Development of Calibration Methods for a Photon Emission Microscope to Analyse Light Emitted from Semiconductor Detectors

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Contents

1 Introduction ................................................. 1

2 Silicon Photomultiplier .................................. 3
   2.1 Principle of the Avalanche Diode .................. 3
   2.2 Operating Principle of a SiPM ....................... 4
   2.3 Properties of SiPMs .................................... 5
      2.3.1 Photon Detection Efficiency .................. 5
      2.3.2 Dark Rate ........................................... 5
      2.3.3 Optical Crosstalk .................................. 6
   2.4 Back Illuminated SiPM at the HLL .................. 6

3 Theory of Photon Emission in Semiconductors .... 9
   3.1 Fundamental Radiative Transitions ............... 10
      3.1.1 Transitions between Conduction Band and Valence Band ............. 10
         3.1.1.1 Direct Transitions .............................. 10
         3.1.1.2 Indirect Transitions ......................... 11
      3.1.2 Transitions between Donator/Acceptor Level and a Band ............... 12
         3.1.2.1 Shallow Transitions ......................... 12
         3.1.2.2 Deep Transitions ............................. 13
      3.1.3 Intraband Transitions ............................. 14
      3.1.4 Exciton Recombination ........................... 14
   3.2 Photon Emission from Avalanche Breakdown in Silicon Diodes......... 16

4 Photon Emission Microscopy with the PHEMOS-1000 19
   4.1 The PHEMOS-1000 ....................................... 20
      4.1.1 The Microscope .................................... 20
         4.1.1.1 The Optics ..................................... 22
5 The Signal Calibration

5.1 Calibration of the System Response with Light

5.1.1 The Equipment for the Calibration of the System Response with Light

5.1.1.1 Spectrometer

5.1.1.2 Light Source

5.1.1.3 Neutral Density Filters

5.1.1.4 The Fibers

5.1.1.5 The Integrating Sphere

5.1.1.6 The Interference Filters

5.1.2 Implementing the Calibration of the System Response with Light

5.2 Calibration of the Detector Response

6 Measurements

6.1 Avalanche Diode Array with a High Field Region of 10µm Diameter

6.1.1 Analysis of the 10µm Array

6.1.1.1 The Total Array Emission

6.1.2 Analysis of a 10µm Cell

6.1.2.1 Comparison of the Total Light Emission of Cell [1,3] vs. Cell [3,3]

6.1.2.2 Cell Analysis of Cell [1,3]

6.1.2.3 Cell Analysis of Cell [2,3]

6.1.2.4 Cell Analysis of Cell [3,3]

6.2 Avalanche Diode Array with a High Field Region of 25µm Diameter

6.2.1 Analysis of the 25µm Array

6.2.1.2 The Total Array Emission

6.2.2 Analysis of a 25µm Cell

6.2.2.1 Analysis of Cell [3,3]

6.3 Estimate of the Minimal Detectable Photon Rate

7 Summary and Conclusions
Appendix A

A.1 Transmission of the Fibers .................................................................66
  A.1.1 Transmission of the 50µm Fiber ....................................................66
  A.1.2 Transmission of the 100µm Fiber ..................................................67
  A.1.3 Transmission of the 400µm Fiber ..................................................68
A.2 Calibration Data of the System Response with Light .....................69
  A.2.1 Calibration Data for 20x Lens at Low Amplification ....................69
  A.2.2 Calibration Data for 20x Lens at High Amplification ....................70
  A.2.3 Calibration Data for 100x Lens at Low Amplification .................71
  A.2.4 Calibration Data for 100x Lens at High Amplification .................72
A.3 Image Processing .................................................................73
  A.3.1 Image Mapping .................................................................73
  A.3.2 3D Emission Image .................................................................74
A.4 Different Camera Systems ............................................................75

Literature .................................................................77
Introduction

The Halbleiterlabor (HLL) of the Max-Planck Institutes designs and manufactures semiconductor detectors for imaging, spectroscopy and tracking of particles. The detectors are used in a wide field of applications from astrophysics to material science. Back illuminated pn-CCDs offer highest frame rates, radiation hardness, 100% fill factor and high quantum efficiency in a wide spectral range, for NIR photons as well as for X-rays up to 30keV. A novel CCD type incorporating an electron multiplying readout for low light level imaging with single photon sensitivity is being developed. Many research groups still using photomultiplier tubes are interested in silicon photomultipliers (SiPMs) for single photon counting because of their small size, low operating voltage, high multiplication factor, insensitivity to magnetic fields and low costs.

The SiPM principle is based on the avalanche effect. A charge carrier generated by a photon excites an avalanche breakdown in a reverse biased pn-junction. However one disadvantage of this technology is that the avalanche breakdown itself generates photons by electroluminescence. Thus in an array of avalanche diodes there is an coupling between neighbouring cells, called optical crosstalk, which might affect the correlation between the number of incident photons and signal pulses. Since the back illuminated SiPMs developed by the HLL rely on a fully depleted bulk to obtain high quantum efficiency, the crosstalk probability is potentially higher. It is an inevitable need to minimise the optical crosstalk. In order to develop effective countermeasures the light emission of an avalanche breakdown has to be understood in detail.

In this thesis the location, the intensity and the spectral distribution of the light emission are analysed. For this purpose a photon emission microscope (PEM) is used as basic system. Originally the PEM was developed to localise weak light emissions in faulty integrated circuits. In order to measure the intensity and the spectral distribution of the light emission the PEM is modified and a spectral signal calibration is implemented. Additional equipment is added to the PEM including a calibrated light source, spectrometer, integrating sphere, bandpass filters and neutral density filters. Several laboratory prototypes of avalanche diode arrays are analysed by the advanced PEM.

This thesis has the following structure: In Chapter 2 the principle of the avalanche diode and the characteristic features of the SiPMs are explained. Chapter 3 lists radiative transitions in semiconductors and several light emission mechanisms in avalanche diodes. The technical details of the PEM are presented in Chapter 4. In Chapter 5 the signal calibration is described followed by the analysis of two avalanche diode arrays in Chapter 6. Chapter 7 contains the summary and conclusions of this work.
Chapter 2

Silicon Photomultiplier

Silicon Photomultipliers (SiPMs) are detectors for single photon counting [1]. A SiPM is an array of cells connected in parallel, each consisting of a Geigermode photodiode, a charge and discharge circuitry. The basic principle is, that every cell generates the same signal per incident photon and that single photons can be counted by adding the signal pulses of the whole array. Therefore the dynamic range of the array depends on the density of cells.

The advantages of SiPMs are the compact size, low operating voltage, high multiplication factor, insensitivity to magnetic fields and high photon detection efficiency. Drawbacks of this technology are the dark rate and the optical crosstalk [2]. Front illuminated (FI) SiPMs have recently become commercial products. The MPI-HLL develops back illuminated (BI) SiPMs which compared to FISiPMs offer a higher quantum efficiency and 100% fill factor. In this chapter, the principle of the avalanche diode and the characteristic features of the SiPMs are explained and the basic principle of the BISiPMs is described.

2.1 Principle of the Avalanche Diode

An avalanche diode consists of a reverse biased pn-junction. In order to generate an avalanche breakdown, two conditions must be fulfilled:

- The reverse bias must be higher than the breakdown voltage.
- At least one free carrier has to exist in the high field region to initiate the breakdown.

The first condition is guaranteed by an external power supply and by doping. A free carrier can be either generated thermally or by external excitation, e.g. by light. The free carrier is accelerated by the electric field in the avalanche diode and gaining so much energy that subsequently it generates new electron-hole-pairs by impact ionisation [3] which again generate further electron-hole-pairs and so on, resulting in a so called avalanche breakdown.

In fig. 2.1 this effect is explained on the basis of the band-scheme of an avalanche diode.
Figure 2.1: Band-scheme of an avalanche diode [4]. The incident photon generates an electron-hole-pair. By impact ionisation these charge carriers are multiplied resulting in an avalanche breakdown.

2.2 Operating Principle of a SiPM

In silicon photomultipliers the avalanche effect is used for detection and amplification of incident photons. The photons generate free charge carriers in the photosensitive (= depleted) area of the cell [2]. The free charge carriers drift into the high field region, generated by an external applied voltage and by doping and the avalanche breakdown is triggered ((1) in fig.2.2b). The avalanche multiplication would continue infinitely because both, electrons and holes, cause impact ionisation. By an external circuitry the avalanche has to be stopped. This operating mode is called Geiger-mode [5]. During the avalanche breakdown the diode capacitance $C_D$ is discharged. The voltage applied to the diode is decreased below the breakdown voltage $V_b$ and the avalanche breakdown is stopped ((2) in fig.2.2b) until there are no free charge carriers in the high field region. The quenching resistor $R_C$ charges the coupling capacitance $C_C$ and again activates the high field region ((3) in fig.2.2b). In fig.2.2a a schematic electric circuit of an avalanche array is shown. The quenching resistor, the coupling and the diode capacitor define the recovery time of one cell by the formula:

$$\tau = (C_C + C_D) \cdot R_C$$

By contrast, in the proportional mode of an avalanche photo diode (APD) only one type of charge carriers is responsible for impact ionisation, the avalanche breakdown stops automatically and the generated charge is proportional to the number of charge carriers triggering the avalanche breakdown [6]. This operating mode is not feasible for avalanche diodes in SiPMs but in the avalanche amplifying readout of a pn-CCD [7].
Chapter 2: Silicon Photomultiplier

2.3 Properties of SiPMs

2.3.1 Photon Detection Efficiency

The photon detection efficiency is defined by [6]:

\[
PDE = \text{quantum efficiency} \cdot \text{fill factor} \cdot \text{avalanche efficiency}
\]  

(2.2)

Depending on the wavelength the quantum efficiency is up to 100% for BISiPMs. The geometrical fill factor dominates equation 2.2 because the efficiency of the avalanche diode can be tuned to 100% for an arriving photon. The fill factor is defined by the ratio of the light-sensitive surface to the whole SiPM surface. Structured surfaces e.g. conductors made of aluminium, cell boundaries and trenches are inactive areas. Therefore, the sensitive surface is reduced for front illuminated SiPMs, because the light hits the chip on the structured side. Back illuminated SiPMs have a fill factor of 100% because the structures are located on the front side of the chip and incident light is not reduced. Light is partially reflected by the surface of the entrance window or absorbed by the non-depleted layer [2]. By means of additional anti reflective coatings (ARC) the reflection can be reduced. The charge losses in the non-depleted layer are minimised by extracting the generated electrons towards the depleted bulk and by detecting them.

