

Bangor University

DOCTOR OF PHILOSOPHY

Combined dual wavelength interferometer and laser scattering monitor for multi-wafer MOCVD

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Award date: 2004

Awarding institution: Bangor University

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Combined Dual Wavelength Interferometer and

Laser Scattering Monitor for

Multi-Wafer MOCVD

A Thesis Submitted By

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To the University of Wales in the candidature for the degree of

Philosophiæ Doctor



Department of Chemistry University of Wales, Bangor September 2004



Abstract.

This project entailed the investigation of laser light reflection and scattering from thin film surfaces. To carry out this research, two testbeds, one *ex situ* and one *in situ*, were constructed to study two and three dimensional growth in II-VI semiconductor films. The instruments were tested by analysis of Metal Organic Chemical Vapour Deposition [MOCVD] growth of CdTe and/or CdS onto either ITO/glass or Si(001) substrates, using dimethylcadmium (DMCd), with the addition of diisopropyltellurium (DiPTe) and/or di-tert-butylsulphur (DtBS).

An Angle Resolved Scattering [ARS] monitor was used *ex situ*, and the ARS distributions recorded. These results were correlated against Atomic Force Microscopy image roughness analysis. The findings were verified with angle resolved scattering theory, and were found to be in good agreement.

A combined dual wavelength Reflectance Interferometer and Laser Light Profile [LLP] system was used to record 2D&3D growth data in real-time, *in situ*. Traditional single wavelength and the dual wavelength interferometer data were compared, and were found to agree well, although the fitted values for the extinction coefficient were consistently double the referenced value. In the LLP study presented here, the general behaviour of the new technique of laser light profiling was shown, and a model was presented. Again, the data were found to correspond well to theory. The recording of the LLP data has been possible by redesign of the combined instrument, where the detection electronics were changed to a CCD-array. This also resulted in a much simpler instrument, as the detection can be carried out in one single detector, by matching laser wavelengths with the pixel colour.

Throughout the dual wavelength measurements, a wavelength dependent interferometer response time has been observed during nucleation. The longer wavelength (655nm) has shown a lag in response, compared to the shorter wavelength (532nm).

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1 Introduction.

1.1 Project context.

There can be no doubt that the most important discovery that has happened in modern time is the invention of electricity, or perhaps rather the electrification of the world. There were naturally different sources of power even before the invention of the electrical generator and the National grid, like steam powered factories and acetylene lighting, but as soon as electricity became widely available, these sources were obsoleted. The economy, convenience, safety and the universality of the power source is, and will always be, without comparison.



Evolution from 1971 to 2000 of World Total Primary Energy Supply* by Fuel (Mtoe)

Fig 1World energy production, as divided into fuels.

Today electricity is, almost without exception, the power source in manufacturing, research, engineering and in all other aspects of daily life. The technology of power generation has not evolved much during the century or so we have had public access to it. There are several renewable sources, like hydro, wind, wave and others, but the technology involved is pretty much the same as in the Watt/Edison generator. - A large generator harnesses the momentum of a turbine, or some sort of a pendulum. Indeed, one of the chief energy sources, coal, is still the same as in the original generator, even if the basic design has changed with the W. Rankine steam turbine invention of 1859. Oil has nowadays overtaken coal as the number one source, with gas in third place. Those are the three chief sources of energy today, cumulating to about 80% of the world production¹, see figure 1, and probably will be for the foreseeable future. The burning of fossil fuels for power is of course far from being the ideal solution, even if the system of a National Grid as a supply channel is mostly adequate. The trend in renewable power sources is towards decentralisation however, with distributed small power sources. The advantage with this approach would be less interruption of service, as the power would be produced in vicinity of the consumer, and grid failures would generate only highly localised power cuts. The disadvantage is the loss of large scaled production effects, with the higher cost that follows. We also recognise there are quite a few applications that cannot be supplied by means of a national grid. Just to mention three important examples:

- Desolate settlements. There are just no practical way of connecting some settlements to a large scale grid. For, for instance, third world villages, or small populations on very remote places this can certainly be said to be true.
- The mobile workplace. A large movement for mobility-at-work and outdoors life has taken place in the last couple of years. The traditional way of using batteries for the appliances one use, or would like to use, in these environments are mostly inadequate. For example; the latest technology battery for use in a notebook costs over £100, and lasts for about two hours on one charge.
- Space applications. More and more satellites are sent up in an effort to keep up with the growing demand on information exchange. New in the sky is also ISS, the International Space Station, and an ever larger number of research

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platforms are sent up every year. All of them are in need of a constant and reliable supply of power.

During the last few decades large efforts have been put into solving these incompatibilities. Examples of approaches that have been taken are:

- The Fraunhofer-Institute are, in collaboration with Siemens, discussing
 replacing the battery of standard mobile electronics with micro fuel cells², thus
 enabling a five-fold increase in "battery" time, or a full days work in a single
 "charge". These cells can of course be reloaded in a couple seconds by simply
 refilling the cell with a suitable gas.
- Photovoltaic technology is nowadays used in everything from small solar cell powered pocket calculators, or GPS gadgets, to huge satellite power arrays. This is achieved by direct conversion of light to electricity in a purpose made diode³. Especially, and for obvious reasons, the space applications are totally dependent on this technology.

In some instances, there are already cheap technology on the market, but there will always be room for improvements and optimisation of yield, stability and cost. The Optoelectronic Materials Chemistry group, Bangor are presently researching CdS/CdTe type solar cells, grown onto cheap glass substrates^{4,5} coated with some transparent conductive oxide [TCO]. These are, in theory, promising materials in terms of high yield - low cost power cells⁶. As a part of this research, investigations of different aspects of crystal growth and *in situ* Metalo-Organic Chemical Vapour Deposition [MOCVD] growth control^{7,8} are also done, and it is within this framework the instrumentation project has been carried out.

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1.2 Project presentation.

To sum up the different activities, that have been carried out on the "Combined Interferometer and Laser Scattering Monitor for Multi-Wafer MOCVD" instrument development project: the project can be separated into two different aspects, one film growth and one surface roughness monitoring aspect, where the solutions to both problems have been combined into one simple instrument. The aim was to develop a next generation MOCVD growth monitor, to replace the currently used single wavelength, reflectance only, laser reflectance interferometer. This is because of limitations in the range of materials and growth mechanisms being monitorable using the current technology.

The development and growing of finer structures, higher yield devices and a general need for better control of doping levels and relative concentrations in the films with, sometimes, even new growth methods, has almost made the current monitoring technology obsolete. For example: the measured intensity in interferometry can be described as a sum of reflected, absorbed and scattered partial intensities [R, A, S]. Using the current reflectance interferometry, there is a problem of unambiguously determining which effect: scattering, absorption or interference, is related to which intensity contribution, making such an instrument potentially inaccurate.

A dual wavelength system was therefore researched. The scattered intensity in such a system will be inversely proportional to the fourth power of the wavelength, as described by Rayleigh⁹, while the absorption and interference patterns are explained in the reflectance equation of the virtual interface theory¹⁰. This returns a cosine function for the interference pattern combined with an exponential for the absorption, but this will be more thoroughly discussed in the

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theory chapter. The three functions for the [R, A, S] intensities are separable, thus making it possible to reliably decide the different contributions.

The ability to follow three dimensional growth of the surface was warranted by industry, as an important add-on to the growth monitor; recognising 3D surface properties will determine the interface quality of layered device structures. Certain materials also include manipulating roughness parameters as a step in the growing procedure^{11,12}. Real time roughness monitoring would therefore be of great interest. In doing this, angle resolved scattering from the normal incident lasers has been considered as a solution and investigated. Because of the limited optical access stipulated in the project proposal, $\sim\pm3^{\circ}$, this was later discarded as a method with possibilities only to *ex situ* analysis. The reflected laser beam images were also looked at, where the profile distribution can be used for investigating the surface roughness of the films, and this seems to be the simplest solution. Because of this, and because of the robustness, the well understood theory, and the simplicity of the two techniques the combination of interferometry and laser light scattering/profiling was chosen.

Two different instruments were built for the purpose of investigating this, firstly, *ex situ* - the angle resolved scattering [ARS] and, secondly, *in situ* – the combined interferometry and laser spot profile data [LLP]. The *in situ* instrument was later modified to the dual wavelength setup. The instruments will be presented and discussed further in the methodology chapter.

The text that follows will introduce some important concepts and the devices and materials used in this project. Especially the photovoltaic device will be presented, as this is the device most measurements have been performed upon. A discussion on the material chosen for the development of a thin film PV device will be held, and the MOCVD growth technique will be presented.

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1.1.1 The photovoltaic device.

Photovoltaic cells are all basically different kinds of diodes^{13,14}. The diode device structure is also the one that the Opto-electronics Materials Chemistry [OMC] group, University of Wales Bangor, grows and uses^{15,16,17}. The OMC devices presently consist of a Pilkington Space Technology manufactured Indium/Tin oxide [ITO] on glass substrate. On the ITO a CdS:Cl n-type buffer layer is grown, with a p-type, As doped, CdTe active layer on top. Metal contacts are evaporated onto the CdTe in order to complete the device and the device is tested with regards to electrical properties. The OMC device structure is shown in figure 2.



Fig 2 Common CdTe solar cell layering structure.

In an electronic sense the solar cell devices can function in three fundamentally different ways, with either a homojunction, heterojunction or Schottky junction. The three different approaches are presented below^{13,14,18}.

1.1.1.1 The homojunction diode.

A device made up from the same semiconductor on both sides of the junction is a homojunction type device, where the required energy differences in acceptor/donor bands are typically produced by n and/or p dopants. The majority of commercial solar cells are of this type, such as the common bulk silicon and GaAs solar cell are. The bulk technology has severe limitations because the electron hole pair is created near the n side surface, which makes recombination very likely throughout the bulk. Si minority carrier diffusion lengths are actually very long, but unfortunately the absorbtion is also weak, which means thick layers are needed.

The technique of layering homostructures of course reduces problems of lattice mismatch and film stress significantly. Also, the reduced problem of diffusion in these materials makes them stable over long time. The technology is old, well known, and therefore relatively cheap, which is why, in spite of the lower theoretical power conversion factors for these type devices, this can still compete with the theoretically more efficient heterojunction devices.

1.1.1.2 The p-n heterojunction diode.

The p-n, p-i-n, or heterojunction diode is the device which is normally formed as two semiconductors with different bandgap are connected. Common diode (i.e. solar cell) materials are CdTe/CdS, ITO/InP or GaInP/GaAs. The devices are typically grown by some thin film technique, which makes the use of more expensive materials possible, as well as producing films with high purity to minimise carrier recombination. Because of the thin and selective films, it is also possible to layer several acceptor films of different wavelength bandgaps, thus creating multijunction cells, or even grade the composition of a device layer. This would make the devices absorb over large parts of the spectrum, enhancing the efficiency of the cells. Without this multi-layer design, while trying to absorb the same range in one layer, device heating and breakdown will inevitably become a problem. Doping of these type cells is made to enhance carrier concentrations. Little is still known of the mechanisms of charge and

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mass transportation in junctions of polycrystalline thin film materials in general, which is why this kind of thin film solar cell has still to make an impact commercially. Single crystal devices are well understood, but are instead expensive to produce by these methods.

1.1.1.3 The Schottky barrier diode.

A third device structure is the Schottky or metal insulator Schottky [MIS] diode. The Schottky diode is, like the homojunction diode, a unipolar device. The major advantage of a Schottky device is that this, as opposed to the homo- and heterojunction device, is a majority carrier device. This makes the device insensitive with regards to minority carrier lifetime issues, which is the largest issue in degradation of the power conversion factor. The device structure is formed by the single semiconductor, in combination with the contact metal. The metal-semiconductor [MS] junction contains a Schottky barrier, produced by the mismatch between the MS valence bands. The photocurrent is produced by excitation of electrons in the metal to a higher potential than the barrier. A second photocurrent is produced in the normal way by the formation of electron/hole pairs in the semiconductor depletion zone. Like the heterojunction device, this is a device where much work still has to be done, and likewise, there are no commercial devices available.

Furthermore, there are a range of devices somewhat different to the semiconductor devices. After studying biological systems, Michael Grätzel invented the so called "artificial leaf"¹⁹. Based on this research, he later also constructed a new type of solar cells - the dye sensitised cells²⁰. They rely on a conductive glass/TCO substrate, whereupon a porous sintered acceptor bulk is

attached. The acceptor material is generally rutile, TiO₂, but variations are being researched. Several organic molecules has also been looked at for the purpose of acceptors. The porous bulk is submerged in a I₃ solution, and contacts are added to close the cell circuit. These cells are easy to make and cheap, with for instance screen printing as an option for manufacturing. The liquid phase makes it sensitive for temperature and pressure[TP] changes, and for instance space applications are because of this out of the question.

1.2.1 CdS/CdTe thin film solar cell materials.

Certainly, a huge amount of work has been put into choosing the right material for the photovoltaic devices mentioned above. By comparing the bandgap of different standard semiconductors to the theoretical efficiency maximum, a few alternatives have shown to be optimal.

CdTe is, as shown in figure 3, one of the best alternatives²¹. It also has a direct bandgap, which, for instance, Silicon does not have. This makes it even more attractive as a candidate. It has a 1.5 eV bandgap at Standard Temperature and Pressure [STP], with a theoretical conversion efficiency of 28% at 1.5AM²² (100mW·cm⁻²)*. The highest measured efficiency to date (achieved 2001 by Wu et al) is about 16.5%²³. Traditionally, the view has been that the CdTe/CdS device works on a p-n heterojunction basis¹³, but recent research by Dharmadasa, *et al.* is interestingly now contending this, and the authors suggests the Schottky diode model is more representative²⁴.

^{*} AirMass; The ratio of the mass of atmosphere in the actual observer-sun path to the mass that would exist if the observer was at sea level, at standard barometric pressure, and the sun was directly overhead. AM0 corresponds to the solar spectrum in outer space, and the reference spectrum for Standard Test Conditions[STC] was defined to be AM1.5



Fig 3 Some common semiconductors photovoltaic conversion efficiency. Ideal solar-cell efficiency at C=1 and C=1000 solar concentrations.

1.2.2 The MOCVD film growth technique.

Metalo-organic[OM] chemical vapour deposition [MOCVD] is a growth technique that recently has become more and more popular. The technique is based on flowing precursor, organo-metallic[OM], gases over a hot substrate in a controlled environment, leading the reactants directly to the heated reaction site, thus depositing a film on top of the substrate. The process is illustrated in figure 4. The technique has become popular because of several reasons, for example²⁵:

• The purity of layers grown in this kind of reactors, and this kind of pressure regime is excellent.



Heat

Fig 4 Principle of MOCVD mass transport.

- The reaction takes place at relatively low temperature, as the material is brought to the substrate in the gas phase, and need not to be melted.
- Very thin layers can be grown with high precision, which saves resources.
- Growth parameters can be controlled very accurately, which makes optimisation of properties achievable.

Drawbacks of the MOCVD technique are mainly:

- The cost of the chemically exotic precursors, as they are often manufactured in very small quantities. Fortunately, very small quantities are needed.
- Substrates are often single crystal wafers, which are quite expensive. A lot
 of efforts is going into researching alternative substrates, mainly made of
 glass or suitable polymers.

Three different growth modes are generally accepted as possible mechanisms for initial and continued growth of thin films, as discussed below²⁶.

1.2.2.1 Vollmer - Weber island growth mode.

Vollmer - Weber, or island mode, is a growth mode where nucleation occurs directly in the surface gas super saturation, with condensation into multiple islands. This is the result of thermodynamic preference to nucleate onto its own phase, instead of onto the substrate. Many systems, including metals on halides or insulators and graphite grow by this mode.

1.2.2.2 Frank - van der Merwe layer growth mode.

The opposite mode is the Frank – van der Merwe, a growth mode where the condensate of material from the surface adsorbed gases directly forms a mono-layer on the surface. Further layers are grown in the same way. This mode is usual in, for example, semiconductor growth on semiconductors.

1.2.2.3 Stranski - Krastanov layer and island growth mode.

Layer and island growth mode is the intermediary to the two first. It starts off as a layer growth mode, but as the first (few) monolayer(s) have condensed, the neighbouring thermodynamic environment has changed, in favour of islands. Not only can this be true, but as nucleation often occurs in defects and other disturbances on the surface, and the first few layers of the growth are most often highly strained, this makes an excellent place for changing mode. This mode frequently occurs in metal-metal and metalsemiconductor systems.

1.2.3 Contemporary MOCVD growth monitoring techniques.

Growth monitoring in MOCVD reactors is done generally at a pressure that disallows the use of electron beam techniques. The emphasis in this project has therefore been on developing techniques that instead depend on interaction of light with the growing sample. Basically, there are two different optical effects that are presently used: layer reflectance interference and anisotropic absorption/reflection. The first makes the base for the reflectance interferometry technique and the latter the ellipsometry technique. There are also several hybrid techniques, like reflectance anisotropy spectroscopy[RAS], reflectance difference spectroscopy[RDS] and surface photoabsorption[SPA], and the RAS technique will be presented as an example. Most of these hybrids have a limited use in special applications. As stated in the foreword, the development of finer structures, and an increased knowledge into processes in interfaces between film layers and layer/atmosphere. This has prompted an increase in research into controlling three dimensional surface growth, as this to a large extent decides the interface. Surface scattering and laser spot profiling will be discussed as a solution to monitor this parameter.

