard layers.
Chapter 5. Detector Applications

Figure 5.2: Thickness profiles of the separated (a) coating and (b) cellulose layer.

The measured thicknesses of the coating and the cellulose layer are different from their actual thicknesses. This is because, the used method considers only the primary X-ray interactions within the sample. Also, the measurement is affected by high energy Compton contributions from the fluorescence sources, which was verified by a Monte Carlo N-Particle simulation. It was assumed that the core and the coating layer consist only of cellulose and calcium carbonate, respectively. In reality, they also contain air gaps, fillers and ink pigments, if produced with recycled fibres. In a practical application, the measurement system has to be calibrated depending on the amount of these contents in the paperboard. Fluorescence sources yielded a large focal spot, resulting in a low spatial resolution. The measurement is also affected by the measurement errors due to the large energy bin size of the used detector system. A detector system with a bin size of 0.4 keV might limit the relative errors to 10 %, leading to a more accurate thickness calculation.

5.2 Uniformity in the Cellulose Layer in Paperboards

The uniformity in the coating layer is affected by the cellulose structures inside the core layer. In an ideal case, all paperboards which are produced using the same coating technique and material, should have a similar quality, i.e. same burn-out index\[85\] (also discussed in the next section). But, in reality, due to the differences in the cellulose layer structures, they have different burn-out indices. Phase-contrast X-ray imaging has been used with a flat panel detector to investigate four paperboard samples with different burn-out indices, which are produced using the same method and material, to characterize their cellulose layer structures (Article VIII).
5.2. Uniformity in the Cellulose Layer in Paperboards

The investigated samples have 8.23, 9.81, 13.47 and 16.87 as their burn-out indices. The samples with the burn-out numbers 8.23 and 16.87, are considered as very good and bad paperboards, respectively, according to the paperboard industry standard. The recorded dark-field signals from the four samples differ from each other. The dark-field signal of an object is highly dependent on the micro-structures inside that object.

In order to further characterize the scattering power of the fibre structures, four sinusoidal functions were fitted to the normalised (due to variations in thickness) logarithmic dark-field signals (figure 5.3) from each sample at all orientations[86, 87]. The isotropic and anisotropic scattering coefficients of the samples (table 5.1) were extracted from the dark-field signals. The fibre orientation on the core of the paperboards follows the direction of their production. When the fibre orientation is parallel to the grating bars in the interferometry, the sum of the isotropic and anisotropic dark-field signals are measured. However, only the isotropic signals are measured if the fibres are oriented perpendicularly. These two coefficients can be used to understand the fibre structures inside paperboards.

Figure 5.3: The logarithm of the dark-field signal as a function of the sample orientation.
Table 5.1: Isotropic (A) and anisotropic (B) scattering coefficients of the samples.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Burn-out index</th>
<th>A</th>
<th>B</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sample 1</td>
<td>8.23</td>
<td>0.2448</td>
<td>0.0730</td>
</tr>
<tr>
<td>Sample 2</td>
<td>9.81</td>
<td>0.2392</td>
<td>0.0534</td>
</tr>
<tr>
<td>Sample 3</td>
<td>13.47</td>
<td>0.2210</td>
<td>0.0602</td>
</tr>
<tr>
<td>Sample 4</td>
<td>16.87</td>
<td>0.2125</td>
<td>0.0681</td>
</tr>
</tbody>
</table>

It has been observed that the lower the burn-out index, the higher the isotropic scattering coefficient of a sample. This correlation (figure 5.4), if performed by taking multi-layered fibre structures into account, can be used to replace the burn-out method, allowing for non-destructive and automatic quality inspection of paperboards.

Figure 5.4: The isotropic scattering coefficients of the samples in relation to the burn-out indices.

In order to extract the directional scattering coefficients for all samples, sine-fitting has been performed to the dark-field signals from each sample at four relative angles. Mathematically, measurements at four angles are sufficient to construct a periodic modulation[88] of the dark-field signals, which is necessary for the calculations of the scattering coefficients. Measurements with more samples and at more angles might produce a more precise correlation.
5.3 Replacing Burn-out Methods

In the burnout test method, a paperboard sample is treated with an NH$_4$Cl test solution (25 g/liter NH$_4$Cl in a 50/50 volume mixture of 2-Propanol /water), after which it is placed under a heat gun or in an oven for two minutes to burn it out. The burnt sample (figure 5.5) is then examined for color change/development. The intention is to carbonize the paperboards’ contents and produce contrast between the layers. Areas with thicker cellulose layer and thinner coating layer appear darker in the burnt sample.

The burnt sample is either observed by a human or analyzed by a computer software. Based on the texture of the burnt sample, a quality index is produced and assigned to the sample. This index is called burnout index and commonly used by the industries as a standard.

![Figure 5.5: Photo of burnt paperboard.](image)

This method is a destructive process and the samples cannot be processed back to their original state. The process lacks in repeatability and can be affected by many factors, e.g. temperature, chemical concentrations and solution distribution over the sample surface. The burnout method can be applied only to certain types of paperboard[89].