2.3.2 Dark Rate

Thermally generated charge carriers can also trigger the avalanche cell. These signals cannot be distinguished and are referred to as dark rate. The generation of charge carriers depends on the defect concentration, which is high at the surface and in doped...
volumes because of the implantation. The pure silicon bulk has a small defect concentration and therefore a low dark current.

At room temperature the dark rate is approximately $1 \text{MHz/mm}^2$ for FiSiPMs, dominated by the large doped area of the avalanche diode.

BiSiPMs have a large silicon volume dominating the dark rate, while the doped volume of the avalanche diode is only point-like.

The dark rate depends on the bias and the temperature. The rule of thumb is that every decrease of temperature by 7 K reduces the dark rate in Si by one-half [7].

2.3.3 Optical Crosstalk

The avalanche multiplication generates about $2.9 \cdot 10^{-5}$ photons per electron crossing the avalanche diode [5] (see Chapter 3). These photons emitted from one cell can trigger surrounding cells causing optical crosstalk. In order to reduce crosstalk in BiSiPMs the distance between the avalanche diode and the light sensitive volume of the neighbouring cell should be as large and the diode size as small as possible.

A small diode has a low capacitance $C_D$. Therefore less electrons cross the diode per avalanche breakdown, resulting in a lower photon emission. This is a main advantage of BiSiPMs, which have a spot like avalanche diode. But BiSiPMs have an increased probability to collect crosstalk photons because of their large sensitive volume. The crosstalk can be minimised by thinning the volume.

In FiSiPMs the avalanche diode has nearly the size of the light sensitive area resulting in a high photon generation. However in FiSiPMs the crosstalk can be reduced through optical barriers like trenches between the cells.

2.4. Back Illuminated Silicon Photomultiplier at the HLL

The MPI-HLL develops BiSiPMs for many applications e.g. the Major Atmospheric Gamma-ray Imaging Cerenkov Telescope “MAGIC”. The schematic cross-section of an avalanche BiSiPM is shown in fig.2.3. The incident photons pass the entrance window of the backsurface and generate electron-hole-pairs in the bulk [8]. By p+ rings, a drift field is created so that the carriers drift into the high field region and trigger the avalanche breakdown.

In laboratory prototypes the diameter of the high field region varies between 5µm, 10µm and 25µm.
Figure 2.3: Schematic cross-section of a BISiPM [5]: Incident photons pass the entrance window and generate electron-hole-pairs in the depleted silicon bulk. The electrons are drifted towards the high field region and trigger the avalanche breakdown.
Chapter 3

Theory of Photon Emission in Semiconductors

Photon emission is a process requiring energy. In principle this energy is set free in case an electron from a higher energy state drops into a lower energy state. The energy-difference of the beginning and the final state can be emitted in form of radiation completely or partially by participation of a phonon. The radiation rate $R$ depends on the density $n_u$ of carriers in the upper state, the density $n_l$ of empty lower states and the transition probability $P_{ul}$ of both energy states [9], [10] according to:

$$ R = n_u \cdot n_l \cdot P_{ul} \tag{3.1} $$

The Van Roosbroeck-Shockley relationship states that in equilibrium the optical generation of electron-hole-pairs is equal to their radiative recombination. Thus in equilibrium there is no light emitted. For more detailed information see “Optical Processes in Semiconductors”, Pankove [9].

The equilibrium can be disturbed by external excitation resulting in light emission called luminescence. Different types of the external excitation result in different types of luminescence e.g.:

- electric current (electroluminescence)
- optical excitation (photoluminescence)
- excitation through an electron-beam (cathodoluminescence)
- mechanical excitation (triboluminescence)
- thermal excitation (incandescence)

Depending on the time difference $t < 10^{-6}$ s or $t > 10^{-6}$ s [11] between excitation and emission one distinguishes between fluorescence or phosphorescence, respectively.

This chapter describes fundamental radiative transitions in semiconductors and some emission mechanisms in a silicon avalanche diode.
3.1 Fundamental Radiative Transitions

3.1.1 Transitions between Conduction Band and Valence Band (Interband Transitions)

If an electron drops from the conduction band into the valence band, the whole or a part of the energy-difference is emitted as electromagnetic radiation. One distinguishes between direct transitions and indirect transitions [12], [13]. Both transitions are discussed below.

3.1.1.1 Direct Transitions

In direct semiconductors e.g. GaAs the minimum of the conduction band is directly above the maximum of the valence band in the momentum space, so that an electron transition can occur without a change in the momentum \((k = 0)\) requiring a phonon. The band-scheme of a direct semiconductor with a direct transition at \(k > 0\) is shown in Fig. 3.1.

![Band-scheme of a direct semiconductor](image)

Figure 3.1: Band-scheme of a direct semiconductor. A direct band-transition at \(k > 0\) is shown. The energy difference between the upper and the lower energy level is emitted as a photon [9].

The emission-spectrum can be calculated by:

\[
L(\nu) = \frac{2q^2 (m_e^*)^{3/2}}{nch^2 m_e^*} (\hbar \nu - E_g)^{1/2} = B(\hbar \nu - E_g)^{1/2} \tag{3.2}
\]

- \(c\): Light velocity
- \(n\): Electron concentration
- \(m_e^*\): Reduced mass \((1/m_e^* = 1/m_e^* + 1/m_h^*)\)
- \(m_e^*\) and \(m_h^*\): Effective masses of electron and hole
- \(q\): Charge
- \(h\): Planck constant
From formula 3.2 one recognises that the lower limit of the energy of a direct transition corresponds to the energy of the band gap $E_g$. The emission-behaviour is shown in fig. 3.3.
By increasing the temperature the Fermi level smears resulting in filling states deeper in the band. Consequently the charge carriers become hot charge carriers because they have more energy than the band gap, which can be emitted as radiation by recombination.
Further causes for filling deeper states are:

- Increase of doping concentration resulting in a shift of the Fermi level [14].
- Intensity increase of the external excitation [9].

3.1.1.2 Indirect Transitions

In an indirect semiconductor e.g. Si and Ge the minimum of the conduction band is shifted against the maximum of the valence band by a certain momentum. Thus for a transition of an electron from the conduction band into the valence band a phonon with the energy $E_p$ has to be emitted or absorbed with the necessary momentum, in order to guarantee momentum conservation [14]. This mechanism is called indirect transition. A scheme for indirect transitions is illustrated in fig. 3.2.

Figure 3.2: Band-scheme of an indirect semiconductor. The dashed and the solid arrows show the transition by absorption and emission of a phonon, respectively [9].
According to [9] the transitions with absorption of phonons is less probable than with phonon emission, so the absorption is not taken into further considerations. The emission spectrum for indirect transitions is defined by formula:

$$L(\nu) = B'(h\nu - E_g + E_p)^2$$  \hspace{1cm} (3.3)

Thus the lower limit of the energy of an emitted photon is as follows:

$$h\nu = E_g - E_p$$  \hspace{1cm} (3.4)

As already described in Chapter 3.1.1, emissions with higher energies than $E_g$ are possible by recombination of hot carriers, but they are less probable. The emission spectra for direct and indirect transitions are shown in fig. 3.3. One clearly sees the parabolic increase for the indirect transition and square root behaviour for the direct transition.

![Figure 3.3: Emission spectrum for direct and indirect transitions [9]. The direct and the indirect spectrum show a square root and quadratically behaviour, respectively.](image)

3.1.2 Transitions between Donator/Acceptor Level and a Band

3.1.2.1 Shallow Transitions

In doped semiconductors transitions from conduction band into the donor level are possible as well as transitions from the acceptor level into the valence band. The valence band and acceptor level have an energy difference $E_i$ of some tens of meV similar to the conduction band and the donor level. At this transition photons are emitted with wavelengths in the infrared. For n- and p-doped semiconductors the band-scheme is illustrated in fig. 3.4. D and A mark the energy level of the donors and acceptors.
3.1.2.2 Deep Transitions

Transitions from conduction band to the acceptor level as well as from the donor level to the valence band are called deep transitions. In fig. 3.5 a band-scheme of deep transitions is shown. The emitted energy for direct transitions is given by the formula:

\[ h\nu = E_g - E_i \]  \hspace{1cm} (3.5)

And for indirect transitions because of the participating phonons \( E_p \):

\[ h\nu = E_g - E_i - E_p \]  \hspace{1cm} (3.6)
3.1.3 Intraband Transitions

At transitions within a band, one type of carrier is involved only. In the conduction band an electron can either drop directly or indirectly with participation of phonons from an excited state in a lower energy state. The released energy is emitted as light. In the valence band transitions between light and heavy holes can occur.

3.1.4 Exciton Recombination

A free electron and a free hole are attracting each other by the coulomb force because of their different polarity. The particles can form a quasi-particle called exciton analogous to the hydrogen atom. Both particles orbit around a common centre with discrete energies that are in the range of few meV according to:

\[ E_x = \frac{-m_r^* q^4}{2\hbar^2 e^2} \cdot \frac{1}{n^2} \]  

\[ \varepsilon : \text{Dielectric constant} \]
\[ n : \text{Quantum number} \]

The exciton states are located below the conduction band. If the electron and the hole recombine, the radiation energy will be smaller than the band-gap.

\[ h\nu = E_g - E_x \]  

(3.8)

Excitons have discrete energy levels. Consequently the emission of narrow spectral lines is expected, separated only by a few meV. The intensity of excited excitons decreases at higher orders [9].