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2 Monitoring theory and relevant context.

In this chapter the different theories that have been used in the project will be discussed in greater detail . The emphasis will be on discussing *in situ* monitoring theory¹, which will be divided into the interferometry and ellipsometry theory. Furthermore the Reflectance Anisotropy Spectroscopy[RAS] technique will be discussed as an alternative (intermediate) technique, mainly because of the large current interest in its application in MOCVD. These techniques rely on two dimensional layer growth theory. When it comes to three dimensional surface growth, two different but related techniques will be presented: Laser light scattering in the form of angle resolved scattering and laser dot profiling. The text will show why, in the case of this project, a combination of dual wavelength interferometry combined with laser dot profiling was ultimately considered as the optimal solution. However, to start with, a brief overview of the constraints imposed by the reactor geometry stipulated in the project proposal.

2.1 The Aixtron 2600 reactor.

The Aixtron 2600 production reactor is a multi-wafer planetary rotation reactor with two different planetary systems; one 8x3" wafer and one 5x(3x2") wafer rotating satellites, as shown in figures 5a and 5b. The planetary system is monitored from above, using an existing top-plate pyrometer port, with crosssection measurements seen in figure 6. The planet susceptor is made out of graphite, which moves under the pyrometer port, thus alternating graphite and the compound material being grown at the time. Because of different reflectance of the different materials, a repeated pattern of sample/graphite reflected square waves will be created. This makes an excellent reference, both for the determination of uniform growth on the individual wafers², and for controlling

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drift in the system. The drift control is achieved by realising that the graphite reflectance will stay constant throughout a growth run, and therefore can be used as a stable (zero) point. Any variance in this signal can then be attributed to drift.





Fig 5a The Aixtron 8x3" planetary rotation system.

Fig 5b Five 3x2" planetary rotation system.



Fig 6 Geometry and refracting data of reaction chamber.

The pyrometer port contains a 6 mm diameter silica rod, 72 mm long, which is located directly on top of a 6 mm guartz plate some 64 mm over the planet susceptor. Simple geometry determine that an opening of about $\pm 3^{\circ}$ is available (refraction in plate and rod included in the calculations) for probing the surface. Because of parasitic growth on the top plate, it is very difficult to get an analytical signal. In fact, the low power lasers (5 & 10mW) that have been used in the instrument throughout the experiments, are too weak for achieving this. Weeks et al^3 , has studied the top plate deposits in detail, with the conclusion that only a high powered IR laser >50mW can give a reasonable reflection signal. Of course, with the approach of this project, IR lasers are not an option, as the CCD detector is not sensitive in that area. The reflection on the topside top-plate surface will, of course, also be significant with powerful lasers, and without taking precaution, this will saturate the detectors, or at least make the noise contribution significant enough to render the signal useless. Caution must therefore be taken to ensure that the top-plate primary reflection can be angled off the detector. In practice, this means that the instrument must be able to work over a few degrees off-normal, and therefore that the beam splitters (see chapter 3 on the instrument design) must be adjusted to the correct size. A more reasonable solution would be to change the design of the top plate to incorporate a dedicated gas-purged port. The Aixtron Reactor design is unlikely to be changed, though, as temperature and flow dynamics would change with a redesign, and the knowledge base of the reactors would have to be re-researched.

2.2 Two dimensional layer growth.

This section will focus on single and dual wavelength reflectance interferometry, Spectrometric Ellipsometry[SE] and the adaptation: Reflectance Anisotropy Spectroscopy [RAS].

2.2.1 Single wavelength reflectance interferometry.

Laser reflectance interferometry^{4,5,6} is based on the measurements of interference patterns created as coherent light reflects off different layers in a multilayered sample. The magnitude of interference depends on the incident intensity, the material absorption at the specific wavelength, and the path length of the light in the material. In reflectance interferometry the film thickness is thus measured as a function of time throughout a growth run, where the signal intensity can be broken down into its four components; reflected [R], absorbed [A], scattered [S] and transmitted [T] intensity;

$$I = I_R + I_A + I_S + I_T$$
 Equation 1)

From the refractive index and the complex refractive index the growth rate can be determined. This can be achieved by using the virtual interface model^{7,8}, or rather the single layer modification of the model developed by Irvine *et al*⁹. The multilayer interface equation is here reduced to a single layer situation. The model basically treats the complex multi-layer case as a single-layer, by treating all already deposited layers as a "virtual substrate". This is possible, as the optical variables are all constant within this "substrate". In effect, all interfaces within the substrate have therefore been collected in one "virtual interface", coinciding with the substrate surface. The next layer can then be described by a much simpler single layer equation. According to this virtual interface model, the reflectance can be written as¹⁰:

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$$R = \frac{r_1^2 + r_2^2 e^{-\alpha d} + 2r_1 r_2 \cos \Delta e^{-\alpha d}}{1 + r_1^2 r_2^2 e^{-\alpha d} + 2r_1 r_2 \cos \Delta e^{-\alpha d}}$$
 Equation 2)

Where:

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$$r_1 = \frac{n_f - 1}{n_f + 1}$$
 Equation 3)

$$r_2 = \frac{n_s - n_f}{n_s + n_f}$$
 Equation 4)

$$\Delta = \frac{4\pi d n_f}{\lambda}$$
 Equation 5)

$$\alpha = \frac{4\pi k_f}{\lambda}$$
 Equation 6)

R is the reflectance. n_f and n_s are the refractive indices of the film and the substrate respectively. The film thickness is designated by d, k_f is the extinction coefficient (or the complex part of the refractive index) of the film, and λ is the laser wavelength. The two equations 3 and 4 treats the reflection in the gas/surface interface and the film/virtual substrate interface respectively, and the equations 5 and 6, the absorption.

Note that the reflectance in equation 2 is:

$$R = I_R + I_A$$
 Equation 7)

The growth rate is the differential of *d* with regards to *t*:

$$g.r = \frac{d d}{d t}$$
 Equation 8)

Or, analytically (see also equations 10-14):

$$g.r \simeq \frac{\Delta d}{\Delta t} = \frac{d_u - d_{u-1}}{t_u - t_{u-1}}$$
Equation 9)

Where u is the datum index. By supplying data for the different variables in Equation 2, with reflectance as a function of layer thickness, there will ideally be an oscillating curve with the frequency dependent on the layer thickness

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(constructive and destructive interference), and an amplitude changed by the increase or decrease in absorption in the different layers, figure 7.



Fig 7 Principal interferogram, with reflectance as a function of film thickness.

Consideration must, as shown in equations 2 - 6 above, be taken to changes in the refractive index of the samples, and therefore also to the incident angle of the light, if not normal to the sample.



Fig 8a Interferogram, showing the growth of a Fig 8b Interferogram, showing the growth of a highly absorbing film. low absorbing film

The theoretically calculated examples in fig 8a and 8b show two different cases. The first with a large extinction coefficient, and therefore absorption, and the second with a small k_f . These examples show the low sensitivity of the method when extreme values of k_f are used.

In between these extremes, materials are normally monitorable with respect to both parameters. As can be seen from the two different example graphs, the first extreme, with the large extinction coefficient, hinders monitoring of long growth runs, as the signal dies out after just a very short while. The extinction coefficient, and hence the composition can be determined with some accuracy. The second extreme has, with similar reasoning, the opposite properties.

A problem can also arise from light scattering, which results in a loss in reflectance intensity. This affects determination of the extinction coefficient, and therefore also determination of the composition. Figure 9 shows an interferogram recorded by Irvine *et al.*, with a marked scattering loss¹¹.





A scattering light loss correction line has been added to illustrate this. The graph shows a solar cell device structure growth, where the first peak, between about 1000- 1900 seconds, is a CdS buffer layer grown onto a glass/ITO substrate. This is followed by a temperature ramp, between 1900- 2700

seconds, followed by the CdTe photo-active layer grown on top, from 2700 seconds and onwards. The OMRL 389 index is the catalogue number for the sample in question.

The technique is well understood theoretically, simple and robust. The latter is due to the insensitivity to small differences in angle, and relaxed demands on optical window specifications. It is therefore the technique of choice when working in difficult environments, especially industry. The information produced is the growth rate, and if optical constants are known, the layer composition.

2.2.2 Dual wavelength reflectance interferometry.

To counteract the inherent ambiguity of single wavelength interferometry, discussed in chapter 2.2.1, a second wavelength can be used: The tracking of the three intensities I_R , I_A and I_S must be achieved independently at all times, in order to unambiguously and separably detect differences in a) Growth rate, b) Film composition and c) Surface roughening. Unambiguously separable is here used in a mathematical sense, with it being possible to split a multidimensional equation into several one-dimensional equations, and physically means that: with any of these parameters being allowed to change independently, the growth parameters are still accurately monitorable.

The combination of two lasers with a wavelength that would satisfy the two cases in figures 8a and 8b would be ideal for a dual wavelength instrument. In the situation of figure 8a, a fast indication on the roughness parameter can be determined, while for the longer wavelength in figure 8b, the growth rate would be readily monitorable throughout long runs. Normally, a UV laser in combination with an IR laser would satisfy this for most semiconducting materials of interest. However, in the case of the instrument built within this

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project, a CCD- array was used. The use of CCD arrays means the lasers' wavelengths have to be matched to the CCD pixel peak detection wavelengths, which might not be the ideal wavelength. A more general approach supported both by scattering theory and the virtual interface theory will therefore be necessary.

The CCD approach was used, firstly, to enable the laser spot distribution to be recorded, as that is what will be used for determining the roughness parameter. Secondly, there was the issue with Fabry-Perot filters, and trying to reduce complexity of the optics in the instrument. Fabry-Perot filters are dependent on the angle of incidence of the light being filtered. If used, precession of the wafers will create an effect similar to noise. The filters and the beamsplitter, that would be used to split the combined laser beam into its base colours in an analogue instrument, can with this design be left out totally. Also, the instrument is fully digital with the change from photodetectors, which also mean a lower noise level. All these effects increase the signal to noise ratio of the instrument significantly.

The Irvine reflectance equations does not actually treat the surface roughening effects, which means the scattering intensity, *I*_s, must be known from other means, before it will be possible to solve the equations for rough surfaces. The scattering intensity will therefore be independently determined by laser light profiling, and will be subtracted from the reflectance and absorption intensities. The procedure for collecting and calculating the scattering information will be discussed separately throughout chapter 2.3, and only a few remarks on the scattering information that is directly available from the dual interferometry data will be discussed here. 2.2.2.1 Reflected (interference) intensity.

The reflected interference intensity monitors the growth rate. If instead of looking at equation 2, the important features are separated from each other, and the problem is reduced to two one-dimensional equations. Thus, with the extinction coefficient set to zero, equation 2 is;

$$R = \frac{r_1^2 + r_2^2 + 2r_1r_2\cos\Delta}{1 + r_1^2r_2^2 + 2r_1r_2\cos\Delta}$$
 Equation 10)

From equation 8, the growth rate dependent equation would then be;

$$R = \frac{r_1^2 + r_2^2 + 2r_1r_2\cos(\frac{4\pi n_f}{\lambda}\frac{dd}{dt}t)}{1 + r_1^2r_2^2 + 2r_1r_2\cos(\frac{4\pi n_f}{\lambda}\frac{dd}{dt}t)}$$
Equation 11)

Analytically, with the use of equation 9, the growth rate dependent equation instead is;

$$R = \frac{r_1^2 + r_2^2 + 2r_1r_2\cos(\frac{4\pi n_f}{\lambda}\frac{d_n - d_{n-1}}{t_n - t_{n-1}}t)}{1 + r_1^2r_2^2 + 2r_1r_2\cos(\frac{4\pi n_f}{\lambda}\frac{d_n - d_{n-1}}{t_n - t_{n-1}}t)}$$
Equation 12)

Where t is time, d is film thickness, and R the reflectance. With proper control, d and t will have a linear dependency with a pre-programmed growth rate, but this cannot always be presumed to be the case.

The refractive index is of course also dependent on the wavelength, and the equations will have to be solved for each wavelength at any given point, i.e. 532nm and 655nm. The shortest wavelength decides the minimum time for knowing for certain the growth rate, which is a quarter wavelength of the interferogram oscillation for that wavelength; the fitted oscillation function (or its derivative) has to go through a zero point, and reliably change sign.

2.2.2.2 Absorbed intensity.

In a similar fashion, the absorbed intensity indirectly contains the information of the composition. Indirectly, because it has to be derived from the refractive index, which is calculated from the real-time data and has to be matched empirically to a composition¹². Following the above discussion on separating variables in equation 2, the result is;

$$R = \frac{r_1^2 + r_2^2 e^{-\alpha d} + 2r_1 r_2 e^{-\alpha d}}{1 + r_1^2 r_2^2 e^{-\alpha d} + 2r_1 r_2 e^{-\alpha d}}$$
Equation 13)

The growth rate dependent equation is similarly;

$$R = \frac{r_1^2 + r_2^2 e^{-\alpha \frac{dd}{dt}t} + 2r_1 r_2 e^{-\alpha \frac{dd}{dt}t}}{1 + r_1^2 r_2^2 e^{-\alpha \frac{dd}{dt}t} + 2r_1 r_2 e^{-\alpha \frac{dd}{dt}t}}$$
Equation 14)

Of course, only the reflection asymptote as $d \rightarrow \infty$ is interesting here. This is the reflection of the growing film, and therefore is simply dependent on the films refractive index. Hence, the composition can be detected;

$$\lim_{d \to \infty} R = r_1^2$$
 Equation 15)

$$\lim_{d \to \infty} R = \left(\frac{n_f - 1}{n_f + 1}\right)^2$$
 Equation 16)

$$\lim_{d \to \infty} R = \frac{n_f^2 - 2n_f + 1}{n_f^2 + 2n_f + 1}$$
 Equation 17)

Where the *limit* of *R* is the mean value of the negative and positive asymptote to the reflection equation, and calculated from the interferogram.2.2.2.3 Scattered intensity.

The scattered intensity is not possible to predict through the virtual interface equations. In spite of that, there are some predictions that can be directly made from the two laser signals. In the case of a normal mode

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instrument there is only one prediction to make. Because two different wavelengths are being used, according to Rayleigh, it can be predicted that;

$$I_{s_1} \propto \frac{1}{\lambda_1^4}$$
 Equation 18)

and similarly that;

$$I_{s_2} \propto \frac{1}{\lambda_2^4}$$
 Equation 19)

where the proportionality constant is the same for equation 18 and 19. This constant has to be set in real-time, by simultaneous but separable measurements. The procedure is, as stated in section 2.2.2.1, outlined throughout chapter 2.3. With proper care taken, some predictions/ approximations can be made using the linearity of the change in the scattering intensity, as researched by Stafford *et al*^{11,13} in investigating the "Roughening/Smoothening factor". This procedure places restrictions on the data, especially that the refractive index is linearly dependent with respect to change in the film composition. These restrictions are unnecessary if there already are two separable and simultaneous means of detecting correctly the scattered intensity. This is true with the combination instrument, and no approximations have to be made.

2.2.3 Spectroscopic ellipsometry.

If a sample is illuminated with light, near the Brewster angle, and linearly polarised by means of a rotating polariser, a change in the polarised component intensity in parallel[p] and perpendicular[s] orientation can be achieved at the interaction with a sample surface. This is due to different reflectivity, as illustrated in the Silicon/Air example¹⁴, figure 10. This light is reflected, passed through a rotating analytical polariser, much like in figure 11, and detected.

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This is the general principle of reflective ellipsometry. Of course, there are techniques to investigate the transmitted and scattered polarisation states too, but as the project premisses stipulates the reflection is the only usable intensity, these two sources will not be considered here.



Fig 10 Reflectance, R_P and R_s , as a function of the angle of incidence (φ), for the Si/Air system. $\lambda = 546.1$ nm, $n_{Si} = 4.05$. After reference 14.



Fig 11 Typical ellipsometer geometry. After reference 16.

There are a few techniques of reflective ellipsometry to consider, such as null ellipsometry, and also photometric- and interferometric ellipsometry. -The null ellipsometer is the most basic instrumentation, detecting the "null" angle between two of the polarisation angles in the optics arrangement of Polariser-Compensator-Surface-Analyser[PCSA]. With the "null" angle is meant the angle where the light leaving the analyser has been extinct. It follows there are six permutations of the P,C,A and the compensator retardation, but only the three permutations (P,C), (P,A) and (C,A) are ever used, as using the compensator retardation means at least an order of magnitude lower resolution. While using any single permutations, the last angle is held constant together with the compensator. Normally the (P,A) option is used, with the compensator set at $\pm \frac{1}{4}\pi$, and the retardation to quarter-wave. -Combine a *null* ellipsometer and a rotating analyser, and you get a rotating analyser ellipsometer[RAE]. which is of the second class: photometric. The data is shown as a sinus wave, and it is related to the ellipsometric angles Ψ and Δ of equation 20, with the Fourier series equation 21. The detected signal is related to the ratio of the complex Fresnel reflection coefficients, r_p and r_s , for the p and s reflection, and is thus written^{15,16,17}:

$$\rho = \frac{r_p}{r_s} = \tan \Psi e^{i\Delta}$$
 Equation 20)

Where Ψ and Δ are still being the ellipsometric angles.