Considering these problems and the related disadvantages, a non-destructive method, such as X-ray imaging, could help the industries to perform the quality indexing without destroying the samples (Article IX). Analysing and quality indexing could be done based on the correlation between the visible patterns in the burnt samples and the structures in the
reconstructed phase-contrast images. Measurements using X-ray are repeatable, and offer better consistency comparing to measurements involving chemical treatments. It would be possible to take the whole production under observation, if the system is automated, including final analysing with a computer.

PCXI method has been applied with a flat panel detector in order to image a paperboard sample. The resultant images have been analysed in order to observe a correlation between the visible patterns in the burnt sample and the structures in the phase-retrieved image (figure 5.6).

Figure 5.6: (a) Phase-retrieved X-ray image of the sample. Red contour line is selected based on color difference. (b) Photo of the same paperboard with contour lines from the X-ray image.

The above measurement has been repeated for two more samples. A direct correlation in patterns is found between the phase-retrieved X-ray projection images and photos of the burnt samples, burnt using traditional burnout method. The results indicate that the method can be directly applied on paperboard to produce burn-out like images, skipping the burning procedure.
5.4 Detecting Cr in Water

In this work, measurements using X-ray fluorescence have been performed with a CdTe-based spectrometer in order to detect Cr in water (Article X). Measurements were conducted on several water samples with different known Cr concentrations. The same method can be used in landfills or waste dumping stations in order to monitor Cr concentration in leachate. This would allow fast and also, continuous and automatic inspection.

The energy calibration of the detector was performed using the fluorescent photons with an energy of 3.7, 6.4 and 8.04 keV, coming from Calcium Carbonate, Iron and Copper, respectively. The X-ray tube voltage was 20 kV. The detector has been used to record the fluorescent photons from normal water without Cr and also, from the water samples with added Cr concentrations of 0.5, 1.0, 2.0, 3.0, 4.0 g/L. In order to suppress the background or noise signals, the signal from normal water was subtracted from the signals generated by the samples with Cr.

![Figure 5.7: Measured spectra from water with different Cr concentrations (background subtracted).](image)

Figure 5.7 shows the Cr peaks at 5.4 keV in the spectra recorded from all of the samples. It is evident that the Cr peak gets higher as Cr concentration in water increases.
Chapter 5. Detector Applications

In order to calculate the characteristic Cr photon counts from each sample, the Cr peaks in all measured spectra have been separated after subtracting background and then, integrated. The Cr K$_\alpha$ photon counts from the samples show nearly a linear relation with respect to the level of Cr in them (figure 5.8).

Figure 5.8: Cr K$_\alpha$ photon counts from water with different Cr concentrations.
CONCLUSIONS

A conclusion is the place where you got tired thinking.
– Martin H. Fischer.

In this thesis, the technology of the PERCIVAL monolithic active pixel sensor has been discussed. A PERCIVAL prototype detector has been characterized in order to study its uniformity, sensitivity to the low target energy range, noise properties and charge collection efficiency. Simulation of energy deposition inside and electrical properties of PERCIVAL have been conducted. The simulation results will contribute to the further developments of the PERCIVAL design.

Measurements have been performed with a PERCIVAL prototype detector using 5.9 keV photons coming from an Fe55 source. The measured spectrum of collected charge has been compared to a simulated one. The used prototype detector was later diagnosed with high contamination and thus, had high recombination rate. This could be the reason behind the low charge counts in the measured spectrum.

The drift-diffusion transient simulation of charge transport inside the PERCIVAL epitaxial layer was useful to see the time required for charge collection and charge sharing between pixels. Most of the generated charges are collected in the two closest anodes from the generation point in the simulated 2D structure, under an electric field created by external biasing. Charge sharing at three different energies have been investigated. As expected, simulation showed that the charge sharing increases as the energy gets higher.

Several industrial and environmental applications of other modern X-ray detectors have been demonstrated. Three novel methods to inspect product qualities in the paperboard industry have been developed. All of these methods can potentially be used for non-destructive quality controlling in the industry. The methods are described below.

• Spectroscopic X-ray imaging was used to measure the thicknesses of the layers inside a paperboard. Due to the limited spectral and
Chapter 6. Conclusions

Spatial resolution offered by the measurement set-up, the results were different from the real thicknesses of the layers inside the sample. Suggestions have been made in order to improve the measurements.

• It was possible to measure the X-ray scattering dependant dark-field signals from several paperboard samples with different quality indices using phase-contrast imaging technique. Isotropic and anisotropic scattering coefficients have been calculated from the dark-field signals. A near-linear correlation has been observed between the isotropic coefficients and the quality indices of the samples.

• Phase-contrast X-ray imaging could resolve the cellulose structures inside paperboards. The resemblances between the resolved structures in the phase-contrast images and those, visible in the corresponding burnt samples were astonishing. This method can be used to produce quality index like burn-out index without burning samples.

A commercial spectrometer has been used to record the characteristics fluorescent photons coming from water samples with different Cr concentrations. The $K\alpha$ photon counts from the samples found to be linearly related to the Cr levels in the samples. This method can be used to detect contamination of Cr and other toxic elements in leachate in waste-dumping sites.

The advancement in detector technology and imaging techniques, and their new applications are dependant on each other. Detectors with new and useful features will open the doors to novel applications. In other way, innovative applications will set demands and motivate the scientific community to push forward the science of detectors beyond its limit.
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