At indirect transitions the photon emission occurs assisted by a phonon to guarantee the momentum conservation. The phonon emission also requires energy, therefore formula 3.8 must be reduced by the energy of the phonon at indirect transitions:

\[ h\nu = E_g - E_x - E_p \]  

(3.9)

The recombination for a direct and an indirect transition are schematically shown in fig. 3.6.
Chapter 3: Theory of Photon Emission

Figure 3.6: Recombination of excitons: (a) The direct transition. (b) For the indirect transition a phonon is required [9].

However the emission of one or more phonons is possible also at direct transitions, as long as there is no change of momentum. This condition is fulfilled at $k = 0$ for optical phonons. This type of photon emission is unlikely but possible. The band-scheme for phonon-assisted transitions is shown in fig. 3.7. Formula 3.9 has to be modified by an integer multiple of the phonon energy.

$$h\nu = E_x - E_{ed} - mE_p$$  \hspace{1cm} (3.10)

Figure 3.7: Band-scheme for the direct recombination of excitons by means of one phonon (a) or several phonons (b). The energy of the photon is reduced by an integer multiple of the phonon energy [9].

In order to recognise different energies of excitons, a spectral resolution is required at least in the meV range.
3.2 Photon Emission from Avalanche Breakdown in Silicon Diodes

The emission of light through an avalanche breakdown was first described 1955 by Newman et. al. [15]. In the following two theories of photon emission from avalanche breakdown in silicon are presented.

According to the first theory [16], [17] the emission of light in an avalanche breakdown is caused by three different mechanisms. The emission below 2 eV is stimulated by indirect interband transitions, from 2 eV to 2.3 eV bremsstrahlung is generated and from 2.3 eV direct interband transitions dominate.

In fig. 3.8 a typical emission spectrum of an avalanche breakdown is shown. As one can conclude from the measured spectrum, emission can be expected within an energy range between 1 eV and 3.5 eV and a wavelength range of 350 nm-1100 nm respectively.

![Figure 3.8: Experimental and theoretical emission spectra generated by an avalanche breakdown in silicon. A multimechanism model for photon generation is assumed [17].](image)

According to the second theory [18] the light emission can be described by an indirect interband recombination model. This theory considers the self absorption by silicon, which exponentially reduces the emission rate.

\[
R_{s}(\hbar\omega) = R(\hbar\omega) \exp(-\alpha(\hbar\omega)x_j)
\]  

(3.11)

\(\alpha\): Absorption coefficient depending on wavelength  
\(x_j\): Depth of the junction  
\(h\): Planck constant  
\(\omega\): Photon frequency  
\(R(\hbar\omega)\): Photon emission rate at a certain electron temperature depends on the electric field.

The emission rate depends on the junction depth and electron temperature. A high electron temperature increases the intensity of light and shifts the intensity peak towards
higher energies (fig. 3.9a). The junction depth affects the intensity especially for photon energies higher than 2 eV. If the junction is located directly at the surface, there is no intensity reduction as shown in bold in fig. 3.9b.

![Figure 3.9: Simulated emission rate depending on the electron temperature and the junction depth (x_j = 0.8 µm) [18].](image)

By formula 3.11 the theory fits the experimental results in good agreement. A comparison of theory and measurements is shown in fig. 3.10.

![Figure 3.10: Comparison of the theoretical and the experimental light emission of a reverse biased pn-diode [18].](image)
Photon Emission Microscopy with the PHEMOS-1000

Photon emission microscopy is used in the semiconductor-industry for the localisation and the analysis of failures in integrated circuits (ICs). Weak light emissions from ICs are localised by an optical microscope. An image is generated at a CCD-detector, whose signals are evaluated by a computer. The microscope image is visualised and analysed. In fig. 4.1 a photon emission microscope is shown schematically. The photon emission microscope operates in two different modes:

- Like in normal light microscopy the sample is illuminated and the pattern image of the sample surface is visualised by reflected light.

- Without additional illumination the light emissions of the device under test (DUT) are localised in an emission image and analysed.

Superimposing emission image and pattern image of the sample, emissions are related to the emitting structures. The high magnifications of the lenses enable optical resolution in the micron range. Depending on the optics and the detector system, light with wavelengths from ultraviolet to infrared can be measured [10].

Figure 4.1: Schematic drawing of a photon emission microscope. [10]
4.1 The PHEMOS-1000

The photon emission microscope PHEMOS-1000 from Hamamatsu consists of a light microscope covered by a dark box (2). The dark box blocks light from external light sources. The system rack (3) houses the power supply. The water-cooling (1) cools the detector CCD in order to improve the performance. By means of the PC (4) the PHEMOS is controlled and the microscope images are acquired, visualised and analysed.

![Figure 4.2: Overview of the PHEMOS-1000 components: (1) water cooling, (2) dark box with the microscope inside, (3) system rack, (4) computer [19].](image)

4.1.1 The Microscope

The microscope and the inside of the dark box are black-coated to absorb any residual light within the box. Without this even a few scattered photons can influence the emission measurement and falsify the result, since the intensity of the light emissions from the sample can be extremely weak.

In the HLL setup lenses are available with four different magnifications. With the macro lens having the lowest magnification (0.8x) samples are tested for light emission. The 5x lens shows coarse location of the emissions that can be determined more precisely by the 20x lens. The local intensity distribution of the emission is visualised in detail by the 100x lens.

There are two ports for cameras. At the first port a CCD camera is mounted. The second port can be used for optional optical components and other cameras. The optics and the camera are mounted on an xyz-stage controlled by the PC to observe the region of interest within an IC.
The PHEMOS is equipped with a wafer prober. The wafer can be electrically contacted by a probe card or needle holders mounted on a fixed platform. In order to contact another chip, the chuck can be moved by an xy-stage, which has contact and a non-contact position in the z-direction.

Figure 4.3: (a) Optical microscope inside the dark box. (b) Schematic drawing of the microscope.

For analysing single ICs a driving board is screwed on an aluminium disc (fig. 4.4), which is fixed on the vacuum chuck.

Figure 4.4: Driving board mounted on a wafer dummy.
4.1.1.1 The Optics

Lenses from Mitutoyo are available with magnifications of 0.8x, 5x, 20x and 100x. The lenses are selected by the lens revolver controlled by the control panel or by the computer. In table 4.1 the main features of the lenses are shown.

<table>
<thead>
<tr>
<th>Magnification</th>
<th>Working Distance (mm)</th>
<th>NA</th>
<th>View Size (mm²)</th>
<th>Spectral Transmittance Range (nm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.8x</td>
<td>24.7</td>
<td>0.40</td>
<td>16.25x16.25</td>
<td>600-1700</td>
</tr>
<tr>
<td>5x</td>
<td>37.5</td>
<td>0.14</td>
<td>2.6x2.6</td>
<td>480-1800</td>
</tr>
<tr>
<td>20x</td>
<td>20.0</td>
<td>0.42</td>
<td>0.65x0.65</td>
<td>480-1800</td>
</tr>
<tr>
<td>100x</td>
<td>12.0</td>
<td>0.50</td>
<td>0.13x0.13</td>
<td>480-1800</td>
</tr>
</tbody>
</table>

Table 4.1: Objective lens data according to the user manual of Hamamatsu.

The working distance is defined by the distance from the front side of the objective lens to the sample surface. The numerical aperture is defined as:

\[ NA = n \sin \alpha \]  

\( n \): Refractive index (air ~ 1.0)  
\( \alpha \): Half object opening angle

The aperture is the sine of the half object opening angle. Lenses with large aperture collect light from a large solid angle thus having a higher brightness. Consequently the 100x lens with the largest aperture has the highest brightness of the lenses. However one has to take into account that only the light from a small surface area is collected because of the high magnification as compared to the other lenses.

4.1.1.2 CCD-Detector, A/D Converter

Light is detected by a back illuminated monochrome CCD, cooled to -50°C. The chip has a size of 13.3 mm x 13.3 mm with 1024 x 1024 pixel, resulting in a pixel size of 13 µm x 13 µm. It is sensitive in the range between 200 nm and 1100 nm. A 12-bit A/D converter transforms the analogous detector signal in 4096 digital levels. Depending on the emission intensity of the sample the detector operates with the amplifications "low" and "high". At very weak light emissions the amplification is set to "high" while "low" is reasonable for strong emissions.
4.2 The Measuring Principle

The sample is fixed on the chuck and positioned below the microscope. During the operation of the PHEMOS the dark box is closed to suppress light from the environment.

The PHEMOS records two types of images: a pattern image and an emission image. Both images show the same region of the sample. By combining both images the so called “superimposed image” is obtained. Light emissions can be localised precisely on the basis of the superimposed-image.

For the pattern image the sample is illuminated with coaxial lighting.

For detecting emissions the lighting is turned off and the operating voltage of the sample is switched on. After an integrating time between 1 s and 9999 s the emission image of the sample is recorded.

Emission intensities are displayed with different colours. Red corresponds to a high intensity, while blue or black are representing very weak or no signal.

Figure 4.5: (a) Pattern image of an avalanche SiPM array with 20x lens. Aluminium reflects the light and appears bright. (b) Emission image of an avalanche SiPM array with 20x lens. Red spots mark emissions. (c) Superimposed image of an avalanche SiPM arrays with 20x lens. By this way emissions are exactly localised.
Signal Calibration

For the calibration of the system response the known light intensity of a source is measured at different wavelengths. Several bandpass interference filters are successively inserted into the optical pass and the resulting light intensity is measured. A calibrated light source is used as a sample to be imaged by the microscope. The light emitted by this calibrated source passes the optics and is detected by the CCD. Integrating the emission image and dividing it by the integrating time results in the number of digitized intensity units per second corresponding to the light power of the calibrated source. For the calibration of the system response with light this procedure is repeated for all bandpass filters in order to cover the whole wavelength range.