If the detected intensity at any given time can be expressed as the Fourier series:

$$I = a_0 + a_2 \cos(2\omega t) + b_2 \sin(2\omega t)$$
 Equation 21)

Then the complex reflectivity can be written as;

$$|\rho| = \tan \Psi = |\tan \varphi| \sqrt{\frac{a_0 + a_2}{a_0 - a_2}}$$
 Equation 22)

Where φ is the azimuthal angle of the polariser.

The complex angle is written:

$$\arg \rho = \Delta = \arccos\left[\frac{b_2}{\sqrt{a_0^2 - a_2^2}}\right]$$
 Equation 23)

By determining the polarisation state of the reflected light, and calculating the dielectric function $\langle \epsilon \rangle^{18}$, the growth monitoring results are displayed:

$$\varepsilon = \sin^2 \varphi \left[1 + \tan^2 \left(\frac{1 - \rho}{1 + \rho} \right) \right]$$
 Equation 24)

Where the optical constants are found from the dielectric equation 25:

$$\varepsilon = n - ik$$
 Equation 25)

The Drude law can be applied to investigate the real and imaginary parts versus the wavelength¹⁹.



Fig 12a Principal ellipsometer growth spiral. Spiral starts at substrates optical characteristics, and ends showing the films properties.

The ellipsometric angles can be plotted against each other as in figure 12a, and forms a spiralling graph for absorbing films. The spiral starts at the optical characteristics of the substrate, with respect to the ellipsometric angles, to
converge at the properties of the film²⁰. The ellipsometric spectra for a Si wafer is also shown in figure 5b¹⁷.

The Brewster angle is used for minimising the plane polarised contribution to the reflection, thus creating a data stream with a good signal to noise ratio. This makes it a good technique for very thin film analysis²¹. Composition information is available via modelling, as illustrated in equations20-25. Information about the growth rate can also be extracted by using the generic reflectance/interference signal. A similar approach is achieved by exchanging the analyser for a polarisation modulator, e.g. a Pockel cell. The instrument is then referenced as a Polarisation Modulated Ellipsometer[PME]. If using one of these photometric instrument with a broadband light source, and resolving the input over the relevant wavelength spectra, the technique has been defined as Spectroscopic Ellipsometry[SE].

Lastly, the *interferometric* ellipsometer can also be setup as an instrument using, usually, the Michelson interferometer geometry. In this arrangement one measure the optical parameters over a larger part of the surface, instead of a point shape. As all ellipsometers treats the sample as an isotropic one, this removes one of the fundamental approximations.

Ellipsometry is an excellent surface analysis technique, and it is also used as a highly accurate tool in the studying of surface processes^{22,23,24}. However, the technique is dependent on the Brewster angle, which does not handle very well changes in the angle of incidence, see figure 10. As this would be expected if precession is present because of a rotating sample/substrate, and has to be counteracted by using, for example, mechanical mirroring²⁵. It also has the drawback of demanding different ports, one for the incoming light, and one for the reflected. The Brewster angle is of course also material specific, which

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means the port angles have to be variable, if every reactor top-plate is not to be specially manufactured for every single material to be grown. Because of this, the technique suited mainly for *ex situ* analysis or for research reactors only in the case of *in situ* analysis. Fortunately, most interesting Brewster angels are near 75°. More specifically, the technique is unsuitable to this project because of the reactor optical port being situated normal to the substrate.

2.2.4 Reflectance anisotropy spectroscopy.

The last current technique to present is the technique of Reflectance Anisotropy Spectroscopy [RAS]. The technique is basically an adaptation of ellipsometry to industry demands, especially the optical access limitations of the MOCVD reactor, as discussed in section 2.2.3. It is therefore in effect an ellipsometer, which works at a constant incident angle of 0 degrees, whatever the material. The reflectance interferometry signal is also used for growth monitoring. The polariser is set to the two principal axis of the surface, i.e. for (001) it would be <10> and <110>. For the reasons mentioned in sections 1.4.2 and 2.2.3, the Brewster angle has been abandoned for a normal incidence approach here. The signal from such an instrument contains not only the reflectance interference pattern as shown in regular interferometry, but also a "fingerprint" pattern, which show the anisotropic absorption of light in the film²⁶. This pattern can be visually observed and symmetry features can be designated to different developments on the surface. For example in the case of a (100) surface, the signal is²⁷;

$$\frac{\Delta r}{r} = 2 \frac{r_{[\bar{1}10]} - r_{[110]}}{r_{[\bar{1}10]} + r_{[110]}}$$
Equation 26)

And similarly normalised;

$$\frac{\Delta R}{\overline{R}} = 2 \frac{R_{[110]} - R_{[110]}}{R_{[110]} + R_{[110]}}$$
Equation 27)

Where $r_{[110]}$ and $r_{[110]}$ are the complex reflectance of polarised light intensity along the noted axis. *R* is here the modulus of the reflected and incident intensity, as indexed.

Like the ellipsometer, this is a powerful surface analysis tool^{24,28,29}. Similarly, the technique also has problems with precession, and the commercial instruments regularly use mirrors to combat the problems. Because of the movement mechanics and optics that are needed in order for it to work properly, it is also a substantial instrument to house. For example; it would require the larger part of the glove box that protects the loading mechanism of the Aixtron 2400 reactor. Because of the complexity of the RAS monitor relative to the simple design of a reflectance interferometer, and as the surface sensitivity can be achieved also there with the use of short wavelength lasers, RAS was also discarded as a technical solution for this project.

2.3 Three dimensional layer growth.

This section will cover a discussion of a few techniques that can be utilised for the monitoring of three dimensional growth in a film. The discussion will concern laser light scattering, in the form of angle resolved scattering, and also laser spot profiling. The point of investigating these techniques, is to find the scattered intensity of reflectance interferometry, which will unambiguously make 3D growth monitoring possible. Scattering and surface roughening is also connected, which will be shown, where roughening can be easily monitored, as long as the scattering contribution is known.

2.3.1 Angle Resolved Scattering.

Rayleigh scattering is the elastic scattering of light from matter. The effect arises from disturbances from all optical scattering centres in the material, in the form of defects^{30,31}, grain boundaries³²/particulate sizes³³, surfaces^{34,,35} and other impurities/imperfections^{36,37} in the sample. The normal behaviour for Rayleigh scattered light is well known. The scattering intensity distribution is shown in figure 13, and is radially uniform around the scattering point. According to the Rayleigh relations it is distributed as cos²θ for polarised light,

and as $1 + \cos^2\theta$ for unpolarised³⁸.



Fig 13 Scattered intensity distribution with minima (zero by definition) parallel with sample.

Figure 14 shows the effect of surface roughness on the scattering intensity distribution, where the leftmost show the distribution of a perfectly smooth surface. The scattering intensity is by definition zero here. The middle distribution show a slightly rougher surface, and the almost Gaussian appearance of the distribution. Further right, there is an example of a very rough surface. As a result the scattering distribution has gone through an "inversion", where the centre of the distribution has no longer the largest

intensity. This is due to the change from a mostly horizontal surface, when looking at individual grains, towards a mostly vertical.



Fig 14 Scattering increases with the onset of lateral growth. Leftmost beam is specularly reflected. Further right, rougher surfaces scatter the beam, and the reflected intensity decreases.

Because of the constraints in the optical access of the reactors explained in section 2.1, only the case of normal incident laser light will be considered. Thus, the scattering intensity will go through a minimum parallel to the sample, normal to the laser. A maximum will also be present normal to the sample and parallel to the laser, if the scattering is not too severe. This intensity is due to the specular reflection. This gives an excellent way of calibrating the azimuthal θ -axis of the angle resolved scattering I/θ graphs, as 90 degrees is exactly the angle between the maxima and minima. This is, of course, only true for low to moderate scatterers, see figure 14.

The above uniformity really only applies to a point-shaped scatterer, and to the simple uniformity rule, components like surface diffraction and orientation³⁹ can change the scattering. This though, has the advantage of giving new information if these effects are present. Unfortunately, for the application considered here, the narrow optical access does not permit angle resolved scattering [ARS]. It *does* help with understanding what happens when a surface grows rougher, though, and some data will be presented in the results section. An assessment of the features of the central beam, and its corresponding reflection, is more or less the only option available for detection of scattering with limited optical access. The integral of the AR scattering distribution[I_S] over all solid angle is, by definition, equal to the total scattered intensity[TIS], $I_S = TIS$, discussed later in section 4.1.2.2 and also in 4.2.3.

2.3.2 The scattered interferometry signal.

A major drawback of normal interferometry is, as discussed above, the inability to distinguish between absorbed and scattered light loss in the data in all but the trivial cases; i.e. when the scattering intensity or the extinction coefficient is zero. Analysis of the data after a growth run can always be done, but as one of the fundamentals of growth monitoring is that it has to be done real-time, this is not enough.

The way to distinguish the difference is by either measuring transmittance and reflectance of the material, which gives the absorption, or by measuring the scattering directly. Transmittance of light through the sample in a production reactor is, for practical reasons, not possible, which leaves the scattering. Measuring scattering directly can be done in dual wavelength reflectance interferometry, by referencing the first laser beam to a second wavelength laser with a suitable wavelength, that only scatter during growth. In this case, with the use of CCD matched frequencies, two differently scattered wavelengths instead have to be relied on for referencing. The scattering intensity is then correlated as shown in section 2.2.2.3. See also equation 37 and the related discussion. Scattering is a desirable feature to investigate in its own right.

For good performance in electronic devices, control over surface morphology is critical⁴⁰. Some manufacturing methods, like for GaN devices,

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even involve monitoring a roughening step, as part of the normal growth procedure⁴¹. Surface roughening occurs in CVD as initial two dimensional Frank-van der Merwe growth transfers into three dimensional growth. In the experiments that will be presented in the results section, the growth would be of Stransky-Krastanow type, but any relevant type would be monitorable. Roughening will occur sooner or later for most materials depending on the specific thermodynamics, and must be controlled.

By measuring the scattering from two different laser beams as described above, where the two beams are absorbed and scattered by different amounts in the sample, a situation (from equation 1) is achieved, where:

$$I_{\lambda_n}^0 = I_{\lambda_n}^R + I_{\lambda_n}^A + I_{\lambda_n}^S + I_{\lambda_n}^T$$
 Equation 28)

The intensities are of incident[0], reflected[R], absorbed[A], scattered[S] and transmitted[T] type respectively. With the Silicon wafers used in the experiments, the transmittance would be zero, and the equation reduces to;

$$I_{\lambda_n}^0 = I_{\lambda_n}^R + I_{\lambda_n}^A + I_{\lambda_n}^S$$
 Equation 29)

Normalised, this would be;

$$1 = \frac{I_{\lambda_n}^R}{I_{\lambda_n}^0} + \frac{I_{\lambda_n}^A}{I_{\lambda_n}^0} + \frac{I_{\lambda_n}^S}{I_{\lambda_n}^0} = {}^0I_{\lambda_n}^R + {}^0I_{\lambda_n}^A + {}^0I_{\lambda_n}^S$$
Equation 30)

All intensities are normalised in the discussion from now on.

As scattering is wavelength dependent, this has to be accounted for, where the Rayleigh dependence of $I \propto \lambda^{-4}$ would be expected. However, Carniglia *et*. *al.* has showed a dependence of⁴²:

$$I^{S}_{\lambda_{n}} \propto \frac{1}{\lambda_{n}^{2}}$$
 Equation 31)

for surface induced scattering, and only one arbitrary surface.

Furthermore, by combining equation 30 and 31:

$$1 - I_{\lambda_{n}}^{R} - I_{\lambda_{n}}^{A} = I_{\lambda_{n}}^{S} \propto \frac{1}{\lambda_{n}^{2}} = k \frac{1}{\lambda_{n}^{2}}$$
Equation 32)
$$k = (1 - I_{\lambda_{n}}^{R} - I_{\lambda_{n}}^{A})\lambda_{n}^{2}$$
Equation 33)

Equation 33)

Where k is an aggregate of constants and variables.

Taking into account the two wavelengths, equations 31 and 33 gives:

$$I_{\lambda_1}^{s} = k \frac{1}{\lambda_1^2} = (1 - I_{\lambda_2}^{R} - I_{\lambda_2}^{A}) \frac{\lambda_2^2}{\lambda_1^2}$$
 Equation 34)

Finally, equations 33 and 34 give:

$$1 - (I_{\lambda_1}^R + I_{\lambda_1}^A) = (1 - I_{\lambda_2}^R - I_{\lambda_2}^A) \frac{\lambda_2^2}{\lambda_1^2}$$
 Equation 35)

or either of the two functions:

$$(I_{\lambda_1}^R + I_{\lambda_1}^A) = 1 - \frac{\lambda_2^2}{\lambda_1^2} (1 - (I_{\lambda_2}^R + I_{\lambda_2}^A))$$
 Equation 36)

$$\frac{I_{\lambda_1}^3}{I_{\lambda_2}^s} = \frac{\lambda_2^2}{\lambda_1^2}$$
 Equation 37)

As stated above, all intensities are normalised against the incident intensities. The point of equation 36 is just to show a relation between the two different data sets; the left hand sum, is what is measured at one wavelength in normal laser reflectance interferometry (see equation 7), and the similar parenthesis on the right hand side the measured intensity at the other wavelength. Equation 37 instead shows the relationship between scattered intensity, which would be the measured intensity subtracted from theoretical. A dual wavelength system will, thus, correct the scattering problem of the single wavelength interferometer. This would enable the monitoring of the composition of the material.

2.3.3 Monitoring roughness with scattering measurements.

Le Bosse *et al.*^{43,44}, discussed the theoretical aspects of the problem in some detail, and state that Rayleigh scattering, as a model, is far from accurate in most cases. They instead deployed a model that does not assume that the distribution is of any particular kind. The resulting equation shows a dependency between the scattered intensity and surface roughness⁴⁵:

$$l = \sum_{p=0}^{\infty} \frac{(-1)^p}{(2p)!} u^{2p} \alpha_{2p}$$
 Equation 38)

Where I is the factor needed to correct the measured reflectance I_{meas} to the theoretical reflectance I_R , or:

$$I_{meas} = I_R l$$
 Equation 39)

Factors $u \alpha$, μ and γ from equation 38 are defined as follows:

$$u = 2k_z^+ \sigma = 4\pi \cos\theta \frac{\sigma}{\lambda}$$
 Equation 40)

$$\alpha_{2p} = \frac{1}{2} \sum_{k=0}^{2p} \binom{k}{2p} (-1)^{2p-k} \mu_k \mu_{2p-k}$$
 Equation 41)

$$\mu_n = \frac{\gamma_n}{\sigma^n}$$
 Equation 42)

$$\gamma_n = \int_{-\infty}^{+\infty} z^n \, p(z) \, dz \qquad \qquad \text{Equation 43}$$

where σ is the (rms) surface roughness, p(z) is the distribution function and z the surface function ordinate. k^+z is the component of the light wave vector, perpendicular to the surface. These equations require the lasers to be linearly polarised. For surfaces with a small amount of roughness, in the case of the studies done within this project framework: under about 20nm, the first two terms of equations 41 are a good approximation. The resulting equation is then basically the Rayleigh relation for polarised light, see also figure 13:

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$$l = 1 - \left(\frac{4\pi\sigma}{\lambda}\right)^2 \cos^2\theta$$

According to Ohlidal *et al*⁴⁶, the root mean square (rms) of the surface roughness σ , is related to the scattered intensity. Assuming an illuminating beam, with an incident angle of θ_{θ} :

$$\ln I^{S} = \ln I^{0} - k_{z}^{2} \sigma^{2}$$
 Equation 45)

maintaining same variables as the equation 28-37, and;

$$k_z^2 = \left(\frac{4\pi\cos\theta_{scat}}{\lambda}\right)^2$$
 Equation 46)

Where a requirement, as above, is a reasonably smooth surface;

$$k_z^2 \sigma^2 \ll 1$$
 Equation 47)

with σ the root mean square (rms) roughness⁴⁷.

Yang⁴⁸ *et al.* also stated that within an interval, at least ± 4 degrees azimuthal, the scattered intensity can be written:

$$\frac{I^{s}}{I^{0}} \propto \frac{1}{\left|\theta_{scat} - \theta_{0}\right|^{3}}$$
 Equation 48)

which here, with the incident angle $\theta_0 = 0$ is:

$$\frac{I^{S}}{I^{0}} \propto \frac{1}{\theta_{scat}^{3}}$$
 Equation 49)

Which is an interesting approximation, as this is within the parameters of the project.

Research by Bennett and Porteus⁴⁹ led into the simplified scattering theory, where the macroscopic roughness (Fourier surface wavelength > $\lambda/2$) is written⁵⁰:

$$R = R_0 \exp\left[-\left(\frac{4\pi\sigma}{\lambda}\right)^2\right]$$
 Equation 50)

Where R_{θ} is the reflectance from a non-scattering sample, and σ is the roughness. This seems to be in agreement with the Ohlidal equations 45-46.