For a calibration of the detector response the number of electrons generating one digitized intensity unit per pixel is determined. For this purpose the detector is exposed to gamma- and X-rays from a $^{109}$Cd source. Depending on their energy the gamma- and the X-rays generate a certain number of electron hole pairs in the silicon CCD resulting in a related digitized intensity level.

In this chapter the calibration equipment and the calibration of the system response with light is described followed by the calibration of the detector response.

5.1 Calibration of the System Response with Light

5.1.1 The Equipment for the Calibration of the System Response with Light

The calibration of the system response with light is carried out for all different magnifications i.e. for all lenses. For this reason, a light source with a variable intensity is required to avoid saturation of the detector at different magnifications. A further reduction of the intensity can be performed by neutral density filters or by the adjustable mechanical attenuator built in the source. Since the source intensity is variable, the source has to be measured by a spectrometer for absolute irradiance measurements before calibrating the microscope. In order to collect all the emitted light of the calibrated source its size has to be smaller than the smallest image area (130 µm x 130 µm for the 100x objective lens) and the numerical aperture must be smaller than 0.14 to fit to all objective lenses. By means of fibers these conditions are fulfilled. The transmission of the bandpass interference filters is angle dependent and in the optical path of the microscope light hits the filters at different angles. Therefore the
transmission of the interference filters has to be measured with the filters build in the microscope. For this measurement an integrating sphere is necessary to integrate the light from different angles.

5.1.1.1 Spectrometer

The grating spectrometer HR-4000 including the 200 µm coupling fiber was calibrated by its manufacturer OCEAN Optics. The HR-4000 has a spectral range from 200 nm to 1050 nm for absolute irradiance measurements with a resolution up to 0.8 nm (FWHM) [20].

5.1.1.2 Light Source

Light is generated by the source HL-2000-FHSA-LL made by OCEAN OPTICS. It is a tungsten halogen source with a colour temperature of 2800 K [20]. The light is focused by a lens coupled into a fiber via an SMA 905 connector. Between source and lens a filter slot is located. The intensity can be adjusted from 0 to 100 % by a built-in mechanical attenuator. The manufacturer guarantees an intensity drift smaller than 0.3 % per hour. Fig. 5.2 shows a typical spectrum.

![Figure 5.2: The spectral intensity of the source is measured with the spectrometer. Above 900 nm the noise increases because of the reduced detector efficiency in this range.](image-url)
5.1.1.3 Neutral Density Filters

Neutral density filters from Laser Components GmbH consisting of metal-coated quartz glass are used to attenuate the light intensity within a large wavelength range. The transmission is characterised by the neutral density “ND” according to:

\[ T = 10^{-\text{ND}} \]  

(5.1)

In order to calibrate the source it is important to determine the neutral density of the filters in a spectral range between 400 nm and 1050 nm. For this purpose the HL-2000 source is connected to the spectrometer (fig. 5.3) and a reference spectrum \( R(\lambda) \) is recorded. The source intensity is reduced by the mechanical attenuator as long as the detector of the spectrometer is not saturated with an integrating time of 3.8 ms. Filters with a neutral density of 1 and 2 are inserted one after another into the filter slot of the source and the spectra \( S(\lambda) \) of the attenuated source are stored.

\[ T(\lambda) = \frac{S(\lambda)}{R(\lambda)} \]  

(5.2)

By this method we obtain the transmission depending of the wavelength for the filters ND1 and ND2. By formula 5.1 the spectral ND can be calculated and the filters ND1 and ND2 are calibrated.

![Figure 5.3: Schematic drawing of the setup for the determination of the neutral density. The ND filters are inserted in the built-in filter slot of the source. The source is coupled by a fiber to the spectrometer.](image)

The filters ND3, ND4 and ND5 strongly reduce the intensity, thus the dynamic range of the spectrometer is not sufficient. Instead the source power is maximised by opening the mechanical attenuator completely so that there is still a measurable signal \( S(\lambda) \) after inserting the filters ND3, ND4 or ND5. The reference spectrum \( R(\lambda) \) has to be taken using one of the calibrated filters ND1 or ND2, because the detector would be saturated in this setup without any filters. According to formula
and formula 5.1 the neutral density for the filters is obtained.

\[
T(\lambda) = \frac{S(\lambda) \cdot T_{\text{ND1}}(\lambda)}{R(\lambda)} \tag{5.3}
\]

Since light is reflected between the metal-coated filters and the source, more light is transmitted by the filters resulting in a lower ND. Moreover, the manufacturer specifies ±5% tolerance of the neutral density value.
5.1.1.4 The Fibers

Generally the light generated by the source HL-2000-FHSA-LL is fed into fibers. Depending on the required light intensity fibers with diameters of 50 µm, 100 µm and 400 µm are used. To send the light e.g. into the objective lens of the microscope a special chuck with SMA905 connector for the fibers was designed.

A fiber consists of a core with refractive index $n_1$ and a coating with refractive index $n_2$. Since $n_1 > n_2$ light is totally reflected up to a certain angle at the surface between core and coating. This is defined due to [21]:

$$\sin(90^\circ - \vartheta) = \frac{n_2}{n_1} \Rightarrow \cos \vartheta = \frac{n_2}{n_1} \quad (5.4)$$

The fiber of OCEAN OPTICS has a silica core and a doped fluorina silica coating resulting in an acceptance cone and field of view (FOV) of $2\vartheta_0=25^\circ$. The numerical aperture of the fibers results from formula [22]:

$$NA = n \sin \left( \frac{FOV}{2} \right) = 0.216 \quad (5.5)$$

To collect all the emitted light from the fibers the numerical aperture of the lenses has to be larger than the NA of the fibers. Thus only the 0.8x, 20x and 100x objective lenses can be used for calibration purposes.

![Diagram of a fiber](image)

Figure 5.5: Construction and principle of a fiber. Only light within the acceptance cone can be reflected totally.

The data sheets of the fibers are shown in the appendix [A.1].

5.1.1.5 The Integrating Sphere

The integrating sphere FOIS-1 of Ocean Optics Inc. integrates diverging or converging light. Via the 9.5 mm port light enters the integrating sphere. The inner surface of the sphere consists of the diffuse reflector spectralon. After several hundred reflections, the light is partially fed into a fiber connected to the sample port. It is a principle of the
integrating sphere to have the entrance port and sample port arranged towards each other in an angle of 90° in order to avoid a direct coupling. The integrating sphere is mounted at the second camera port. With a xyz-stage, the entrance port can be positioned exactly in the optical axis of the microscope. The light from the optical path of the microscope completely enters the integrating sphere at different angles and is detected by the spectrometer connected to the sample port. The integrating sphere has the disadvantage, that it attenuates the light intensity tremendously. Assuming for example that the light is reflected 400 times at a reflectivity of 99 % (spectralon), only 1.8 % of the original light power can be detected. This efficiency is reduced even more considering that most of the light leaves the integrating sphere through the 9.5 mm entrance port. Thus using the integrating sphere requires maximum power of the light source so that there is still enough light for the spectrometer.

5.1.1.6 The Interference Filters

The PHEMOS is not able to distinguish between different wavelengths because of the monochrome CCD-camera. Thus bandpass filters are inserted into the optical path between the microscope and CCD-camera. To measure a complete spectrum of an unknown source, different bandpass filters are inserted one after the other sampling the spectrum. The intensity measured in a certain bandpass is influenced by the width of the bandpass, the detector-efficiency and the transmission of the optics. This is compensated by the calibration of the system response. The interference filters from Edmund Optics Inc. are bandpass filters characterised by the parameters Central Wave Length (CWL) and Full Width Half Maximum (FWHM) (fig. 5.6) The Full Width Half Maximum corresponds to the width of the transmission curve, where the transmission is the same and higher than the half peak transmittance according to:

\[
FWHM = \left| \lambda_{(0,5,\text{peak} / +)} - \lambda_{(0,5,\text{peak} / -)} \right| \tag{5.6}
\]

Definition:
\( \lambda_{(0,5,\text{peak} / +)} \): Wavelength, with a positive slope of the transmission curve and a transmission equal to the half peak transmittance
\( \lambda_{(0,5,\text{peak} / -)} \): Wavelength, with a negative slope of the transmission curve and a transmission equal to the half peak transmittance

The central wavelength is defined by the following formula:

\[
CWL = \frac{1}{2} \cdot (\lambda_{(0,5,\text{peak} / +)} + \lambda_{(0,5,\text{peak} / -)}) \tag{5.7}
\]
Interference filters consist of a series of thin layers with different refraction-index. Within the bandpass incident light interferes constructively and is transmitted. At the remaining wavelengths the light interferes destructively and is annihilated. It is obvious that the interference depends on the angle of incident light to the filter surface. Because of the microscope optics the light hits the filters at different angles resulting in a shift and a broadening of the bandpass. Therefore and in order to guaranty a high precision of the calibration the filters were characterised in the microscope. To calibrate the filters in the microscope, the schematic setup shown in fig. 5.7 was used.

Figure 5.7: Setup for measuring the interference filter characteristics. The filters are inserted into the optical path between microscope and integrating sphere, which is coupled to the spectrometer.
The light of the HL-2000 source is sent into the microscope. After passing the integrating sphere the spectrometer detects the light. Dividing the light intensity $I_{\text{with filter}}(\lambda)$ measured with bandpass filter by the intensity $I_{\text{without filter}}(\lambda)$ without filter results in the filter transmission:

$$T(\lambda) = \frac{I_{\text{with filter}}(\lambda)}{I_{\text{without filter}}(\lambda)}$$

Fig. 5.8 shows the transmission curves of the interference filters. Each filter is represented by another colour. A strong noise signal can be seen under 480 nm and over 950 nm. The reason is the decreasing efficiency of the spectrometer. The FHWM of the interference-filters with a CWL of 457 nm and 950 nm can therefore be hardly determined.

Figure 5.8: Filter transmission curves being noisy at the edges of the measured band because of the low efficiency of the spectrometer in this range.

The FHWM and the CWL were calculated by formula 5.6 and 5.7. Table 5.1 shows a comparison of the filter data specified by Edmund Optics and the filter data measured in the microscope. All measured data are within the tolerance of Edmund Optics. Because the spectrometer is limited to wavelengths shorter than 1050 nm, no filter data for the filter with a CWL of 1064 nm could be measured.
Chapter 5: Signal Calibration

5.1.2 Implementing the Calibration of the System Response with Light

A calibration can be accomplished with the 0.8x macro lens, 20x lens and the 100x lens only, because the aperture of the lenses is larger than the numerical aperture of the fiber as described in chapter 5.1. The 0.8x lens can not resolve structures of the avalanche diode, thus for practical reasons it will not be used for spectral measurements and will not be calibrated. For the calibration of the 100x lens only fibers of 50 µm and 100 µm in diameter can be used (compare with table 4.1) because the fiber diameters must be smaller than the width of the image area. At lower magnifications the view size is always larger than the largest available fiber diameter, so fibers with a diameter of 50 µm, 100 µm and 400 µm can be used.

<table>
<thead>
<tr>
<th>Filter data from Edmund Optics</th>
<th>Measured filter data</th>
</tr>
</thead>
<tbody>
<tr>
<td>CWL</td>
<td>FHWM</td>
</tr>
<tr>
<td>450 nm ± 15 nm</td>
<td>80 nm ± 25 nm</td>
</tr>
<tr>
<td>500 nm ± 15 nm</td>
<td>80 nm ± 25 nm</td>
</tr>
<tr>
<td>550 nm ± 15 nm</td>
<td>80 nm ± 25 nm</td>
</tr>
<tr>
<td>600 nm ± 15 nm</td>
<td>80 nm ± 25 nm</td>
</tr>
<tr>
<td>650 nm ± 15 nm</td>
<td>80 nm ± 25 nm</td>
</tr>
<tr>
<td>676 nm ± 2 nm</td>
<td>10 nm ± 2 nm</td>
</tr>
<tr>
<td>700 nm ± 15 nm</td>
<td>80 nm ± 25 nm</td>
</tr>
<tr>
<td>766 nm ± 2 nm</td>
<td>10 nm ± 2 nm</td>
</tr>
<tr>
<td>800 nm ± 2 nm</td>
<td>10 nm ± 2 nm</td>
</tr>
<tr>
<td>830 nm ± 2 nm</td>
<td>10 nm ± 2 nm</td>
</tr>
<tr>
<td>880 nm ± 10 nm</td>
<td>50 nm ± 15 nm</td>
</tr>
<tr>
<td>950 nm ± 10 nm</td>
<td>50 nm ± 15 nm</td>
</tr>
<tr>
<td>1064 nm ± 2 nm</td>
<td>10 nm ± 2 nm</td>
</tr>
</tbody>
</table>

Table 5.1: Filter data of Edmund Optics and measured filter data
For the calibration of the system response the light source should only have a very weak intensity to avoid saturation of the CCD of the PHEMOS camera. The source intensity is attenuated by the neutral density filters and in finer steps by the adjustable mechanical attenuator. The photosensitivity of the spectrometer is too low in order to measure this reduced light intensity. Therefore the spectral intensity $I$ of the source is measured by the spectrometer without any neutral density filter. As in chapter 5.1.3 already described the neutral density filters are calibrated. The spectral ND as well as the transmission $T_{\text{filter}}$ is known for each filter. The intensity of the source after reduction $I'$ through the neutral density filters can be calculated by the following formula:

$$I'(\lambda) = I(\lambda) \cdot T_{\text{filter}} = I(\lambda) \cdot 10^{-ND(\lambda)} \left[ \frac{\mu W}{nm \cdot cm^2} \right]$$

(5.9)

The intensity is further reduced by the transmission $T_{\text{fiber}}$ of the fiber that connects the source with a special designed chuck via a SMA905 connector.

$$I^*(\lambda) = I'(\lambda) \cdot T_{\text{fiber}} (\lambda)$$

(5.10)

The emitting area $A$ of the fiber is assumed as ideally circular and is calculated on the basis of the known fiber diameters $d$ (see chapter 5.1.4). Multiplying the emitting area of the fiber with the intensity after the filter results in the spectral light power $P_{af}(\lambda)$ at the exit of the fiber:

$$P(\lambda) = I^*(\lambda) \cdot A = I(\lambda) \cdot 10^{-ND(\lambda)} \cdot T_{\text{fiber}} (\lambda) \cdot A \left[ \frac{\mu W}{nm} \right]$$

(5.12)
We are defining the light power at the end of the fiber as the calibrated source power.

The interference filters are inserted into the optical path of the microscope. For each filter the related emission image of the source is stored. The digitized intensity units \( n_i \) of all pixels are integrated over the entire emission image according to:

\[
N = \sum n_i \quad (5.13)
\]

The sum \( N \) of digitized intensity units is divided by the integration time of the image thus getting the digitized intensity units per second at the CWL of the filter:

\[
N_i(CWL) = \frac{N(CWL)}{t} \quad (5.14)
\]

The source power and the digitized intensity units per second are correlated by the spectral calibration factor \( K \).

\[
P(CWL) = K(CWL) \cdot N_i(CWL) \quad (5.15)
\]

The calibration factor depends on the efficiency of the optics, the filters, the detector, the amplification and the FHWM of the filters.

Fig. 5.10 shows the pattern image and the superimposed image of a 400 µm fiber with the 20x lens. In the pattern image the core and the coatings of the fiber can be seen. The superimposed image shows the emission using 700 nm interference filter with the integrating time of 20 s. A high emission (red) can be seen at the edge of the core decreasing to the centre (blue).

![Pattern image and superimposed image of a 400 µm fiber](image)

Figure 5.10: Pattern image and superimposed image of a 400 µm fiber. For a better visibility the core is marked yellow (a). The emission intensity decreases from the edge to the centre (b).
The integrated intensity from fig. 5.10b of $2.76 \cdot 10^7$ digitized intensity units/s corresponds to a source power of $1.25 \cdot 10^{-7}$ $\mu$W/nm. According to formula 5.15 this results in a calibration factor of $4.53 \cdot 10^{-15}$ $\mu$Ws/nm/digitized intensity unit.

In fig. 5.11 the calibration data of the system response is shown for the amplification low and for the 20x lens. (For further calibration data see appendix [A.2].)

![Figure 5.11: Calibration data for 20x lens at low amplification. The data are shown in table 5.2.](image)

<table>
<thead>
<tr>
<th>CWL [nm]</th>
<th>Source Power [(\mu\text{W/nm})]</th>
<th>Integrated Intensity Units/s</th>
<th>Cal. Factor [(\mu\text{W/nm/integrated intensity unit})]</th>
</tr>
</thead>
<tbody>
<tr>
<td>450</td>
<td>7.64274e-09</td>
<td>478620</td>
<td>1.59683e-14</td>
</tr>
<tr>
<td>500</td>
<td>1.73351e-08</td>
<td>2.41706e+06</td>
<td>7.17196e-15</td>
</tr>
<tr>
<td>550</td>
<td>3.17036e-08</td>
<td>5.16648e+06</td>
<td>6.13641e-15</td>
</tr>
<tr>
<td>600</td>
<td>5.95470e-08</td>
<td>1.72650e+07</td>
<td>3.44901e-15</td>
</tr>
<tr>
<td>650</td>
<td>8.54634e-08</td>
<td>1.39659e+07</td>
<td>6.11943e-15</td>
</tr>
<tr>
<td>676</td>
<td>1.09276e-07</td>
<td>2.36345e+06</td>
<td>4.62359e-14</td>
</tr>
<tr>
<td>700</td>
<td>1.25118e-07</td>
<td>2.76157e+07</td>
<td>4.53069e-15</td>
</tr>
<tr>
<td>766</td>
<td>2.19151e-07</td>
<td>5.62697e+06</td>
<td>3.89465e-14</td>
</tr>
<tr>
<td>800</td>
<td>2.79132e-07</td>
<td>5.24239e+06</td>
<td>5.32452e-14</td>
</tr>
<tr>
<td>830</td>
<td>3.30972e-07</td>
<td>5.68739e+06</td>
<td>5.81941e-14</td>
</tr>
<tr>
<td>880</td>
<td>4.16526e-07</td>
<td>2.14181e+07</td>
<td>1.94474e-14</td>
</tr>
<tr>
<td>950</td>
<td>5.67762e-07</td>
<td>1.15289e+07</td>
<td>4.92468e-14</td>
</tr>
<tr>
<td>1064</td>
<td>6.73116e-07</td>
<td>3.57544</td>
<td>1.88261e-12</td>
</tr>
</tbody>
</table>

Table 5.2: Calibration data of the system response is shown for the amplification low and for the 20x lens.
The black “x” in fig. 5.11 shows the integrated intensity units. The intensity units are strongly influenced by the FHWM of the filters. Narrow bandpass filters pass less light resulting in less digitized intensity units compared to the broader ones. The filter properties were already presented in table 5.1.

The red curve marks the spectral power of the calibration source. The characteristic increase of the source power at higher wavelengths is obvious. The data were extrapolated up to 1064 nm in order to calibrate the microscope with the 1064 nm filter, too.