2.3.4 Laser light profiling.

The central reflected beam of the interferometer is, as stated, the only source for measuring scattering data. This is because of the narrow optical access to the reactor, an orifice normal to the substrates, with an opening of $\pm 3^{\circ}$. The basis of this new approach is the changes the Gaussian profile of the laser beam will have when it interacts with a developing surface. The integral over the whole central reflection spot is, of course, only the intensity normally used in interferometry. The change in the Gaussian intensity distribution of the laser, can be of use to study the surface, though. By interaction with the rough surface, the basic laser light profile will change, as modelled and explained in section 4.3.7. The Gaussian intensity profile is written the normal way:

$$I = I_0 e^{-kx^2}$$
 Equation 51)

rearranging this gives the linear expression:

$$\ln \frac{I}{I_0} = -kx^2$$
 Equation 52)

By plotting the logarithm of the normalised intensity against the square of the physical pixel number, a linear dependence should be present, as in equation 52. The experiments presented in this thesis, show a monitorable trend in the changes of the Gaussian profile with increase in atomic force microscope [AFM] correlated (rms) roughening. This is consistent with the assumption made, that an increase in scattering is likely to change the LLP slope (k in equation 51-52) with a monitorable amount, as intensity is lost because of ARS. The data in the AFM correlation measurements must be normalised with respect to both wavelengths in the dual wavelength

combination instrument. By then using the Ohlidal⁴⁶/Bennett/Porteus⁴⁹ equations on the AFM correlated roughness values, the scattered intensity can be calculated. This scattering intensity is treated as in equation 37, and can be compared to the interferometer scattering data. Determination, unambiguously, of scattering intensity contribution and hence the film roughening is thus possible.

2.3.5 Orientation effects.

Even though large efforts are being made to change substrates from the expensive crystalline materials, to cheaper materials, like glass or plastic, there are still large issues to be solved before this becomes viable. One such issue is controlling growth epitaxially, where a preferred orientation can be grown onto a known crystal structure. This can generally not be controlled by using amorphous substrates. This make single crystalline substrates desirable in certain applications, in spite of their higher price.

Scattering data, collected off-centre of a rotating sample, would directly show signs of preferred orientations, if any are present. Sterligov *et al.*⁵¹ studied GaAs, grown homoepitaxially onto single crystalline (100) GaAs, where the resulting graph, in figure 15, shows scattered intensity at different azimuthal angles. We can easily make out different GaAs crystal planes, as overlaid in figure 15, and decide the orientation of the film. A film grown on top of an amorphous substrate would, treated the same way, show only a simple Gaussian distribution, with its maximum in origin.



Fig 15 GaAs sample showing orientation effects. A few directions have been inserted. After reference 51.

Church *et al.*^{52,53,54} also discussed artefacts from ordered surfaces, modelled as macroscopic surface functions. These usually arise from, for instance, micro-machining or polishing effects on the surface, although epitaxial films should be analogues. Until now, the discussion has mainly concerned smooth surfaces, as these are common when working with optical films, but as some of the experiments have been performed to leave a rougher surface on a highly ordered substrate (single crystal), macroscopic scattering must be discussed for completeness. These surfaces are certainly not as rough or ordered as the tooled ones, but are in the border zone between smooth and rough films.

For smooth films, there is the diffraction model that explains the scattering for surfaces with a roughness under about 20nm. After this, a geometrical model is used, which takes into account facetting; real reflections from developed crystal facets. This scattering becomes prominent as all incident

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light are being translated into scattering (as compared to specular reflectance), which happens at about 100nm. Some orientation growth data will be presented in the results section 4.2.4, where CdTe have been epitaxially grown onto single crystal Si(001). This data shows an example of the interval in between the two modelled regions, with intermediate surface roughness at the magnitude 10-100nm. Here, some ordering is evident, although no defined grating effects can be seen.



Fig 16Different principal surface textures and the scattering that would be expected from theory. After reference 52.

Church explains the behaviour of scattering with the combination of one or several effects. Figure 16 shows the progress of roughening of the surface, from the trivial case of non-scattering, perfectly smooth surfaces (a), through random 2D build-up of unordered speckle points (b), or facets, until eventually ordered structures. As he discusses ordered surfaces, i.e. with a preferred orientation, he also distinguishes between the randomly spaced(c) and the periodic case (d). Any permutations of the four cases are possible, but a mix between all four is likely, if orientated tooling has occurred. At any of these permutations, the resulting scattering would be the combination of the distributions in question.

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3 Instrument design and experimental.

Three instruments have been designed, throughout the duration of this project. The first instrument was an *ex situ* instrument, designed in order to investigate the angle resolved scattering [ARS] over a large interval of surfaces, ranging from very smooth to very rough. After having studied the ARS scattering of dozens of samples, the conclusion was that, even if the data were very informative, the AR technique, as such, cannot resolve the issues with the narrow optical port of the 2600 reactor. A second, *in situ*, instrument was therefore built, with the ability to profile the laser beam reflection. A standard, single wavelength, normal incident interferometer was built, with the exception of the detector, which was exchanged for a CCD-camera. The profiling system was considered to be suitable, as a generic solution to the optical port problem. The final instrument was therefore based on this, only with the addition of a second wavelength, to solve the inherent problems of the single wavelength interferometry.

3.1 Experimental setup I. Angle Resolved Scattering.

In order to develop a combined instrument, partly based on collection of scattered light, certainly a more in depth analysis of scattering data in a test environment would be desirable. Assuming Rayleigh scattering as described in the theory section, an instrument would have to be designed to resolve the angular dependence, in order to find a way to resolve the problem of the narrow geometry of the (Aixtron reactor) pyrometer port.

3.1.1 Instrumentation remarks.

Finding out the distribution of scattered light from thin film photovoltaic surfaces is the major concern, as this has not been previously characterised for the CdTe/CdS system^{1,2,3}. As the distribution can be considered radially

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uniform, and centred from the incident beam, an instrument that makes it possible to scan the whole 90 degrees azimuthal had to be built; i.e. from parallel to normal relative to the wafer. Signal recovery in the extreme angles was possible only for the parallel part (~ 90 degrees azimuthal), as the detector moved in the way of the laser within two degrees of the normal part (~0 degrees azimuthal). This was resolved later, with the addition of a beamsplitter to the setup. The beamsplitter could then move in front of the laser, with a signal still being recorded. Some scans were made on the backside (transmittance) of the samples, to establish the 90 degree point; the backside was originally scanned to obtain the transmittance the wafers. There was generally very low transmittance, but one could notice a small but significant transmittance about where the 90 degree point was expected to be. The transmittance was confirmed to be the from the central beam, as the scattering distributions were symmetric about this point. The samples were illuminated with 1.5W of the 514 nm laser line of an Ar⁺ laser, which means that the transmittance would be close to zero at normal light intensities.

An apparatus was constructed to scan over a range of 90 degrees from the reflected normal with computer control of the scanning process. Manually moving the scanner at a constant rate for a long time with constant interval stops, would have been very inaccurate. As a class 4A laser was used, health and safety regulations also required a scattered light confinement container to be constructed.

3.1.2 Instrument geometry.

A motor gondola was built by using three aluminium L-profiles and a stepper motor. Two gears, a motor gear and a drive gear, were connected at a right angle, thus enabling a 2:1 gear ratio. A threaded rod was fitted with an

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end bearing which was then screwed into a nut, welded directly onto the drive gear . This assembly made up the linear drive as shown in figure 17.



Fig 17 Showing three Al L-profiles assembled to a motor-gondola(1). A threaded rod (2) moves linearly, after the circular motion of a motor gear(3) has been translated 90 degree into a driver gear(4). This gear is fitted with a nut fitting the threaded rod.

A 12 mm steel rod was threaded at one end, and fitted with a ball bearing as in figure 18. The rod was intended to be the centre column of the AR scatterometer. On top of this centre rod a flange was attached, in order to hold the sample wafer vertically. A flat iron bar was drilled with 50 mm spacing, centred on a middle hole. The flat iron was then fitted onto the centre column rod by using this middle hole, and was bolted to the ball bearing. This made up a variable radius turntable.

The detector was housed in a small instrument box, with additional wiring and BNC couplings, and with a 0.25 mm slit fitted in front of the detector in order to enhance resolution. The box was provided with a steel rod as a mounting pin, for mounting on the turntable. The setup is shown in figure 18.



Fig 18 Simplest possible setup to make a turntable for the 90 degree scanner. Ontop a centre rod(1) is a sample holder for mounting wafers vertically(2). The "turntable", is an iron bar(3) connected to the centre rod via an end-bearing. A detector(4) is mounted onto the stock.



Fig 19 Complete scanner setup, with computer controlled linear drive, which translates linear movement into a rotatory movement on the turntable.

All this was assembled on the laser testbed, as seen in figure 19, with the sample directly in front of the beam. The drive rod was attached to the second (10cm) iron hole at right angles to the bar, with the gondola attached 5 cm further down. The detector was added, at either the + or -10 cm hole (depending on if the transmitted or the reflected scattering of the sample was being scanned).



Fig 20a Device interior showing motorgondola, turntable and detector setup.

Fig20b Experimental exterior, showing encasement and beam path protection between laser and case.

The device was covered by a 50x 50x 25 cm riveted aluminium box, with a hole at one end to let the laser beam in. A black box kit was fitted at the other end, in order not to allow any stray reflections or scatter. Between the aluminium enclosure and the Ar⁺ laser, a 5cm diameter PVC pipe was fitted. The enclosure can be seen in figures 20a and 20b.

A black cardboard was laid as a floor of the container, while everything else was painted matt-black in order to minimise reflections. Hinged doors had to be added for changes of the samples, and for access for alignment. These were sealed with rubber draft sealant, to seal out stray light.

3.1.3 Electronics.

The stepper motor driver was based on a RS 217-3611 unipolar 2A eurocard stepper motor drive board. As the device was supposed to be controlled from the parallel port of a PC, and the logic gate of the RS driver board was 12V, a



Fig 21 Circuit schematic for the opto-electronic coupler between 5V TTL on the parallel port of the computer, and the 12V CMOS logic of the stepper motor driver board.



Fig 22 Stepper motor steering unit, powered from a 240AC/24VDC unit. The setup is controlled from a PC parallel port.

converter between the PC 5V TTL and the 12V logic was made. The converter was fitted also with a Hewlett&Packard HCPL2300 opto-coupler on each channel, in order to protect the PC motherboard from the relatively high current on the driver board side. A schematic diagram of the converter is shown in figure 21. The device was added between the driver board and parallel port, as shown in figure 22, and was powered by a 12V line-out of the RS driver board. This 12V was regulated to 5V, which is the operating voltage of the HP opto-couplers.

3.1.4 Software drivers and applications.

The software to be written for this study had to he redesigned several times. The problem was the sleep() function, needed to let other processes use the processor when not in use by the stepper program, for instance the IDE bus, and the A/D card. The sleep() function takes just one clock cycle to start, but as the processor queues have to be scanned as it awakes, several milliseconds are used. At a 1 kHz timer rate, this is obviously not possible to combine with a 90 MHz processor. The highest usable rate was instead about one tenth of the preferred. Finally this was resolved by ignoring the sleep, putting the processor on 100% idle, and instead calculating the exact cycle an analogue sample is to be taken, and hardcoding it into the program. Everything was also buffered into RAM, in order to reduce the use of sync(), and the experiments were run in minimal mode, shutting down all unnecessary processes.

Everything was programmed in C++, using libstdc++ 2.8.0 and gcc g++, the GNU C++ compiler. It was run and compiled on a standard Mandrake 7.0 system, upgraded to kernel 2.2.16. The COMEDI toolkit was also used, allowing the use of the Advantech PCL711b A/D sampler card. The generic parallel port was used to transmit the signal to the RS driver board, as four

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separate signals were needed; start/stop, clock, direction and half/full step. With the addition of opto-couplers to the setup, the TTL logic was negated, and had to be altered (for further information on the software used, the C++ source code is listed in Appendix 7.1).

3.2 Experimental setup II. Single wavelength reflectance

interferometry.

The second system built, was an adaptation of single wavelength interferometer. The design differs on two accounts: the detection apparatus was changed for a CCD-camera, in order to be able to profile the laser beam. A system for creating Newton's rings on the surface was also added. The rings were intended to facilitate the monitoring in that it would separate the true scattered and reflected intensity.

3.2.1 CMOS - Detector array.

The CMOS-array in use for these experiments was an Agilent HDCS-1000 detector, with a resolution of 352x288 (CIF) pixel at 24-bit depth and 8fps. The array was taken from a standard Dexxa(TM) Webcam, a very low priced web camera. The sensor array can be seen in figure 23.



Fig 23 The HDCS-1000 sensor array.

The pixel-sensors, each square with a 7.8 micron side, operate by means of four sub-pixels, which are divided into one blue, one red and two green pixels. The two green pixels are there, because the human eye perceives the green as a "bright" colour, and one of the green pixels therefore controls "brightness".

The RGB colour response can be controlled separately, and the frequency range of the different colour pixels can be seen in figure 24.



CMOS response in 400-800nm interval.

Fig 24 Spectral Response of the Agilent HDCS1000 CMOS-array.

The range was decided by mounting the CMOS on a SPEX 1681 0.22m spectrometer, and measuring the response as a function of wavelength between 400 -800 nm, as shown in figure 24. The relative intensities of the three different colours, are just an artefact of the individual red, green and blue settings at the time of the experiment. They are not to be referenced against each other. Agilent was contacted prior to this experiment, in order to get the reference values, but declined to release that information. When referencing this to other, similar CCD- arrays, for example the Sony ICX414AQ, or the Philips FXA1012, the results were comparable.

3.2.2 Testbed geometry.

Initial studies concentrated on *ex situ* measurements, and the results characterised the behaviour of scattering from CdTe and CdS films. An *in situ* testbed was then built, which was intended to be mounted on top of a

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horizontal Thomas Swan reactor. This determined the specifications of the optics and its dimensions. The testbed was constructed as a tripod (figure 28) and three levels of optical mountings were attached to this. The first, uppermost mounting plate, held the laser, together with a tilting mount which can adjust the X-Y placement of the laser spot, as seen in figure 25.



Fig 25 First plane holds a laser(1), which holder(2) can be adjusted in 3D with three screws.



Fig 26 Second plane with CMOS detector(3), beamsplitter (inside the white nylon holder (4)). A second detector(5) was added in order to measure the initial intensity of the laser.

The second plane, in figure 26, hold a focusing optics, a beamsplitter and the two detectors: a CMOS array, for the imaging and for the simultaneous interferometry signal, and a photodetector to measure the incident intensity. The second detector is only there as a precaution, to measure any change in intensity of the lasers due to thermal drift. Any signal from the CMOSdetector, is therefore normalised. The third plane holds a lens for beam shape focusing. This can be viewed in figure 27.



Fig 27 The third plane holds only a lens arrangement for focusing the image onto the CMOS array.

The dimensions of the triangular planes were 180 mm sides, on a 5 mm aluminium plate, with a 20mm hole drilled in the centre of the first two, and a centred 50 mm hole in the last. The planes were spaced by 110 mm, which was not critical, only enough to fit the optics. The tripod legs have been shaped, in order to mount and centre the testbed over the TS reactor. The complete assembly can be seen in figure 28.



Fig 28 Normal incidence combined interferometer and profiler with 3 planes of optical mounts, and space for a Thomas Swan single wafer reactor underneath.

3.2.3 Software used.

All software was compiled and run on a Linux Mandrake 8.1 system, with the 3.0.3 gcc compiler, and glibc 2.4.4. The software is loosely based on w3cam 0.6.6, as written by Rasca Berlin, but very much rewritten. To control the CMOS camera, the qce-ga CVS drivers was used. This can be found on SourceForge. The photodetector was controlled with COMEDI, the Linux Common Measuring Device Interface. Once again, the C++ code of the software is listed in Appendix 7.1.1.

3.3 Experimental setup III. Dual wavelength reflectance

interferometry.

With the data from the second instrument setup having been evaluated, the design was finalised. The experimental results showed the single wavelength instrument worked as specified, and two new lasers with the wavelengths of 532nm and 655nm were purchased. The two colours were chosen to match the CCD pixel wavelengths. As the red laser was a diode laser, the wavelength will drift with temperature. The 655nm wavelength was chosen, because the red CCD pixel has a response plateau for about 10nm around that point, see also figure 24.

3.3.1 The dual wavelength combination interferometer and profiler.

The arrangement of three triangular aluminium plates from section 3.2, was assembled with the three threaded rods in the same fashion as in the single wavelength instrument. The first plate was modified to hold, from above, an arrangement of two low powered, one 5mW and one 10mW laser, mounted to supply a single overlapping two colour beam. The 10mW laser was reduced in intensity by adding a 50% narrow band filter, to match the power of the 5mW laser. The second plate assembly was modified to hold laser light dispersion optics, and a variable neutral density filter, together with the detectors and optics, already discussed in the single wavelength instrument text. The third plate was left untouched.

The two laser wavelengths were chosen as 655 nm (red) and 532 nm (green) after performing a spectral response measurement on the CCD-array, see section 3.2.1. The CCD-array was also changed to a Philips PCVC740K, because the frame rate was found to be too slow with the Agilent device. The

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new CCD had the same spectral response as the Agilent. At the same time, the Philips CCD had a VGA resolution, which is four times that of the Agilent CCD. Philips also sponsors and support their devices on multiple platforms, and was very helpful when asked for information. Agilent would not supply any device specifications, which for example led to the spectral response measurements. The Agilent drivers are currently maintained by voluntary developers, but the quality of the drivers are not as good as the Philips', simply because of lack of documentation from the manufacturer.



Fig 29 The second modification on the normal mode interferometer differs on two accounts. Firstly, the laser setup has changed to a dual wavelength setup, as can be seen on the first of the three shelves. The second change is the new CCD, with VGA resolution and 16 fps.

Figure 30 show the final instrument prototype, mounted over the OMC Thomas Swan 2" single wafer reactor, The attachment of second laser and tripod mounting rods are clearly shown, in comparison to the figure 29, as the whole setup is viewed from the opposite direction. The analogue (photodiode) electronics boxes were placed outside of the reactor fume cupboard for convenience, even if this adds some noise. The analogue signal was just for laser drift correction, though, so the sampling time can be fairly substantial to reduce the noise. The analogue electronics can be removed from the instrument, as an an engineering prototype is built; during the experiments, the two lasers were stable, and did not need to be corrected.



Fig 30 The dual wavelength combination instrument, mounted over the Thomas Swan single wafer reactor.

The rest of the instrument is digital, and cable lengths are not contributing to the noise as long as the bus specifications are met. The instrument is plugged directly into the control computer through the USB port. The power supplies to the lasers are an issue when it comes to noise though, and would preferably be of a better specification.

3.3.2 Software used.

At the time of the experiments, a generic Mandrake 9.0 system was used, with the gcc-3.2.1 compiler suite, and standard linux-2.4.19. A proper user interface was coded this time, based on the SDLcam-0.4.7 code, and therefore also the Simple Directmedia Layer. Appendix 7.1.2 hold the listing of the regression code used in these experiments.
- M. U. González, J. A. Sánchez-Gil, Y. González, L. González, R. García, A. San Paulo, J. M García, Surface characterization of III-V heteroepotaxial systems by laser light scattering, J. Crys. Grow. 201/202 (1999) 137-140.
- 2 M. U. González, Y. González, L. González, M. Calleja, J. P. Silvera, J. M García, F. Briones, *A* growth method to obtain flat and relaxed In_{0.2}Ga_{0.8}As on GaAs (001) developed through in situ monitoring of surface topography and stress evolution, J. Crys. Grow. **227/228** (2001) 36-40.
- 3 D. Oelkrug, J. Haiber, R. Lege, H, Stauch, H-J. Egelhaaf, *Temporal stability of vapor-deposited* molecular films as studied by laser light scattering, Thin Solid Films **284/285** (1996) 581-584.

4 Experimental results and discussion.

In this section there will be a presentation of the experimental data and interpretations done throughout the project. Steps of the analysis will be detailed for a few representative data sets, and the rest will be tabulated. Two different sample indexations will be used: OMRL#, samples taken from the Opto-electronics Materials Research Laboratory, North East Wales Institute, Wrexham, Wales. CB#, samples grown by the author to test the final instrument. Furthermore some reference to a OMC set will be made, with samples from the Opto-electronic Materials Chemistry group, University of Wales, Bangor, Gwynedd, Wales.

Firstly, the ex situ angle resolved scattering[ARS] data, measured throughout this project, will be presented using the OMRL samples. The scattering distributions of interest will be shown, and an analytical discussion of this will be presented. A discussion on why this will not be appropriate as an in situ monitoring technique will be held. Secondly, a novel technique, using changes in the specularly reflected laser spot due to interactions with the sample, will be presented using the CB samples. By using properties of the specularly reflected beam, instead of off-normal scattering, there will also be a possibility to use interferometric growth monitoring in combination with the roughness monitoring. A section on single and dual wavelength interferometry, with data that confirms the possibility to use this in conjunction, and a section discussing cross-correlating the relative scattering proportions of the laser intensities will be included. The primary objective of the instrumentation project was to find a way of extracting roughness data from the specified narrow orifice. But at the same time using the interferometer data, which can be measured without any extra complications to the hardware or software, would be adding another attractive feature to the final instrument.

4.1 Angle resolved scattering.

As explained in the introduction, the angle resolved scattering[ARS] from the semiconductor surfaces¹, was the first subject of study. This section concerns this scattering, and a presentation of the results of the relevant measurements.

4.1.1 Materials used.

The samples which were analysed, came from the Opto-electronic Materials

Chemistry[OMC] laboratory, department of Chemistry, University of Wales,

Bangor, and were grown at the different reactor settings shown in table 1. The

samples were the materials reported in the paper by Irvine at al², already

discussed in the theory section.

Table 1 MOCVD growth conditions and results for some CdS/CdTe photovoltaic runs. The dopant levels are shown as flow through their respective bubbler, and the II/VI ratio are the precursor concentration entering the reaction chamber.

OMRL No.	CdS growt h t/°C	CdTe growth t/°C	S:Cd ratio @ growth	Te:Cd ratio @ growth	Cl(CdS) mol. ratio	As(CdTe) mol. ratio	CdTe Growth rate Å/s
366	290	320	0.2	1.04	4	2	6.1
367	290	320	3.93	1.07	4	2	7.2
381	275	320	1.86	2	0	2	7.4
386	275	320	1.9	0.41	0	2	3.8
391	275	320	1.86	0.57	9	2	4.6
395	275	320	1.86	0.57	9	8	4.6
423	290	350	1	0.6	0	2	4.3

All samples from the Irvine paper were used, except the OMRL388 sample which showed an abnormal behaviour in the initial wafer backside (transmission) scans, see figure 33. The indexation comes from the laboratory catalogue number, and is short for *Opto-electronics Materials Research Laboratory*, which was the name of the research group at its former location, NEWI, Wrexham. The OMRL423 sample was added to the series, because it forms a link between the high and low temperature samples, as it is on the rougher side of the low temperature samples, and on the smoother side of the higher temperature samples. This was grown after the publication of the Irvine paper. The device structures are all slightly different with respect to the composition, and have been grown at slightly different temperatures. The surfaces should not be significantly different, though, in any other way than that the roughness varies. This has also been confirmed by AFM measurements.

4.1.2 Calibrating scans.

As the driver for the experiments is a linear one, but is connected to a radial scanning device, a conversion of the raw time dependence data, to angular dependence is needed. The calculations are based on simple geometry, as seen in figure 31, but the resulting function is nevertheless:

$$acos(\frac{2r^{2}-2x_{0}(z-x_{0})\frac{s-s_{min}}{s_{max}-s_{min}}-(z-x_{0})^{2}\frac{(s-s_{min})^{2}}{(s_{max}-s_{min})^{2}})-atan(\frac{1}{2})=\theta$$

Equation 53)

where θ is the angle of the circular translation in radians, as a result of the linear translation *s* in stepper motor steps. s_{\min} and s_{\max} are the step indices, where the azimuthal is 90 and 0 degrees respectively. The entities x_0 , *z* and *r*, are marked in the principal sketch of the AR scatterometer geometry in figure 31. Angle II is the analytical angle θ .

Figure 31 shows the AR scatterometer seen from above, where the laser enters the instrument from the top, and hits the sample mounted vertically in the centre of the instrument. The linear drive is attached in the lower left corner, which makes x_0 the length of the linear drive at 90 degrees azimuthal. The variable x_j is the length of the drive at an arbitrary angle of the scan, and the constant z is the length at 0 degrees azimuthal. Equation 53 expresses the relation between the angle θ , which is the angle of the detector relative to the sample (positioned at x_j), this time expressed as the number of steps from the stepper motor.



Fig 31 Principal sketch of the AR scatterometer. Sample is placed vertically in the centre of the circles, and the laser beam enters top centre of the instrument. Angle II is the analytical angle θ .

The azimuthal angle-axis (θ) was calibrated with a scan of a set of CdS/CdTe and ITO samples, seen in figure 32, which show the scattered intensity as a function of the step index. Both transmitted and reflected scattering were looked at. The transmitted intensity showed the maximum at 180 degrees azimuthal, as the detector moved into the specularly transmitted intensity. For the transparent ITO film, the detector rapidly saturated, while a smaller intensity was detected for the OMRL388 sample. The specularly reflected intensity was not accessible, as the detector moved into the beam

path, and effectively shadowed the light. Instead, the transparent intensity was used as the 0 degrees azimuthal angle indicator. In the case of the 90 degree azimuthal angle, a combination of all transmitted and reflected intensities pinpoints the position. Figure 34 shows the V-shape that was used to determine this angle. The intensity is, according to Rayleigh, zero at this 90 degrees angle.



Fig 32 Four scans combined to calibrate the x-axis. One ITO backside and frontside scan, and the sample 388 backside and frontside scan. The 388 backside scan is recorded with a 10mV sensitivity on the lock-in amp, while the others are recorded at 100mV sensitivity. Front and backside scans refers to reflected and transmitted scattering respectively.

As discussed before, the sample OMRL388 was abnormal, from a device point of view, as the transmittance was too high, see figure 33. As the device structure is supposed to be used in photovoltaic devices, the transmittance should be close to zero for the final device to be efficient. The absorbing layer was obviously not thick enough here. If viewing the four scans in figures 32 -34, the ITO reflected scattering run starts off at zero scattering intensity, with only a very small, but rising, addition to the intensity throughout the run. This is because the ITO film is very smooth and therefore do not scatter. At about 176000 steps, the detector starts moving into the laser beam path, and a cut-off occurs, best viewed in figure 33. The ITO transmission scan behaves similarly, but with a small contribution to the overall intensity that comes from bulk scattering. At about 173000 steps it starts registering the specularly reflected beam, which saturates the detector just after. The 388 reflected scan is in figure 33 fitted to the ITO reflected scattering cut-off, as it also shows signs to move into that area. This is done while; although the transmitted 388 scan would be enough to show the 0 degree angle, the 90 degree angle is harder to find.



Fig 33 A magnification of the maximum of figure 32. The 0 degree azimuthal calibration peak at about 176000 steps. Again, front and backside scans refers to reflected and transmitted scattering respectively.

In figure 34, all four scans have been calibrated to fit the minima of the respective scans at about 24500 steps. Following the calibration discussion above, the 388 transmission scan maximum, at 0 azimuthal angle or 176000 steps, should then be at exactly 90 degrees from the combined minimum, at 90 degrees azimuthal angle or 24500 steps. -The two scans, ITO and 388, actually consist of a forward scan, as the driver pushes the scanner forwards, and a

second, return scan as it is pulled back again to the starting position. This enables both the mean value of the two to be used, and also to measure the slack of the driver gears.



Fig 34 A magnification of the minimum of figure 32. It shows the region where the correlation information was collected, ie. the V shape at about 24500 steps.

Sample	Run1 forward/steps	Run1 return/steps	Run2 forward/steps	Run2 return/steps
90 degrees	24450	374350	24500	347950
0 degrees	174950	175150	197500	196500

Table 2 Min/max intensity steps at sampling OMRL 388 CdS/CdTe samples.

As shown in the table 2, in which two 388 runs are tabulated, the run values differ somewhat, but the deviation is not large, considering a full scan is 370 200 stepper motor steps for 180 degrees of angular translation. The intensity maxima values differ at most 850 steps from the mean, or about 2‰ on the total. The mean of the four runs is 150600 steps for 90 degrees, and the geometrical equation 53 can thus be completed:

$$acos(\frac{0.2-0.1(\sqrt{0.0325}-0.05)\frac{s-24500}{150600}-(\sqrt{0.0325}-0.05)^2\frac{(s-24500)^2}{(150600)^2}}{\sqrt{0.0005}})$$
$$-atan(\frac{1}{2})=\theta$$

Equation 54)

where s is the linear translation in stepper motor steps. The resulting 90 degree θ -axis can be seen in figure 35. At the end of this ARS section, the scattering intensity will also be related, through AFM measurements, to the real roughness.

4.1.3 Scattering scans.

Eighteen different samples were characterised with the angle resolved scatterometer. The high intensity 1.5 W laser line at $\lambda = 514.5$ (green) of a 5W Spectra-Physic Ar⁺ laser was used. The samples can be divided roughly into three different groups. The two substrates, one glass and one ITO on glass, were grouped together. Seven CdTe/CdS/ITO/glass samples were grouped together to a low temperature group (omrl366 - omrl395), as they were grown at a relatively low temperature, 320°C. The last nine samples (omrl423 omrl434) were grown at 350°C, a somewhat higher, but growth-wise crucially different temperature, and are therefore in a group of their own. An increase of roughness in the high temperature samples would be expected, considering no other significant variation in growth conditions. The substrate group is only included to find the characteristics of the scattering from a low scattering sample. Three typical scattering plots can be seen in figure 35, where OMRL366 is an example of a low temperature, and OMRL434 an example of a high temperature sample. The glass sample was included as an example of a non-scatterer.



Fig 35 An AR scattering distribution which shows the scattering over 90 degrees for three different surfaces. OMRL434 is a very rough surface (63.28nm), with OMRL366 being medium (20.72nm), with a smooth glass surface as a contrast.

Figure 36 show the eight low temperature scatterers, including the OMRL366 (O366) from figure 35. It is easy to distinguish the rise in scattering, which is the integral under the respective graphs, as the sample number increases. This is consistent with AFM roughness measurements on the samples, which show the same tendency. In the figures 36 and 37, all values below 2 degrees azimuthal have been removed, as the intensity closer to the azimuthal is the cut-off intensity caused by the detector moving in front of the laser. Compared to figure 37, with the high temperature samples, the scattering is lower. This would also be expected. Again, it is consistent with AFM roughness measurements on the samples. The samples OMRL 423, glass and ITO have been included in both figures, as these gives an indication of the relation between the intensity axis of the two figures. The intensity of the Ar⁺laser may not be totally stable, as small changes in alignment and the position of the shutters can cause large differences in output.



Fig 36 AR scattering graph of the low temperature samples. The data $> 0^{\circ}$ and $< 2^{\circ}$ have been removed, the data from this section are disturbed by the instrument geometry. Sample O423 is included in both this figure and in figure 37, and can be used to match the two graphs.



Fig 37 AR scattering plot for the high temperature samples. The data $> 0^{\circ}$ and $< 2^{\circ}$ have been removed, as the data from this section are disturbed by the instrument geometry. To better follow the changes in intensity (due to changes in laser intensity), the sample OMRL423 is included both here and in figure 36.

Figure 37 also shows the inversion of the distributions, as discussed in the theory section 2.3.1. The inversion occurs at about 97nm roughness, which is the AFM (rms) roughness measured for the sample OMRL430. The integral under any one of the distributions in figure 36 or 37, squared and multiplied with π , is by definition the total integrated scattering [TIS]. With the logarithm of the intensity as a function of the square of the cosine of the azimuthal angle; Le Bosse's predictions^{3,4} seems very likely. As explained in section 2.3.2, Le Bosse suggests the Rayleigh model cannot properly predict the curvature of the diagrams in figures 36 or 37 for higher scatterers.



Fig 38 Low temperature scatterers from figure 36, where the data have been treated according to the Rayleigh relation. The Glass and ITO sample distributions have been put into both figures as a way of calibrating the distributions.

For high azimuthal angles (close to parallel to the sample), figure 38 shows it is definitely not linear. Linearity should be present for all Rayleigh scatterers in these two figures. This is obviously not true here, but at low angles (close to the normal of the sample) it is.



Fig 39 A magnification of the figure 38 region for low azimuthal angles. Figure 41 contains the high temperature samples.

For the high temperature samples in figure 40, the same reasoning apply as a shift towards rougher surfaces can be seen: High azimuthal angles are not linear. Concentrating on the features in the linear region $\cos^2\theta > -0.6$ is one solution, as the interesting region for monitoring is, as specified in the project proposal, only a few degrees off-normal to the sample for *in situ* measurements. A look into the figures 39 and 41 clearly shows linearity. According to the Rayleigh model, the slope of the line now become an indirect measurement of the total scattering [TIS] and therefore also the surface roughness, see section 2.3.3.

In the linear region of the scattering plots, the root mean square roughness, σ , of the samples are indirectly extractable as the slope of the line. This with the use of AFM/scattering correlation of roughness values. The process will be further explained in section 4.1.5. Studying actual height distributions of the samples in an atomic force microscope, and correlating this to the regressions in the linear region will therefore enable us with a simple and powerful tool for

monitoring of surface roughness: the rougher a surface is, the lower is the





Fig 40 High temperature scatterers from figure 37, where the data has been treated according to the Rayleigh relation. The Glass and ITO sample distributions have been put into both figures as a way of calibrating the distributions.



Fig 41 A magnification of the figure 40 region for low azimuthal angles. Figure 39 contains the high temperature samples.

Low t	-	-	0366	<i>0367</i>	0381	0386	0388	<i>O391</i>	0395
Slope	-	2 	2.300	1.090	0.738	0.656	0.789	0.568	0.667
High t	<i>O423</i>	<i>O</i> 425	<i>O</i> 427	0428	0429	<i>O430</i>	<i>O</i> 431	0433	0434
Slope	0.353	0.557	0.144	0.698	0.128	0.088	0.215	-0.100	-0.140

Table 3 Slopes of the Rayleigh treated AR scattering distributions.