By formula 5.15 the calibration factor $K(CWL)$ was calculated for each filter taking into account the power (red curve) and the integrated digitized intensities (“x”). The calibration factor is shown by the blue “+”. Between 950 nm and 1064 nm, the calibration factor rises strongly because the FHWM is approximately three times smaller for the 1064 nm filter than for the 950 nm filter and the efficiency of the CCD is already very low in this wavelength range.

The green curve shows the spectral power that results by multiplication of the calibration factors with the integrated digitized intensity units/s for each filter. It has to be clearly recognised that there are no bigger deviations from the red curve showing good quality.

### 5.2 Calibration of the Detector Response

In order to verify the reliability of the calibration of the system response the conversion factors of the PHEMOS detector are determined. For this purpose the detector is exposed to gamma- and X-ray photons from a $^{109}$Cd source. The emission-energies of cadmium and their percental distribution are shown in table 4.2 [24]. The half life of $^{109}$Cd is 462.6 days and the specific activity is 95,534 GBq/g [25].

Depending on their energy the gamma- and X-rays generate a certain number of electron hole pairs in the silicon CCD by the photo effect according to formula 5.16 [26]. The expected electron numbers for an impact are shown in table 5.3.

$$N_e = \frac{E_v}{3.67 eV}$$

The generated electron cloud spreads over several pixels of the CCD. For analysis and good statistics 150 pictures at 2 seconds integrating time were recorded for each amplification "low" and "high". A software programme per Interactive Data Language (IDL) summarises the counts of the pixels of one event and shows the histogram of the impacts. The histograms are prepared in fig. 5.12 and 5.13.

<table>
<thead>
<tr>
<th>X-ray* and gamma energy [keV]</th>
<th>Relative rate [%]</th>
<th>$N_e$</th>
</tr>
</thead>
<tbody>
<tr>
<td>21.9*</td>
<td>28.9</td>
<td>~5,967</td>
</tr>
<tr>
<td>22.1*</td>
<td>54.5</td>
<td>~6,022</td>
</tr>
<tr>
<td>24.9*</td>
<td>13.7</td>
<td>~6,785</td>
</tr>
<tr>
<td>88.0</td>
<td>3.6</td>
<td>~23,978</td>
</tr>
</tbody>
</table>

Table 5.3: Gamma- and X-ray energies of $^{109}$Cd.
In every histogram two peaks can be recognised. The peak with less counts is related with the two energy values 21.9 keV and 22.1 keV because their low energy difference can not be resolved. The second peak in the histogram corresponds to 24.9 keV. The cross section for gamma quanta with energy of 88 keV is too small to be detected by this detector.

![Figure 5.12](image1.png)  
(a)  
(b)

Figure 5.12: (a) Histogram at low amplification (b) with extended x-axis. The histograms were prepared with IDL.

![Figure 5.13](image2.png)  
(a)  
(b)

Figure 5.13: (a) Histogram at high amplification (b) with extended x-axis.

According to table 5.3 the emission lines at 21.9 keV and 22.1 keV have together a probability of 83.4%. The emission line 24.9 keV has a probability of 13.7%. For further considerations the emissions at energies 21.9 keV and 22.1 keV are referred to as peak 1, which has a middle energy of 22 keV and the emission at energy 24.9 keV as peak 2. The relative probability ratio between peak 1 and peak 2 is 85.89% to 14.11%. Considering the integral “int” of the peaks in the histogram 5.12b and 5.13b, as a result from following formulas,
a ratio \( W_{\text{peak}1} = 68.5\% \) to \( W_{\text{peak}2} = 31.5\% \) for low amplification and \( W_{\text{peak}1} = 72.7\% \) to \( W_{\text{peak}2} = 27.3\% \) for high amplification is obtained.

These values have to be corrected by the transmission of the detector front glass (thickness 7.15 mm) and the quantum efficiency of the detector. The quantum efficiency in this energy range is not known and therefore not taken into account for further considerations. The transmission of the front glass is calculated by the calculator [27] and is plotted in fig. 5.14. The transmission for 22 keV is \( T_1 = 0.052 \) and for 24.9 keV to \( T_2 = 0.123 \). Therefore the transmission of peak 2 is about a factor 2.37 higher than for peak 1.

![Figure 5.14: Transmission of glass in the energy range between 21 keV and 25 keV [27]. The CCD detector has a front glass with 7.15 mm thickness absorbing most of the intensity.](image)

Weighting the integrals with the transmission one obtains by formula 5.19 and 5.20 a ratio of probabilities for the low and high amplification listed in table 5.4. The measured data fit the specified values in a good agreement, so consequently the supposed energy for each peak is confirmed.
\[
W_{\text{peak1}} = \frac{\text{int}(\text{peak1})/T_1}{\text{int}(\text{peak1})/T_1 + \text{int}(\text{peak2})/T_2} \cdot 100 \quad [\%] \quad (5.19)
\]

\[
W_{\text{peak2}} = \frac{\text{int}(\text{peak2})/T_2}{\text{int}(\text{peak1})/T_1 + \text{int}(\text{peak2})/T_2} \cdot 100 \quad [\%] \quad (5.20)
\]

<table>
<thead>
<tr>
<th></th>
<th>(W_{\text{peak1}})</th>
<th>(W_{\text{peak2}})</th>
</tr>
</thead>
<tbody>
<tr>
<td>highA</td>
<td>86.3%</td>
<td>13.7%</td>
</tr>
<tr>
<td>lowA</td>
<td>83.7%</td>
<td>16.3%</td>
</tr>
<tr>
<td>theoretical value</td>
<td>85.89%</td>
<td>14.11%</td>
</tr>
</tbody>
</table>

Table 5.4: Probabilities of different peaks and amplification settings.

X-ray photons with approximately 22 keV generate about 6,000 electrons in the CCD, which correspond to 1,022 digitized intensity units at low amplification and 4,110 at high amplification. One electron in the CCD e.g. generated by light causes a rise in digitized intensity units of about 0.17 for low amplification and 0.68 for high amplification.

X-ray photons with 24.9 keV cause 6,785 electrons corresponding to 1,159 digitized intensity units at low amplification and 4,654 at high amplification. Therefore one electron causes a rise of about 0.17 digitized intensity units for low amplification and 0.68 for high amplification.

<table>
<thead>
<tr>
<th></th>
<th>conversion factor of the CCD detector</th>
</tr>
</thead>
<tbody>
<tr>
<td>low amplification</td>
<td>0.17 digitized intensity units /e\textsuperscript{-}</td>
</tr>
<tr>
<td>high amplification</td>
<td>0.68 digitized intensity units /e\textsuperscript{-}</td>
</tr>
</tbody>
</table>

Table 5.5: Conversion factors of the CCD detector system at the amplifications low and high.

For peak 1 and peak 2 the rise of digitised intensity units per generated electron-hole-pair is still the same for each amplification which again confirms the energy determination. Finally the amplification is 4 times higher for high amplification than for low amplification.
Chapter 6

Measurements

In this chapter measurements of two avalanche arrays with high field regions of 10 µm and 25 µm in diameter are described. The emission of the entire array and individual cells is analysed. The following aspects are tested:

- Emission homogeneity of the array and individual cells
- Analysis of structural failures
- Voltage dependency of the array and cell emission
- Spectral behaviour of individual cells

The samples are test chips of avalanche diodes arrays. In fig. 6.1 a schematic cross section of an avalanche diode is shown. The high field region (HFR) is located at the region with the shortest distance between the p-doped Si (blue) and the n-doped Si (yellow).

Figure 6.1: Schematic cross section of an avalanche diode. Different doping concentrations are shown in different colours. An avalanche breakdown is limited to the high field region (HFR).
6.1 Avalanche Diode Array with a High Field Region of 10 µm Diameter

This test chip F10 consists of an array with 20x25 avalanche diodes with a high field region of 10 µm diameter. In the further description the array is named "10 µm array". The breakdown voltage of the avalanche cells is about 38 V. This voltage defines the zero point of the overbias.

6.1.1 Analysis of the 10 µm Array

For analysis of the array a superimposed image is recorded with the 0.8x lens (fig. 6.2). The integrating time of the emission image is 60 s at high amplification. An operating voltage of 50 V is applied corresponding to an overbias of 12 V.

Figure 6.2: Superimposed image of a 10 µm array (Settings: 0.8x lens, $V_{overbias} = 12$ V, $t = 60$ s, high amplification). A coordinate system is inserted to improve visibility of the rows and columns of the array. Strongly emitting cells are e.g. in the 3rd and 9th row of the first column.
In fig. 6.2 the strongly reflective gold pads of the chip holder and the bond wires running from the lower image side to the chip can be observed. In the lower left hand corner of the chip a temperature diode is shown. The emissions are spread with a seemingly irregular intensity over the whole array. There are some remarkable bright emissions (red) in the first column of the 3rd and 9th row.

Figure 6.3: Superimposed image of a 10 µm array. The yellow marker shows the image area of fig. 6.7 (Settings: 5x lens, $V_{\text{overbias}} = 12$ V, $t = 60$ s, high amplification).

For further analysis a superimposed image (fig. 6.3) was generated with the 5x lens but with the same settings for the PHEMOS and for the sample like in fig. 6.2. The image shows the upper left corner of the array with 13 cells in x-direction and 11 cells in y-direction. A closer view shows that the emission is distributed over all cells. Especially the emissions of the 3rd and the 9th cell of the first column shine more brightly, as in fig. 6.2 already recognised.

For a more exact analysis of the homogeneity of the array emission, a new image visualisation technique for the PHEMOS was developed with the programming language IDL. The method is called “image mapping” (see appendix) and allows the precise comparison of different emission intensities.
Figure 6.4: Different emission intensities of cells in a 10µm array (Settings: 5x lens, $V_{overbias} = 12$ V, $t = 60$ s, high amplification).