In particular, the slopes for all samples are tabulated in table 3. These measurements have been carried out *ex situ*, and the former discussion has concerned only growth on top of an almost amorphous substrate. This is why the distributions look so smooth and almost featureless. The distributions hold several hundred data points, so deviations from the perfect distribution that seem to be noise are actually features of the distribution itself.

4.1.4 Orientation effects in AR scattering.



Fig 42 AR scattering plot of orientated CdTe on single crystal Si(001). Visible are several orientations, where the intensity is either larger or lower than expected. The scatterometer has here been rebuilt to remove the 2 degree problem of the original design.

For a set of CdTe growth runs using instead single crystal Si(001) substrate wafers, the distribution looks radically different. It is a highly oriented substrate, and surface orientation effects of the film are clearly visible, as in figure 42. The sample series is the same series that was presented in the dual wavelength interferometry section 4.2.2. The AR scatterometer has also been rebuilt to remove the problem with the 2° cut-off coming from the detector moving in front of the laser beam. This was solved by rotating the detector 90 degrees in comparison with what is described in the design section, and instead letting a beamsplitter move into the beam path.

The *ex situ* ARS data show a difference in intensity at several angles, due to developed crystal plane reflections, i.e. faceting. This contrasts with the smooth distribution of figures 35-37, and the section 4.1.3 discussion on linearity in certain regions would not be applicable.

4.1.5 AFM measurements and ARS correlation.

The OMRL samples presented in section 4.1.1 were analysed in a Digital Instruments Nanoscope III atomic force microscope[AFM]. According to the discussion in section 4.1.3, where the rougher surfaces have a lower slope in the $cos^2\theta$ plot, sample OMRL434 would be expected to have the roughest surface, followed by all other samples in the order suggested in section 4.1.3, until the OMRL366 sample.

The samples were imaged, and figure 43 shows AFM roughness image of the OMRL430 sample, shown as an example. The mean value over a 10μ m×10 μ m area was used. Figure 44 instead shows grain size measurements on the same sample, analysed in order to investigate the relationship between roughness and size of the grain. Interestingly, the grain size / roughness ratio is pretty much constant for all of the samples that has been investigated, and is generally about 0.3 (see also table 8 for the CB# sample series).



5.0

2.5

n

10.0 µм

Img. Raw mean 12.253 nm Img. Rms (Rq) 96.826 nm

Mean roughness (Ra) Max height (Rmax)

Surface area Box x dimension Box y dimension

622.56 NM Box Statistics

Імд. Ямах

Z range Mean RMS (Rg)

0208.053

2.5

5.0

7.5

Peak off Area off Summit off Zero Cross off Fig 43 An AFM image of sample OMRL430, showing the computed (rms) roughness data at 96.8nm.



Image Threshold hist. Grain size hist Execute Cursor Erode Dilate Zoom Grain Size Analysis

0208.053

Normal Image Bnd. Grains off Norm. Threshold Box Cursor

The last AFM image, in figure 45, show the same sample at an angle, which better shows the structure of the surface. The 430 sample was used as an example, because this is where the inversion of the distributions, discussed in section 4.2.3, occur.



Fig 45 The same 430 sample as in figures 43 and 44, this time shown at a 35 degree virtual angle.

Low t	Glass	ITO	0366	<i>O367</i>	<i>0381</i>	<i>O386</i>	<i>O388</i>	<i>0391</i>	<i>O395</i>
Slope	-	-	2.300	1.090	0.738	0.656	0.789	0.568	0.667
σ/nm	*	7.873	20.718	*	26.726	*	28.360	*	*
High t	0423	<i>O</i> 425	0427	0428	0429	<i>O</i> 430	<i>O431</i>	<i>O433</i>	<i>O</i> 434
Slope	0.353	0.557	0.144	0.698	0.128	0.088	0.215	-0.100	-0.140
σ/nm	63.280	58.791	*	*	*	96.826	*	*	117.17

Table 4 AR scattering slopes and roughness (σ) of the OMRL sample series.

*The AFM analysis for these samples have not been made.

Out of the AFM height distribution, the corresponding AFM root mean square (rms) roughness was calculated. It is tabulated in table 4, along with the AR scattering slopes taken from section 4.2.3. As discussed above, the relative roughening should be dependent on the relative slope of the distribution. The plot of the slope calculated (scattered) set, verses the AFM (Real) RMS set is shown in figure 46.



Fig 46 A plot of the distribution slopes verses the AFM (rms) roughness. Sample indexes are noted in the figure.

Figure 46 does indicate a inversely proportional dependency, although some additional AFM imaging would probably be necessary to do. Note that this would only be an experimental fit, as no modelling has been done. We have already seen that the behaviour of the scattering distribution is not satisfactory explained by the Rayleigh model, except in special cases. I.e. for lower scattering samples, with a roughness of under about 23nm.

The rotation volume around the intensity axis of the I/ θ distributions is, as already explained, the TIS, and figure 47 show the relationship between the calculated TIS and the measured AFM (rms) surface roughness. The figure show the linear fit of the dataset, that would be expected when viewing the Ohlidal/Bennett/ Porteus/Carniglia equations.



Fig 47 Correlation graph between the TIS and the surface roughness of the OMRL samples.

Obviously, this (ARS) could be used directly to monitor surface roughness. However, because of the very narrow angle to collect data, and because so much of this angular range is taken up by the specularly reflected laser light, this is unfortunately not possible. Instead, a novel approach has to be made. Because of the above, a look into the properties of the specularly reflected light has to be made. As already discussed, the laser light distributions are well known. Any disturbance to the Gaussian distributions, as a result of interaction with samples which are having different surface properties, can therefore be seen as a measure of the roughness. Obviously, all effects arising from interference and absorbtion have to be accounted for and discarded. However, as the instrument is also to be used as an interferometer, a look into the interference signal has first to be made, in order to establish that the lateral growth monitoring signal will actually work according to interferometry theory.

4.2 Reflectance interferometry.

This section will present and discuss the results of several growth monitoring experiments, using the both the final design of the dual wavelength reflectance interferometer and an earlier single wavelength testbed. The section will start with the single wavelength results, and after that discuss the results of the dual wavelength instrument data.



Fig 48 Principal interferometer, consisting of lasers beamsplitters(BS), Photodiode (PD), charge coupled device array (CCD), and some lenses.

Figure 48 shows the principal geometry for the normal incidence instrument, where a laser is used to illuminate a sample through a beamsplitter. The laser is focused on a CCD array by means of two lenses, and the signal is used in two ways: integrated, to emulate the regular interferometer signal, but the beam profile is also measured, for reasons already discussed in section 2.3, roughness monitoring. The beam profile data will be discussed later, in section 4.4.

Note the lack of interference filters and analytical beamsplitters in the new design. This mean that, besides the lenses which focus the surface on the CCD

picture plane, only the central beamsplitter has to be transversed after the beam has interacted with the surface. This should enhance the signal-to-noise ratio further, as this can only reduce the amount of distortion on the light. In a standard dual wavelength interferometer, using analogue photodiodes, this would not be the case. In conjunction with this: the statistics for the integrated CCD signal and a photodiode signal should be identical, except for the time constant. As there is no fifo-buffer[first in, first out] on either the photodiode or the A/D converter, the analogue time constant is the same as the device speed, multiplied with the poll rate, 50Hz. The digital device has a fifo, which is released at every poll. The time constant is therefore 1/16 second.

4.2.1 Single wavelength experiments.

In order to utilise the materials knowledge gathered within the research group, established MOCVD recipes were used to grow the monitored solar cell devices. This meant there was a possibility of relating previous monitoring data from traditional single wavelength interferometry to the new data. In fact the laser from the current reactor setup was used in the experimental section, so the data should be consistent. However, the data from the current reactor setup was only intended as a growth monitor, not an instrument research monitor, which means no raw data was stored. Because of space concerns, with only a 200Mb hard drive on an i486 computer, instead only an averaged signal was saved. This is of course not an issue today, as the size of hard drives has grown substantially the last couple of years. However, the instrument was built a couple of years ago, and at that time hard-drive size was very much an issue.

NB. In between the first "old" set of data, which was used in the ARS measurements, and the second "new" set, used everywhere else, is over a year of reactor downtime, because of a move of the research group from NEWI,

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Wrexham, to University of Wales, Bangor. Some differences between the two datasets are likely to to be found because of this.

4.2.1.1 Material growth.

The materials were grown in a Thomas Swan (1 x 2") wafer MOCVD silica reactor cell. This to evaluate the general instrument design, before using valuable time on an Aixtron production reactorⁱ. The organometallics used in growing the Glass/ITO/CdS/CdTe structures were dimethylcadmium (DMCd), with the addition of diisopropyltellurium (DiPTe) or di-tertbutylsuphur (DtBS) depending on the layer composition. The device structures were grown at 320°C, regardless if the sulphide or the telluride was grown. The thickness and growth time were varied for each run. CdTe growth rate shows fitted value, as interpreted from the interferogram. Standard deviation of the growth rate was 0.09Å/s. This was calculated on a one-half oscillation subset of the total interferogram.

Table 5 Growth parameters of the single wavelength experiment.

No.	CdTe growth t /	Te:Cd OM/	CdTe growth rate/
	°C	molar ratio	Å/s
CB01	320	0.17	2.1

4.2.1.2 Single wavelength reflectance interferometry.

As stated before, these measurements have primarily been made in order to investigate scattering/film roughening. However, it is also possible to recover the reflectance signal concurrently. It would be advantageous to do this, especially as this also helps us verify that it is a true scattering signal we are recording. The reflectance photodiode signal is emulated by integrating the pixel response over all image pixels. Figure 49 shows a

i Actually, the fitting of the instrument on an Aixtron reactor was never done, as the company whose reactor was going to be used had difficulties. The project was therefore considered completed at a situation where a multi-wafer version would be considered likely to function properly.

typical image, where, looking down into the reactor, we can see the Newton's rings on the surface of the wafer, discussed in section 3.2. The focal plane is on the wafer surface.



Fig 49 Image taken of the Newton's ring pattern on the surface of the wafer. The intensity over the whole image is integrated to produce the interferogram signal.

Figure 50 shows the first successful attempt in recording single wavelength growth data. It shows the features expected from the discussion in the theory section 2.2.1. Recognisable are the effects of film growth: Oscillations, due to uniform film growth. The extinction of oscillations later on in the interferogram, due to absorbtion. Finally, the overall loss of intensity, due to scattering, is also distinguishable.

Figure 50 is similar to that of figure 51 in that the CdTe layer, was grown in as much as possible in the same way, excluding the change in temperature of 30°C. Corresponding intervals are, in figure 50, all growth after ~2600 seconds and, in figure 51, growth after ~6000 seconds. The first large half wave in figure 51 is CdS, grown directly onto the glass/ITO substrate. As seen, this layer grows at considerably lower rate.



Fig 50 First successful attempt to extract growth data from the combined interferometer/ profiler. The spike in the data at ~800 s is from turning the laser off to mark the zero point. Note also the irregular look of the first oscillation, which is due to the built-in autobrightness function of the CCD.



Fig 51 Interferogram of OMRL423 growth. The part after ~6000 seconds is the CdTe growth. This is a high scattering (rough) sample, and has also been used as such in the ARS measurements.

The growth run in figure 50 is a simple CdTe growth on top of a ITO coated glass wafer, sample CB01. When comparing the relevant intervals in the two figures, the growth rate can be read out of the diagrams to be about

50% lower in figure 50, compared to figure 51, (see table 8). Everything else being constant: laser wavelength, growth recipe, and optics, means the only variable here can be the growth rate. The relative growth rate is therefore immediately viewable from the numbers of oscillations / time. The growth rate looks reasonable, and calculates to 2.1 ± 0.09 Å/s, whereas the reported growth rate of the OMRL432 growth was 4.3Å/s.² The lower growth rate was most probably due to the lower temperature of the figure 50 growth, with 320°C instead of 350°C.

In the figure 50 diagram, the big dip in signal strength at ~800 seconds, is present because the laser was turned off in order to find the zero "dark" level intensity of the CCD. A certain broadening of the signal is also evident. This is not noise, but rather the effect of precession of the rotating wafer, and thus a small fluctuation in the intensity. In fact, this can be traced reliably using the current sampling rate of 15Hz and 1 (substrate) revolution per second. This could easily be used as a rotation monitor for the single wafer reactor, even though this was not done here, as it was beyond the scope of this project. Before and during the first oscillation quarter-wave turn, there is a disturbance, which was due to a hardware auto-brightness function in the CCD-array. This feature has been disabled in later measurements. The appearance of figure 50 is otherwise what would be expected from the theory.

Figure 51 data is an averaged signal over 50 samplings/data-point, while the figure 50 signal is untreated (raw), which accounts for the cleaner look of figure 51, in spite of the above noise level discussion. The comparison with device structure glass/ITO/CdS/CdTe data, grown previously shows that the intensity used is indeed the reflectance; compare figures 50 and 51.

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Initial concern about the possible non-linearity of the CCD-array pixel

response was therefore proven to be unfounded.

Laser wavelength/ nm	$n_{\rm S} ({\rm ITO})^2$	$n_f (CdTe)^5$	$k_f (CdTe)^5$	
633(Ne/He)	1.75	3.04	0.253	

Table 6 Optical constants of glass/ITO substrate, and polycrystalline CdTe.

Only a few runs were made with this setup, before the dual wavelength instrument was designed. This was because the design was proven, and single wavelength measurements will not solve the fundamental problem of unambiguous monitoring of roughness. The optical constants of ITO² and CdTe⁵ that were used initially in the growth rate calculations are shown in table 6.



Fig 52 Figure 50 recalculated, with theoretical values (blue), the experimental values (red). A scattering residual line (black) showing no oscillation is present after subtracting the theoretical from experimental.

The determination of film thickness was made using the modified virtual interface model, as described by Irvine et al⁶ and discussed in section 2.2. The resulting interferogram is shown in figure 52. The black line in the

diagram is the residual from the subtraction of the experimental signal from the theoretical. Hence, it is the scattered intensity.

Interestingly, the theoretical values fit the experimental values quite reasonably, but for the k_f , which has to be about twice the expected value, here $k_f = 0.5$. Figure 52 also show the danger with using a linear scattering model to solve the scattering problem in the Irvine equations, because clearly the scattering contribution is not linear. The look of the first 250 seconds has been explained above: the first oscillation peak is distorted from auto-brightness switching. During the experiments, the same effect has also been seen when using the instrument without turning on the laser, and with strong lighting coming from lighting tubes. The CCD seem to be sensitive enough to pick up the switching in the tubes, even if this is not noticeable to the naked eye.

4.2.2 Dual wavelength experiments.

The measurement of an interferogram at two different wavelengths in conjunction with scattering data, should enable the different intensities [R, A and S] of the reflected beam to be correctly distinguished, as discussed in the theory section 2.3.2. It should enable detection of drift in the interferogram due to scattering, as the two different wavelengths will respond simultaneously and according to equation 36. The effect will be of different magnitude for the two different wavelengths, as discussed in the theory section, and will be inversely proportional to the square of the wavelength. The total scattering, and therefore changes in overall intensity, can now be determined. With this information, the amount of scattering at any given time will be available, and can be introduced as an extra term in the Irvine equations.

The scattering drift in a system has been detectable even before, because the interferogram has started to drift outside the parameters decided by the optical properties of the material grown. It has never been possible to accurately determine the magnitude of this drift, though, as there have never been any way to reference a single wavelength in real time. The two wavelengths used here, 532nm (green) and 655nm (red), were matched to the CCD detection electronics, as outlined in section 3.2.1.

4.2.2.1 Material growth.

Similarly to the single wavelength experiments, the organometallics used in the MOCVD experiments were DMCd, with the addition of DiPTe, to deposit a CdTe film, grown on top of single crystal Si(001)-wafers. As these measurements were conducted in order to understand the scattering from the surface, it was concluded that a well defined substrate, like the zeroscattering, zero-transmittance Si(001) wafers would be better suited. The Cd:Te OM-molar ratio was 4:3 (2.0×10^{-4} and 1.5×10^{-4} Atm partial pressure respectively, in 1 Atm H₂ matrix), and the temperature was held at 350°C. The only variation was the different growth times, see discussion surrounding figure 53 below.

4.2.2.2 Dual wavelength reflectance interferometry.

The dual wavelength interferogram of the CB02 growth, shown in figure 53, was achieved by integrating the pixel data of CCD images, separated into its base colours, as discussed in the design section. The onset of growth can be seen at about 800 seconds, with growth oscillations being present until about 1500 seconds. After that, the underlying interface has been hidden by the absorption in the film. There was significant intensity loss due to scattering, because the film was roughening. The effect gradually

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increases, thus reducing the specularly reflected intensity. After about 2300 seconds it scatters all the incoming light. The growth was allowed to continue until 4600 seconds, and the AFM roughness measurements made on this sample, in order to correlate the roughness to the scattering, will therefore show on a larger roughness, than can be explained by these measurements. Figure 53 only shows the data up to 2500 seconds.