Fig. 6.4 shows the same area as fig. 6.3. Strong cell emissions can be identified more clearly. The positions of the shining cells are listed in table 6.1. From this table a certain systematic can be recognised because the numerical values 1, 2, 9, 10, 11 and 12 in x-direction and 5, 8, 11 in y-direction occur more frequent by indicating a design error. A possible explanation is obtained with the 20x lens in fig. 6.4 showing cell [1,5]. (cell nomenclature: [columns, rows])

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Table 6.1: Positions of high emission cells.
A varying distance (marked in yellow) between two aluminium structures can be clearly recognised. All cells listed in table 6.1 were tested and the results are shown in the 3rd and 6th column. 13 of 18 cells show a low distance. Therefore the low distance is the dominant factor for an increased light emission.

Figure 6.5: Pattern image of a 10µm avalanche array taken with the 20x lens. A varying distance of the aluminium structures (marked in yellow) can be clearly recognised.

In fig. 6.6a the layout is plotted. The aluminium and the resistor (p-doped) is shown in blue (1) and red (2), respectively. The layers (3) and (4) show the high field region and the deep p-doping. The contact holes (5) contact the aluminium with the resistor. If an avalanche breakdown is triggered, the voltage and the high field decreases. Consequently the p-resistor has another potential than the p-resistor of the neighbouring cell, resulting in a leakage current (marked green) between the two quenching resistors if the distance is too short. Thus the quenching resistor is reduced by an additional resistor connected in parallel (fig. 6.6b) The recovery time is reduced, resulting in a higher dark rate of cell [1,5] and a strong emission.
Figure 6.6: (a) Layout of the cell with the strong emission. If the distance between the p-resistors (red) is too short and if the potential of both resistors differ, a leakage current (green) is generated between the resistors resulting in a strong light emission of the avalanche diode. (b) The equivalent circuit.

6.1.1.1 The Total Array Emission

The behaviour of the light emission depending on the applied voltage is of particular interest. Three factors are influenced by the operating voltage applied to avalanche diodes: dark rate, signal-height and light emission. An optimal compromise has to be found for the operating voltage.

The emission images of the complete array were taken with the 0.8x lens at an applied voltage from 42 V to 50 V. The further analysis of the images is done by a software package programmed in IDL. First, a region of interest (ROI) is defined covering the whole array. The total number of digitized intensity units/s is determined by adding the digitized intensity units/s of the pixels. It is shown in fig. 6.7 depending on the applied...
voltage. With increasing voltage an approximately exponential increase of the intensity can be observed. The light intensities at 42 V and at 50 V differ by about two orders of magnitude. Between 42 V and 48 V the difference is one magnitude only. Consequently the light emission of the array rises particularly from 48 V on.

Figure 6.7: Emission intensity vs. voltage of a 10 µm array. The emission intensity rises by increasing the voltage.

Comparing this graph with the current to voltage characteristics (fig. 6.8) of the array one recognises that up to 48 V the current also increases by one order of magnitude, but the rise by about one order of magnitude, as seen in the light emission between 48 V and 50 V, is missing. This measurement indicates that the light emission is nearly direct proportional to the current up to 10 V overbias.

Figure 6.8: Current to voltage characteristics of a 10 µm array.
6.1.2 Analysis of a 10µm Cell

Fig. 6.9a shows a superimposed image taken with the 20x lens (yellow marked in fig. 6.2). Using the image mapping method the different intensities are clearly visible in fig. 6.9b.

![Image](image.png)

Figure 6.9: (a) Superimposed image of a 10µm array (Settings: 20x lens, $V_{overbias} = 12$ V, $t=60$ s, high amplification). (b) The high emission intensity of cell [1,3] is clearly visible.

Three avalanche cells are selected for further detailed analysis.

- cell [1,3], clearly emits most light
- cell [2,3], emits nothing compared to cell [1,3] and doesn't have any recognisable layout failure
- cell [3,3], emits more than cell [2,3], but less than cell [1,3] and has no recognisable failure

The analysis of these individual cells is done with the 100x lens in order to be able to resolve the emission exactly.

6.1.2.1 Comparison of the Total Light Emission of Cell [1,3] vs. Cell [3,3]

According to fig. 6.9b there is a considerable difference between the emission intensities of the individual cells. For an exact analysis the intensities of cell [1,3] and cell [3,3] were measured as a function of the applied voltage. The resulting curves are shown in fig. 6.10.
Figure 6.10: Total emission intensity as a function of the applied voltage for cell [1,3] and cell [3,3] at high amplification. The emission intensity of cell [1,3] rises strongly between 46 V and 50 V.

The characteristics clearly demonstrates that both cells show about the same increase in the emission from 42 V to approximately 46 V, but cell [1,3] emits at least a factor two more than cell [3,3]. From 46 V to 50 V the intensity of cell [1,3] increases by more than three orders of magnitude. However cell [3,3] doesn't show this rise. Between the minimal intensity at 42 V and the maximum at 50 V there is an intensity difference of more than 5 orders of magnitude for cell [1,3] and more than one order of magnitude for cell [3,3].

In chapter 5 the conversion factors were calculated for both, high and low, amplification settings, so that for fig. 6.10 the number of photoelectrons related to the applied voltage can be estimated by

\[
N_{ph} = \frac{\text{"integrated digitized intensity levels / s"}}{\text{"conversion factor"}}
\]  

(6.1)

At a voltage of 50 V and at an intensity of 650,000 digitized intensity units/s of cell [1,3] and at the specified conversion factor of 0.68 digitized intensity units/e\textsuperscript{−} for high amplification 955,000 photons/s arrive on the detector.
6.1.2.2 Analysis of Cell [1,3]

In figure 6.11 the superimposed image of cell [1,3] in 100x magnification is shown. The image is taken at 50 V operating voltage with an integrating time of 3 s and low amplification in order to avoid saturation and to remain in the dynamic range of the CCD camera. The light emitting area has a diameter of about 10 µm corresponding to the designed size of the high field region.

![Superimposed image of the 10µm avalanche cell [1,3].](image)

The emission image of fig. 6.11 is presented three-dimensionally in fig. 6.11 [A.3]. The emission has a maximum of about 2200 digitized intensity units. One pixel in the image corresponds to 0.127 µm on the sample. Consequently the region of the purple coloured emission “needle” with size 25 x 25 pixels can be calculated to have about 3.2 µm.
Since the light emission of this cell is very strong, the spectral power could be measured at the voltages 50 V and 48 V. The integration time of the weak signal generated using a filter with a CWL of 1064 nm (FWHM 10 nm) at 48 V and at high amplification is 750 s. Measuring a spectrum between 450 nm and 1064 nm at 48 V takes about one hour. According to fig. 6.10 the intensity of the emission decreases at 47 V by about one order of magnitude as compared to that at 48 V. Consequently the measuring time at 47 V would rise by about a factor of 10, i.e. up to 10 hours.

Fig. 6.13 shows the measured power vs. wavelength for both voltages. Below 700 nm the power rises steeply. At 50 V and at wavelengths of above 950 nm there is even a decrease of the power. Additionally the power of the emission vs. photon energy is shown in fig. 6.13c and in fig. 6.13d with logarithmic and with linear scaled y-axis respectively.

Knowing the light power the number of emitted photons/s can be calculated. Dividing the power by the photon energy the number of emitted photons/s depending on the wavelength is obtained:

$$N(\lambda) = \frac{P(\lambda)}{E(\lambda)} = \frac{P(\lambda) \cdot \lambda}{h \cdot c}$$

(6.4)

h: Planck’s constant

c: light velocity

This spectral photon flux is plotted in fig. 6.13b. At 50 V about 1,530,000 photons/s are emitted within the measured wavelength range. At 48 V there are already less than 24,000 photons/s. Increasing the voltage from 48 V to 50 V multiplies the light emission by about a factor 64.
In chapter 6.1.2.1, a rate of $955,000$ photons/s was calculated at 50 V for cell [1,3]. This result is in good agreement with the $1,530,000$ photons/s, which were measured according to the calibration of the system response with light. Please remember: the calibration of the system response with light provides the number of photons emitted by the DUT before entering the optical system. Thus the efficiencies of the optics and of the detector are responsible for the difference in the number of photons/s.

In the following the number of photons emitted into a solid angle of $360^\circ$ is estimated. Assuming the emission is punctual, then there is an emission in a spherical sector with surface $A_{SS}$ and the opening angle of $2\beta=60^\circ$ calculated from the NA of the 100x lens.

$$A_{SS} := r^2 \cdot \int_0^{2\pi} d\varphi \cdot \int_0^\beta sin \vartheta d\vartheta$$  \hspace{1cm} (6.5)
Dividing the surface of a sphere by $A_{ss}$ results in $\frac{4}{2 - \sqrt{3}} \approx 14.9$. Consequently more than 355,000 photons/s and 22,776,000 photons/s at 48 V and at 50 V are emitted into the whole solid angle, respectively. The avalanche breakdown occurs between the surface and 1$\mu$m depth, therefore for simplification the absorption in Si (fig. 6.14) is not taken into account.

In order to do a rough estimation of the number of photons generated per electron crossing the diode, it is supposed for simplification that the current of the whole array is dominated by the strongly emitting cells. In fig. 6.2 between 5 and 30 cells shine more brightly. Assuming that these cells emit about 20,000,000 photons/s and taking into account the current of 1.1 $\mu$A (fig. 6.8) at 50 V, minimal $1.5 \times 10^{-5}$ photons/e$^-$ and maximal $8.7 \times 10^{-5}$ photons/e$^-$ are generated.

Figure 6.14: Absorption coefficient of Si at 300 K (line) und 77 K (dashed line) [12].
6.1.2.3 Analysis of Cell [2,3]

As shown in fig. 6.9b, cell [2,3] emits nearly no light as compared to its neighbours. The exact analysis with the 100x lens (fig. 6.15a) confirms the weak light emission. Fig. 6.15 is recorded with an integrating time of 500 s, high amplification and at 50 V.

Figure 6.15: (a) Superimposed image and (b) 3D emission image of the 10µm avalanche cell [2,3] (Settings: 100x lens, $V_{\text{overbias}} = 12$ V, $t = 500$ s, high amplification).
In the 3D emission image (fig. 6.15b) the weak light emission from the high field region can be seen. The emission nearly disappears in the detector noise. Thus we can not make a clear statement regarding the geometry and size.

A more detailed look at fig. 6.15a shows weak light emissions at the left and right hand side of the image and on the horizontal line crossing the high field region. There are two possible reasons for these emissions:

- emitted light from the horizontal neighbouring cells
- scattered light in the optics

Assuming light of the next cell neighbours, it is remarkable that no light of the upper and lower neighbours is detected. Also from the left neighbour cell [1,3] more light is expected to be seen (compare fig. 6.9b), because this cell emits substantially more light than the right neighbour cell [3,3].

Probably it is scattered light due to reflections in the optics. This cause can not be excluded because it is possible that e.g. the light of the brightly shining cell [1,3] is detected. This argument is confirmed by the fact that there is also a signal located on the aluminium layer. Measuring a spectrum for cell [2,3] is impossible as the integrating time using one filter would exceed the maximum integration time of 9999 s of the PHEMOS.

6.1.2.4 Cell Analysis of Cell [3,3]

Cell [3,3] has no layout failure, but nevertheless emits more light in comparison to the identical cell [2,3]. Fig. 6.16a shows the superimposed image taken with the 100x lens at an integrating time of 400 s, high amplification and 50 V operating voltage. From the image the diameter of the emission is estimated to be approximately 5µm and this is confirmed by its size of 40 pixel x 40 pixel in the 3D emission image (fig. 6.16b). The emission maximum is about 150 digitized intensity units. There is no visible geometrical anomaly and no emissions outside of the high field region.

During long integrating times cosmics hit the CCD detector and can be seen in the superimposed image.
Figure 6.16: (a) Superimposed image and (b) 3D emission image of the 10µm avalanche cell [3,3] (Settings: 100x lens, $V_{\text{overbias}} = 12$ V, $t = 400$ s, high amplification). Some cosmics hitting the CCD detector during long integrating times are marked yellow.

6.2 Avalanche Diode Array with a High Field Region of 25µm Diameter

This chip named C11 is a test array with only 16 avalanche diodes having a high field region of 25 µm diameter. The breakdown voltage of this array is about 38 V.
6.2.1 Analysis of the 25µm Array

For the array analysis a superimposed image (fig. 6.17a) at 50 V and high amplification was recorded. The dark shadows in the picture are bond wires connecting the array. One bond wire runs above the second column partially covering the emission. All cells rather uniformly show an emission.

Figure 6.17: (a) Superimposed image of 25 µm array (Settings: 5x lens, $V_{\text{overbias}} = 12$ V, $t = 300$ s, high amplification). (b) The emission intensities are clearly visible.
The intensity of individual cells is compared in fig. 6.18b. There is no intensity deviation like in chip F10, as described in chapter 6.1.

6.2.1.2 The Total Array Emission

The total array emission is measured in 1 V steps from 42 V to 50 V and is plotted in fig. 6.18. In this voltage range the emission intensity of the entire array rises exactly by an order of magnitude. In this logarithmic plot the curve is nearly a straight line.

![Figure 6.18: Total emission depending on the applied voltage.](image1)

In fig. 6.19 the current to voltage characteristic is shown. The current increases about an order of magnitude from 42 V to 50 V and has a nearly linear behaviour in this logarithmic plot. Comparing the emission curve in fig. 6.18 with the IV characteristic of the array, the emission is roughly proportional to the current.

![Figure 6.19: current to voltage characteristic of a 25 µm array.](image2)
6.2.2 Analysis of a 25µm Cell

For a rough overview the superimposed image (fig. 6.20) is generated using the 20x lens. The image shows the columns 2, 3 and 4 of the array. The emission within the avalanche region shows an inhomogeneous crescent-shaped distribution. It is remarkable that the emission of the avalanche regions is always higher at the left hand side. For a detailed cell analysis, cell [3,3] was selected because it is not covered by a bond wire and it has a relatively high emission in the area of the left avalanche edge.

![Superimposed image of a 25µm array](image)

Figure 6.20: Superimposed image of a 25µm array (Settings: 20x lens, $V_{\text{overbias}} = 12$ V, $t = 120$ s, high amplification).

6.2.2.1 Analysis of Cell [3,3]

In order to resolve the distribution of the emission precisely, a superimposed image (fig. 6.21a) of cell [3,3] with 100x lens is generated. The intensity is essentially divided into a rather high emission in the upper and in the lower area of the cell while there is almost no emission on the right side.
Figure 6.21: (a) Superimposed image of the 25 µm cell [3,3] (Settings: 100x lens, $V_{\text{overbias}} = 12$ V, $t = 120$ s, high amplification). (b) 3D emission image.

The 3D emission image (fig. 6.21b) exactly shows the intensity of the emission. There are two sharp peaks which are caused by cosmics. These particles hit the detector of the microscope during the integrating time of 120 s and are not related to the emission of the cell.
The width of the emission is approximately 150 pixels corresponding to 20 µm. According to the colour table there are between 130 to 150 digitized intensity units in the maximum of the emission. The emission at the edge of the cell is remarkable but cannot be explained.

At this cell an absolute spectral graph of the emission cannot be measured because the intensity within the filters’ bandpass is not sufficient. However, the emitted photon flux can be estimated. The emission caused about 3300 digitized intensity units/s in the detector. Taking the conversion factor of 0.68 into account 4850 photons/s are hitting the detector.

6.3 Estimate of the Minimal Detectable Photon Rate

In fig. 6.15 the deviation \( \sigma \) of the noise floor corresponds to about ten photoelectrons. The signal has to be larger than \( 4 \cdot \sigma \) to be distinguished from noise. Taking into account an integrating time of 500 s the sensitivity is about 0.08 photoelectrons/pixel/s. Assuming the emitting region in fig. 6.15 has a size of about 400 pixel, at least 32 photoelectrons/s have to be generated in this region. Into the whole solid angle at least 760 photons/s have to be emitted by the avalanche process in order to measure a signal.
Chapter 7

Summary and Conclusions

The aim of this thesis was the development of calibration methods for a photon emission microscope to analyse position resolved light emissions of avalanche processes.

A new image processing, called “image mapping” mode, was developed with IDL in order to visualise different emission intensities of individual cells and to correlate them locally. Emissions were plotted three-dimensionally piling up on the superimposed image.

In order to measure the spectral distribution the calibration of the system response with light was accomplished. For this purpose the PHEMOS was equipped with bandpass interference filters inserted into the optical path and the power of a calibrated source was measured.

Moreover, the conversion factors of the PHEMOS CCD detector for high and low amplification were directly determined using X-rays from a radioactive source $^{109}$Cd.

By the PHEMOS the local distribution of light emissions was visualised in the avalanche diodes of a SiPM. It was found that avalanche diodes with 10µm high field region show an emission “needle” in the centre. On the contrary 25µm avalanche diodes emit light mainly from the edge.

By image mapping a design failure in the avalanche diode array could be localised. The quantitative spectral analysis of a defective avalanche diode showed a decreasing intensity in the energy range from 1.15 eV to 2.75 eV. An intensity maximum at about 2 eV as described by the literature was not measured. It is assumed that for our devices this maximum is shifted into the IR. With an optional InGaAs-camera it might be analysed [A.4]. Integrating the spectra shows that 1,530,000 photons/s from a defective cell were collected by the optics.

According to the conversion factors of the CCD detector 955,000 photons/s emitted from the above mentioned avalanche diode are detected. With these results the photon detection efficiency of the full optical chain was calculated to be about 62%.

The newly developed image processing and visualisation techniques, especially the ”image mapping” mode, are excellent tools to localise different emission intensities. The calibration of the system response and the calibration of the detector response perfectly extend the application of the PHEMOS to a detailed analysis of light emitted by avalanche diodes.
Appendix A
A.1 Transmission of the Fibers

A.1.1 Transmission of the 50µm Fiber
A.1.2 Transmission of the 100µm Fiber

---

**Fiber Transmission**

- **Relative Transmission**
- **Wavelength (nm)**

---

**Inspection Checklist**
- Polish: X
- Concurrency: X
- Cap Placement: X
- Labeling: X
- Color Coding: X
- Female Length: X

---

RoHS - Compliant
A.1.3 Transmission of the 400µm Fiber

**Fiber Assembly Report**

Part #: P400-2-VIS/NIR  
Data: February 15, 2007  
Assembly #: GF131077  
Connector 1 #: SMA-805  
Connector 2 #: SMA-805  
Sales Order #: STOCK  

*Ask about our custom line of Optical Probes and Assemblies.*

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**Fiber Transmission**

![Graph showing fiber transmission with wavelength (nm) on the x-axis and relative transmission on the y-axis.]

**Inspection Checklist**

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- Concentricity: X  
- Cap Placement: X  
- Labeling: X  
- Color Coding: X  
- Female length: X  

**RoHS - Compliant**

Inspected by: [Signature]

[Date: 3/3/03]
A.2 Calibration Data of the System Response with Light

A.2.1 Calibration Data for 20x Lens at Low Amplification

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A.2.2 Calibration Data for 20x Lens at High Amplification

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A.2.3 Calibration Data for 100x Lens at Low Amplification

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A.2.4 Calibration Data for 100x Lens at High Amplification

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A.3 Image Processing

A.3.1 Image Mapping

Pattern Image     Emission Image

Superimposed Image

3D Plot

Image Mapping
A.3.2 3D Emission Image
A.4 Different Camera Systems [19]
Literature


http://209.73.52.252/assets/pdf/catsandguides/mppc_kapd0002e02.pdf


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