Fig 53 Dual wavelength interferogram of the CB02 sample, where both wavelengths have been recorded simultaneously on a single CCD-array. Growth times of all CB samples are marked in the diagram, and indexed on the lower time-axis.

Figure 53 not only shows the growth interferogram of the CB02 sample, but all other CB samples grown in the experiment series. Marked along the time-axis in the figure, are the respective times where the growth run was halted for the individual CB samples. As the growth parameters were identical for all the samples, the interferograms overlapped until the runs were stopped. Following AFM measurements on all these samples, "snapshots" of the surface could be taken at several points during growth. Table 8 on page 112 shows more of the growth results. No averaging was necessary for the interferogram, but a certain modulation, due to substrate precession is present. This can easily be removed by an averaging function, or could even be used as a rotation monitoring parameter. A certain mix of the two colours will also occur due to the broadness of the spectral response of the CCD detector shown in section 3. After iteration, a set of mixing factors were selected as 0.075, for green \rightarrow red and 0.115 for red \rightarrow green:

$$I_{green} = I_{green}^{meas} + F_{mix}(R \to G) I_{red}^{meas}$$
Equation 55)
$$I_{red} = I_{red}^{meas} + F_{mix}(G \to R) I_{green}^{meas}$$
Equation 56)

The determination of film thickness was, as before, made using the modified virtual interface model as explained by Irvine et al⁶. It was made by separating the dual wavelength interferogram into two single wavelength, which is discussed further down in this section. The optical constants of single crystal Si⁷, and polycrystalline CdTe⁸, used initially at the two wavelengths can be seen in table 7.

Table 7 Optical constants of single crystal Si, and polycrystalline CdTe.

Laser λ / nm	n_S (Si) ⁷	$n_f (CdTe)^8$	$k_f (CdTe)^8$	
532	4.152	2.867	0.354	
655	3.844	2.743	0.279	

As discussed in the theory section 2.3.2, the total incident intensity can be written as the sum of the reflected, absorbed and scattered light. The transmitted intensity is zero, because of the Silicon wafers. It follows that the total integrated scattering (TIS) intensity will be the difference between a theoretical interferogram, using the values in table 7 as variables in the Irvine equations, and the experimental data. Furthermore, as the initial substrate is a polished single crystal Si wafer, it can be regarded as truly non-scattering. This has been confirmed in the AFM study, where the surface roughness was measured to 0.4nm (rms). This gives a zero point, $I_S = 0$, for scaling the interferogram.

The two single wavelength interferograms, derived from the dual wavelength interferogram, are thus connected through several parameters:

- All data have been recorded simultaneously by the same detection electronics, which means the time is exactly the same for both data sets.
- As already discussed, scattering is zero for both data sets at onset of growth. The measured interferograms can therefore be set to the correct reflectance at t = 0, through the Irvine equations.
- Scattering intensity is related through the Rayleigh relation for both wavelengths. The scattering ratio between the two wavelengths is always known.
- The two wavelengths will show the same growth rate, within errors.

When detecting the two colours in the same detector, another new feature could be seen. The two colours do not respond at the same time at the onset of growth. A certain lag in the red wavelength is evident. There has always been a certain lag in the onset of growth, between when the OMs are introduced into the reactor cell, and the interferogram trace shows a deviation from the straight line. This has always been attributed the nucleation delay, but at least a portion of this is obviously due to the response time of the interferometer with respect to the wavelength. On the green wavelength the onset of growth is shifted about 15 seconds earlier. With a third, blue, wavelength the zero time of the onset could be

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pinpointed. This would certainly be important for the growth of finer device structures, like vertical cavity emitting lasers [VCEL], where the formation of quantum wells with very strict tolerance must be achieved for the devices to work as expected. This response time dependence has been detectable throughout the dual wavelength experiments. Assuming no features below the monitoring wavelength are detectable, which is normally the case, it would mean that the largest surface features reached a size of about 532nm 15 seconds before they reached about 655nm. If then assuming a linear dependency (this is probably not true, but a couple more wavelengths would be needed in order to model the behaviour, and certainly at least three), the real onset of growth would be about 80 seconds earlier than detected by the red wavelength. With a stable growth rate of 2.1Å/second, like in the single wavelength interferogram of section 4.2.1.2, we would already have grown 168Å of material before it is detected.

The resulting two single wavelength diagrams, made up from the original dual wavelength shows the theoretical and experimental data for the green and red wavelengths, figure 54a and 54b respectively. Notice the start of the interferogram in figure 54b, showing the unresponsive nature of the wavelength in the first few seconds discussed above. Gradually, over the first minute, the response will increase to normal. Scattering standard deviation, using a 20 point running mean as the reference curve, is 0.0005 normalised Scattering (= Reflectance) units.



Fig 54 a Theoretical and experimental data for the green wavelength. Scattering plot (black) is experimental value subtracted from the theoretical. Growth rate 58nm/sec. Standard deviation 0.06 nm/sec.



Fig 54 b Theoretical and experimental data for the red wavelength. Again, the scattered intensity is shown in black. Growth rate 58nm/sec. Standard deviation 0.09 nm/sec.

As a starting point, the optical constants were taken from literature, see table 7. This to satisfy the Irvine equations. If the optical constants are applicable to this instrument, then no oscillation should be visible in the scattering plot in figure 54a and 54b. It is likely that these initial constants will not be exactly the same, because of differences in the experimental setup, and also differences in the composition and grainsize of the film. In the current approach, the optical constants n_s , n_f and k_f therefore have to be minimised by iteration, fitted through minimising the scattering error, until the two wavelengths datasets correspond. A weak oscillation is actually present even after fitting, especially in figure 54b, but with further work, where the optical constants can be refined more accurately, this should disappear. The optical constants are all dependent on the wavelength, which effectively doubles the number of variables.

The two wavelengths different response times also have be taken into account, which mean the zero time has to be set differently for the two wavelengths. The total number of variables to iterate in order to minimise the scattering for a fit is thus 8 (2×4). The iteration steps are explained more in depth in section 4.3, as the same work has been done there, with only the addition of relating the overall scattering intensity to AFM measurements of the roughness.

The dual wavelength interferometry system has proven to work just as well as a conventional single wavelength analogue (photodiode) system. This new setup has also been able to picked up on the issue of detection lag in an interferometer, due to different wavelengths being used, which is useful when wanting to grow films with a very tight tolerance. With the inclusion of a second wavelength into the instrument, there is now a way of cross-correlating the laser wavelengths with respect to the scattering, in order to monitor the surface roughness in real time.

4.3 Dual wavelength scattering cross-correlation.

The use of one frequency as a corroborator for the other, in order to determine the scattering, can be achieved by the combination of the Irvine equations, the Ohlidal/Bennett/Porteus approximation, and the Carniglia equation with the manipulations of the scattered interferometry signal, see section 2.3.2.

$$I_{\lambda_n}^0 = I_{\lambda_n}^R + I_{\lambda_n}^A + I_{\lambda_n}^S$$
 Equation 57)

Where:

$$R_{\lambda_n} = I_{\lambda_n}^R + I_{\lambda_n}^A$$
 Equation 58)

$$(I_{\lambda_1}^R + I_{\lambda_1}^A) = 1 - \frac{\lambda_2^2}{\lambda_1^2} (1 - (I_{\lambda_2}^R + I_{\lambda_2}^A))$$
 Equation 59)

Or:

$$\lambda_1^2(1-R_1) = \lambda_2^2(1-R_2)$$
 Equation 60)

$$R_{\lambda_{n}} = \frac{r_{1}^{2} + r_{2}^{2} e^{-\alpha_{n}d} + 2r_{1}r_{2}\cos\Delta e^{-\alpha_{n}d}}{1 + r_{1}^{2}r_{2}^{2}e^{-\alpha_{n}d} + 2r_{1}r_{2}\cos\Delta e^{-\alpha_{n}d}}$$
Equation 61)

$$R_{\lambda_n}^{meas} = R_{\lambda_0} \exp\left[\frac{4\pi\sigma}{\lambda_n}\right]^2$$
 Equation 62)

Where $R_{\lambda\theta}$ is the reflectance from a non-scattering sample, and σ is the roughness. In this case, with the use of a perfectly flat, single crystal Si(001) wafer $R_{\lambda\theta}$ should be approximately equal both the measured and theoretical values separately at t = 0. The theoretical value being calculated from equation 61.

$$R_{\lambda_{n}}^{meas} = \frac{r_{1}^{2} + r_{2}^{2} e^{-\alpha_{n}d} + 2r_{1}r_{2}\cos\Delta e^{-\alpha_{n}d}}{1 + r_{1}^{2}r_{2}^{2}e^{-\alpha_{n}d} + 2r_{1}r_{2}\cos\Delta e^{-\alpha_{n}d}}e^{-\frac{4\pi\sigma^{2}}{\lambda_{n}}}$$
Equation 63)

For non-smooth substrates, the reflectance has to be calculated in equation 62 from the AFM roughness.

Combining this over both wavelengths therefore gives the equation system:
$$R = \lambda_{1}^{2} \left(1 - \frac{r_{11}^{2} + r_{21}^{2} e^{-\alpha_{1}d} + 2r_{11}r_{21}\cos\Delta e^{-\alpha_{1}d}}{1 + r_{11}^{2}r_{21}^{2} e^{-\alpha_{1}d} + 2r_{11}r_{21}\cos\Delta e^{-\alpha_{1}d}} e^{-\frac{4\pi\sigma^{2}}{\lambda_{1}}}\right)$$

$$R = \lambda_{2}^{2} \left(1 - \frac{r_{12}^{2} + r_{22}^{2} e^{-\alpha_{2}d} + 2r_{12}r_{22}\cos\Delta e^{-\alpha_{2}d}}{1 + r_{12}^{2}r_{22}^{2} e^{-\alpha_{2}d} + 2r_{12}r_{22}\cos\Delta e^{-\alpha_{2}d}} e^{-\frac{4\pi\sigma^{2}}{\lambda_{2}}}\right)$$
Equation 64)

All intensities must of course still be normalised. All scaling between the two different frequencies has to be done at t = 0, when the theoretical and measured values, as already discussed, coincide. The *r* optical constants has been re-indexed because the optical constants are all wavelength-dependent.



Fig 55 CB11 growth run dual wavelength interferogram. Diagram also showing the green and red theoretical curves.

The CB11 growth run, seen in figure 55, is basically the same run as the CB02 run discussed earlier, compare figures 53 and 55. Using the data from the above CB11 growth run, and also the optical constants out of table 7, a start to look at the oscillation frequencies for both wavelengths as in figure 56 can be made.



Fig 56 Correlated oscillation (only) of both wavelengths. The two intensities has been scaled to fulfil the initial requirements of the combined equations 61 and 62.

The theoretical oscillations in figure 56 were fitted to the maxima/minima of the measured oscillations. At this point, only the minimum and maximum of the oscillations were of interest, so to enhance these, k_f was set to zero. The thickness d and film refractive index n_f could here be adjusted by iteration for best fit to theory. This gives a good value for the film thickness, which is needed when trying to fit the absorption, as a closer value for the film extinction coefficient is iterated. With the values for d and n_f determined like in the above discussion, the other parameters, k_f and n_s , can be extracted by instead looking at the asymptotes to the interferograms. The absorbed intensity was thus fitted against theory as in figure 55. In doing this, it was noticed the fitted substrate optical constants were closer to the Silica reference than to the Silicon. It was confirmed that the substrate had a thick oxide layer ontop, which made it more proper to use the refractive index of silica.

As can be seen in figure 56, a common feature in all interferograms is, again, that the growth oscillations start at different times for the two different

wavelengths, where typically the lower wavelength has a faster response time. This has been already been discussed in section 4.2.2.2, but it shows this is a reoccurring phenomena. The fit for the theoretical values is also generally bad at the onset of growth for the red wavelength, and neither the phase or growth rate can be considered representative at that time. A way of rationalising this would be to investigate if the growth rate has changed during this badly fitted interval. However, as one would expect this to also be visible in the lower wavelength, which it is clearly not, this is highly unlikely.



Fig 57 The same manipulations were made to the combined absorbed and scattered intensity as outlined in equation 63.

With the four parameters assigned, the two frequencies could be scaled against each other in accordance to the Rayleigh equations. This is done in figure 57, with the plot line "diff_green" being the difference between the theoretical and measured green signal, and "diff_red" the corresponding red values. The "diff_from_green" plot line, is instead the scaled green scattering signal, which slope should be equal to the "diff_red" plot line. The resulting combined

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scattering plot would, using the green wavelength values for the lower part t < 400s and the red for t > 400 s, have the properties necessary to be calibrated to AFM measurements, and cleanly monitor roughness.

The oscillation and absorbed/scattering intensities were in this way correlated on both frequencies, and a roughness index was returned. The roughness index is the uncorrelated and untreated roughness value, and is an arbitrary number, but linearly dependent to the real scattering. This was, as in the AR scattering section, correlated against AFM measurements.

4.3.1 AFM correlation.

The six CdTe/Si samples CB02-CB07 and one blank Si substrate were studied in a National Instruments Nanoscope III, using standard contact mode AFM. The samples showed an increase in roughness and grain size with growth time, as shown in figure 53. These measurements forms the calibration dataset for the combined instrument, to which the roughness data is correlated. Figure 58 show three of the AFM images taken of the surfaces, which clearly show the difference in surface roughness of the different samples.

Sample	Growth time/ min	AFM roughness/ nm	AFM grain size/ nm	Roughness / grain size ratio	CdTe thickness / nm	Estimated TI Scattering
Si substr.	-	0.7	-	-	19 19	-
CB07	5	5.7	17.4	0.33	100	0.045
CB06	8	29.3	96.4	0.30	250	0.118
CB05	12	45	140.3	0.32	400	0.204
CB04	20	53.6	195	0.27	700	0.373
CB03	29	69	222.5	0.31	1000	0.414
CB02	65	154.9	666.7	0.23	3930	0.421*

Table	8 S	urface	AFM	and	growth	data
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*The loss of intensity in the later part of the interferogram will make the measurement of points after ~1000nm film thickness uncertain.



Fig 58 Three of the six AFM images ranging in roughness from smoothest to roughest from left top to right bottom. The images are all $10 \cdot 10 \cdot 1 \mu m (x \cdot y \cdot z)$ in size. Time of growth was 5, 20 and 78 minutes for the samples CB07, CB04 and CB02 respectively. The films were grown on a perfectly smooth Si(001) surface.

By looking at the data in table 8, we can see that the grain sizes, AFM roughness and also scattering look to grow consistently and close to linearly with time. The total integrated scattering (TIS) values were taken from the calculated interferogram of the green wavelength as discussed in the dual wavelength interferometry section. They were also normalised against the indecent intensity. In the table, the TIS is the intensity lost due to scattering in the interferometer measurements. As the incident intensities of the lasers are relatively small, all reflected intensity will eventually transform into scattering, as the roughness grows. As this will happen at angles undetectable by the instrument, there will be a net loss of intensity. This is evident in the last measurement of the scattering, where the linear trend was lost.



Fig 59 The CB11 sample scattering-time dependency as outlined in equation 62.

Figure 59 show the growth run CB11 treated as discussed in the former section, and recalculated as outlined in equation 62. The two frequencies, red and green, can be converted into each other by equation 59. It has been done in the figure, and the result was the two conversion graphs, "green to red" and "red to green". Of particular interest, because of the unclear first portion of the red pixel diagram (t < 200), is the diagram "green to red". The combination of the lower portions (0 s < t < 350 s) of the this pixel diagram and the upper portions of the red diagram gives a clear combined graph. This was used as the scattering data to calculate the in situ part of figure 60. Combining the findings of the AFM study in table 8 with this data, gives the AFM roughness diagram of figure 60. The figure shows the AFM calibration points used for scattering the Roughness axis, together with the CB11 scattering data. The correlation of the CB sample series with respect to the combined interferometry data and AFM data showed a reasonable degree of accordance for that small a data set, with an relative error of about 20%. Here, only the green wavelength was used in the lower part of the diagram, but in order to do the correlation in real time

both wavelengths can be used for better accuracy. Note that the growth rate of the CB11 run are 62.5% of the CB02-CB07 AFM runs, which mean the table 8 time values were recalculated. The standard deviation of the scattering data, using a 20 point running mean as the reference value, was 0.19nm.



Fig 60 The CB11 sample roughness-time dependency scaled against AFM values. Error bars shows a 20% relative error. Last AFM roughness point (t=1750s) was recorded after the instrument cut-off, which can be seen at t > 1250s.

There is still the issue of the first couple of minutes, where the response time of different wavelengths is important. A faster response would be achieved by instead using a blue wavelength, alternatively using all three wavelengths: red, green and blue. The red wavelength also showed to be very hard to fit to the equations. As the green wavelength could be easily fitted, the conclusion would be that the red laser was the issue. The laser power supply has also been replaced after these experiments, with better reproducibility as a result. The technique of cross-correlating the lasers to find the absolute value for the scattering, and therefore the roughness does indeed work, as shown in the above text. However, at lower scattering values the technique has shown on non-responsiveness. As these lower values of scattering correspond to the lower values of roughness in a film or interface, that industry generally require, usually under about 10 nm, this is not enough. The use of a third wavelength, matched to the last blue pixel colour of the CCD could be an answer to this. In the next section, a novel approach, laser light profiling [LLP], which could manage to achieve this using only the two wavelengths, will be presented.

4.4 Laser light profiling.

What follows in this section, is a discussion on roughness parameter extraction with the use of laser light profiles of the surface reflection. The text discuss mainly how the reflected laser dot relates to scattering and therefore also roughness.

4.4.1 Modelling the laser light profiler.

The modelling of the laser light profile, and the interaction with the surface is straight forward. Assuming the laser beam has a Gaussian profile, and assuming any loss due to scattering can be deduced from the ARS profiles, discussed in section 4.1, the change in the Gaussian distribution can be determined by a combination of the Gaussian distribution and the Yang approximation⁹:

$$I = I_0 e^{-kx^2} - f_s I_0 \frac{1}{\theta^3}$$
 Equation 65)

Where f_s is the factor that has to be multiplied to the incident intensity for correcting the measured intensity to the scattered ,and $\theta \propto x \Rightarrow \theta = j \cdot x$, where j is the slope of the conversion function. Varying the scattering factor should

then show the change of the laser profile dependent on the scattered intensity. Figure 61 show a theoretical case, where a Gaussian distribution is corrected with respect to this scattering factor.



Fig 61 Theoretical Gaussian distribution, showing the effect of scattering on the specular reflection. The scaling factor f_s has been varied from 10^6 to 0.01 in 2 magnitude increments, with the addition of the original Gaussian f(0).

The left hand side of the diagram, with the negative θ scale, the additive effects of the two terms of equation 65 are seen, as the scattering function term of equation 65 has the same (positive) sign. In this region, the asymptotes of this function are also seen, which makes this side of the distribution unlikely to represent the true scattering. On the positive θ scale, showing the corresponding subtractive effects, an area of highly curved distributions are shown, that bears striking resemblance to the distributions shown in the ARS section 4.1. Note that the incident intensity in this theoretical case is $I_0 = 1$, which means the functions are one magnitude apart. On the angular scale, the specular reflectance distribution is over about 1 degree, or $-0.5 < \theta < 0.5$, while the Yang approximation is valid over the first ±four degrees around the normal. Within this small angular interval, close to the normal of the sample, the function 65 can be approximated to a linear function. Linearising equation 65 gives:

$$-kx^{2} = \ln\left(\frac{I}{I_{0}} + \frac{f_{s}}{\theta^{3}}\right) = \ln\left(\frac{I}{I_{0}} + \frac{f_{s}}{cx^{3}}\right)$$
 Equation 66)

where $c = j^3$.



Fig 62 The combined distribution, this time linearised according to a simple Gaussian function. The scattering factor has been varied from 0 to 1, with the same increments of figure 61.

When linearising this, using a simple Gaussian function, and overlaying the scattering corrected equation 66 functions for several different scattering factors, a shift from the expected Gaussian linearity (seen in red in the graph) is observed, as the distributions go towards higher scattering. Figure 62 illustrates this. The shift is not linear, but rather logarithmic, which is what we would expect from equation 66. Note that each of the lines have one asymptote truncated (positive θ values in figure 61). The function is actually symmetric about the Gaussian line so as the function for negative θ values in figure 61 are horizontal asymptotes, so the positive θ function will form vertical asymptotes. This because of limitations of the spreadsheet software. Because the data is

somewhat scattered, and with only a limited set of counts, see figures 66 and 65, a good way of investigating this is statistically. By investigating the standard error of the real data from the theoretical linearised Gaussian, and using this as a measure for the change, relating this to the scattering factor f_s , an unambiguous measure of the scattering can be obtained. Then using the Ohlidal/Bennett/ Porteus/Carniglia equations on the scattered intensity, and correlating against the AFM (rms) roughness values, the surface roughness can be easily calculated. Figure 63 shows how the running sum of errors change with the scattering factor.



Fig 63 The linearised distributions standard deviation verses the scattering factor. The scattering factor has been varied as in figures 61 and 62.

There is also the possibility to make use of the ARS data of section 4.1.5. This would be an unmodelled approach, but this would of course be based on experimental data, which means it nevertheless would be highly useful. First, a look into how the Gaussian data changes with different rough films has to be made. It involves growing materials, monitoring this as before, but this time also capturing the CCD-images and analysing them.

4.4.2 Materials used.

The recipe of the CB11 run was the same as the one from the dual wavelength experiments, as presented in section 4.2.2.1. As before, the point of the run was to investigate the surface roughness, which meant using a non-scattering Si(001) substrate, with DMCd and DiPTe, to deposit the CdTe film

4.4.3 Beam profile measurement.

The LLP data are collected from analysing images as in figure 64. The images are analysed with respect to the red and green intensities, in order to determine the laser light distributions. Line scans on the images are made, which are just an integration of a one pixel wide line of the CCD image which are stepped forward one pixel at a time over the whole of the image. The largest diameter is found by assigning the largest diameter to the largest integral. This was made at 10 different angles over the image, to find the middle point, origin, of the distributions. This is set as the intersection of the different lines. Once set, the radially uniform Gaussian distribution around this point can be determined from the linearised plots.

Figure 64 shows an image, taken 16 minutes into a growth run of CdTe, grown onto a Si(001) wafer. The measurements were done by imaging the reflected laser beams of the instrument with a CCD-camera, and the image shows two clearly visible Gaussian distributions in green and red. The CCD-array required the laser beams to be diffused in order to achieve the same sensitivity as a photodiode, as the 8-bit pixels have not got sufficiently good statistics. More detecting pixels are therefore needed to overcome this. Hence, the two beams were dispersed to cover a surface area with a radius corresponding to about 200 pixels on the camera. Images were taken of the dot every 10 seconds during growth, and line scans were performed on all of them

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to find the pixel with the largest dot diameter. The timing interval was chosen somewhat arbitrarily, but evaluation of the programming code has shown that the calculations for a single frame take about 10 seconds on a 1GHz AMD Athlon machine. Hence this interval was used.



Fig 64 A typical image of the two laser spot distributions during growth of CdTe on Si.

Figure 66 shows an example of the two distributions, taken pre-growth on the Si surface. The figure shows the two Gaussian profiles from the laser beams, where the datasets have been plotted as the natural logarithm of the inverse intensity, versus the square of the pixel number from the spot centre. This would show the linearity that would be expected when considering equations 67 and 68 (for the time being; assuming a simple Gaussian profile);

$$I = I_0 e^{-kx^2} \Rightarrow$$
 Equation 67)
 $\ln \frac{I}{I_0} = -kx^2$ Equation 68)



Fig 65 Line scan of laser spot @ 600nm thickness or 51nm roughness (rms). As before, figure data has been linearised according to a Gaussian profile. Error 4%



Fig 66 Line scan of laser spot at 2 wavelengths. Snapshot of Si substrate surface pre-growth, in situ, showing the beam profile. Figure data has been linearised according to a Gaussian profile. Error 4%

A linear dependence should thus be present, as indeed it is in figure 66 (and also 65). The figures shows that the red wavelength has not been affected much by the increase in surface roughness during the 16 minutes of growth, while the green has. This is what would be expected, considering the difference in

wavelength. Gaussian constant (k) was, in the Silicon substrate case (figure 66), -2.85×10^{-6} and -9.78×10^{-6} for the red and green wavelengths respectively, and, for CdTe after 16 minutes of growth (figure 65), -2.85×10^{-6} and -7.40×10^{-6} respectively. The intercept for the red and green wavelength was 4.94 and 5.36 respectively (figure 66), and 4.90 and 4.67 respectively (figure 65).

The anticipated result here would be that the intercept is an abstraction of the specular reflectance, while the change in slope should be the change in the scattering This can be correlated to AFM roughness data. Looking at the estimated total integrated scattering [TIS] from the interferogram, section 4.2, where the scattering data was not separated, the TIS can now be independently measured.

Figures 67 and 68 show two corresponding diagrams, one interferogram and one Gaussian *k* diagram, of the CB11 CdTe on Si (001)growth. Figure 67 show the typical dual wavelength interferogram of a rapidly roughening surface, discussed already in section 4.3. The growth is a close replica of the CB02 sample, which, being one of the first successful monitored runs, had some artefacts in the dataset, see figure 53. Figure 68 shows the corresponding Gaussian laser light profile diagram, represented by the laser light Gaussian constant captured every 10 seconds. If these Gaussian constants can be said to represent the relative scattering value, these should, in some usable way, follow the calculated TIS of section 4.3. Figure 64 shows five reactor images and the corresponding line scans, taken at representative times at the CB11 run. When comparing the figure 68 data with these, it is obvious, though, that it is not the case that the scattering can be modelled on the gaussian constants alone, and that the model presented in section 4.4.1 is more representative. The Gaussian constant changes, but this is more due to the regression being altered by the

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trail-off of the data at the higher x^2 -numbers, as seen in figure 62. The trail-off, marked in figure 64 with blue, is due to the increasing effect of the scattering term of equation 43 and 44, with rougher surfaces. Note that the blue lines in the diagrams are not fitted to the equations, but only a visual aid to show the trail-off.



Fig 67 The dual wavelength interferogram of the CB11 growth, showing the increasingly larger scattering intensity.



Fig 68 Gaussian diagram of sample CB11. Surface roughness (rms) of the final device is 149nm. The initial wafer roughness(rms) was 0.4nm.Spikes in the diagram are due to the crude calculation method, not actual changes in the Gaussian profile. Error 4%.on k.



Fig 69 Composite picture of Gaussian line scans, taken at (from top to bottom) t = 500, 750, 1000, 1250, 1500 seconds. Left hand images corresponds to right hand statistics.

In figure 64, only one line out of several hundred has been used, which means the statistics is not very good (640 counts, instead of 307 200). However, with the use of the whole distribution, and correlating the two colours, the statistics should be good enough to fit the section 4.4.1 model.

An artefact of the Gaussian constant plot, figure 68, is what seems like a high noise level due to precession. A problem with precession is that the Gaussian constant will not have the same values for the left and right side of the peak height: i.e. it is not symmetrical. The Gaussian can be corrected with, for example, a geometrical function to show the angle of the wafer, and therefore also the laser angle. A way of deciding this angle, is by fitting an ellipse to the distribution, and minimising the difference (figure 70). As the distribution in this case measured at several different angles on the CCDimage, which means any skewness should be lost in the averaging process. Some noise is still evident, though, as the parameter extraction is somewhat crude.



Fig 70 Illustration of the fitting of an ellipse to the asymmetrical Gaussian distribution.

A second artefact is that, as the reflected light goes down in intensity, large spikes start to appear in the Gaussian constant diagram. This is mainly due to that the origin can be miscalculated as the intensity goes down, when speckle effect intensity in the reflection will appear as relatively higher in intensity. A finer error correcting algorithm, which prohibits sudden jumps in intensity should remove this.

As shown above, the technique of laser light profiling shows promise as a technique for *in situ* roughness monitoring. It has been shown that there are indeed clear trends in how the laser light profile changes with a correspondent change in roughness, and a model for how this usable for real-time scattering monitoring has been suggested. No AFM correlation has been made for this data at present time, as the dataset is limited, see figure 64, but with some further work, the scattering trends shown should easily be able to be converted to roughness data. Especially the ARS work from section 4.1.5 could be used to calibrate this data to real values.

With the ARS, single/dual wavelength interferometry and laser light profile sections, is has been shown that the combined interferometric reflectance/laser light profiling instrument has potential to replace the currently used single wavelength interferometry instruments. It has also been shown, for the first time, that scattering data can be extracted, not only as part of cross-correlating the interferometry data, but also unambiguously through the profiling of the laser light. This makes this instrument a serious contender for being the standard next-generation MOCVD *in situ* growth monitoring. Some further work has to be done to properly calibrate the profiler, with more AFM measurements being done to support this. Also, an engineering sample of the final instrument should be made to improve the signal to noise ratio in the profiler.

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5 Conclusion.

Concluding this thesis will be a summary, along with some final remarks on the subject of future work. The future work that is mentioned here was not performed during the project, because it was outside the limits of the project description, or unfortunate events outside the control of the author, like the cut-backs in the production of IQE Cardiff, which meant no testing of the instrument could be done on an Aixtron 2600 reactor.

5.1 post-Project Summary.

This project was started with the intention to create a next generation *in situ* MOCVD growth monitoring instrument, in order to solve problems with the currently used technology with respect to versatility and accuracy. The problem is mainly that instruments today are starting to be too inaccurate for modern growth applications, with the move towards finer structures, and therefore towards the need for more accurate monitoring. There is also a larger demand for controlling surface properties during growth, as they ultimately decide the properties of all interfaces within the grown structures. Therefore, in many respects they also decide the properties of the final devices.

As MOCVD reactors are normally run at atmospheric pressure, several contemporary techniques, based on optical monitoring only, were studied as a solution. In the end reflectance interferometry [RI] was chosen as the method, as the initial proposal put restrictions on the geometry of the optical access port. The port cannot be used successfully with any other current technique, not only because of the geometry, but also because the sensitivity of other techniques to precession of the substrate. Reflectance interferometry is relatively insensitive to this. There are some inherent problems of RI which have to be looked into, like

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the interference/scattering signal issue, discussed throughout this thesis. To cure some of these, a dual wavelength laser solution was implemented. This enables cross-correlation between the two wavelengths, and thus better accuracy. The instrument also achieves redundancy with this, as it is still possible to run in single wavelength mode. A switch to digital sensors in the instrument also improved sampling signal quality, with a trade-off in speed. There are digital devices with a significantly higher sample rate available today, though, so it is a problem that can be overcome.

When it comes to the second part of the project, surface roughness monitoring, three different techniques were evaluated:

- Firstly, the simplest and most direct technique was angle resolved scattering [ARS], as modelled already by Rayleigh. The technique is highly usable as an *ex situ* roughness monitor, but as an *in situ* technique it proved to need too large an angle to work directly. The specification of the optical port was about 6 degrees ($\pm 3^{\circ}$), whereas about 20 degrees ($\pm 10^{\circ}$) would have been needed in order to make ARS work properly. The *ex situ* measurements done throughout the project were very helpful, when trying to understand the basics of scattering, especially with the materials used, CdTe/CdS.

- Secondly, cross-correlation of the interferometry signal from two different wavelengths was researched. The scattering/surface roughness signal is the difference between the theoretical and measured intensity. As the scattering is wavelength dependent, correlation between the two can be done. The crosscorrelated scattering signal has proven to be accurate, when related to AFM roughness studies. This uses the same effect as the interferometer, which is unfortunate, as any error in the signal would be shared between the two. These dual wavelength measurements have shown that different wavelengths also have

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different response times. By using a shorter wavelength, a faster response can be achieved. This would be important, as the lack of response in time is one of the major disadvantages with the technology used today. The blue pixel area on the CCD should preferably be used for this.

- Thirdly, laser light profiling [LLP] was investigated as a new approach to *in situ* monitoring. When trying to adapt the ARS technique to an *in situ* solution, it was recognised that the scattering intensity cannot be used directly, because of the pyrometer port restriction. In fact, the only intensity that can be reliably detected with the low powered lasers that are used in the instrument, is the specular reflectance. This has already been used in the cross-correlation of the scattering intensity, but this is unfortunately a ambiguous method, as both the growth data and the scattering data come from the same source and measure the same effect. LLP does use the same intensity source, but instead uses changes to the laser light profile, due to changes in surface roughness, to calculate the scattering. In effect it is using a near normal subset of the ARS. Combined, these two methods are measuring the same parameter unambiguously. The technique is usable for large differences in roughness from growth start to growth end. The precession has proven to be quite possible to follow on the 15Hz CCD-imager in use, which means this also can be used as a rotation monitor.

The combination of reflectance interferometry, cross-correlated scattering and LLP, together with a change towards digital sampling of data, has proven to be far more accurate and versatile compared to the currently used technology. The use of LLP has made it possible to unambiguously separate the surface scattering from the interferometer signal, which effectively adds a roughness monitor to the setup. The short wavelength, whether it is green or blue, also achieves a much higher response time than the original setup, while the long wavelength ensures the growth rate can be monitored even for thicker films. This was achieved while preserving the simple and robust design of the original single wavelength interferometer, and without adding any further complexity.

5.2 Project achievements.

The first year of the project aimed mainly at developing the techniques and a testbed for the ex situ analysis, including electronics and software. This was used to explore the Glass/CdS/CdTe layer thin film structures, already grown within the research group before the project was started, with respect to normal-mode ARS. The structures were selected to behave very much like "snapshots" of a growing film, with respect to developments in surface roughness. The ex situ studies have shown that the ARS-distributions are well-defined and, and have made it possible to accurately predict the scattered intensity distributions from laser illuminated surfaces of different roughness. In situ use of this technique, however, was not possible because of the angular restriction on the optical port. During the second and third year, the AR scattering was also measured on highly orientated films, as there was a switch from glass substrates to single crystal Si (001). These films showed a radically different scattering distribution than the films grown on glass. Even though these kind of measurements have been reported before^{1,2}, this is the first time these measurements have been reported for CdS/CdTe films.

The second year studies have built on those measurements, and a wealth of sample surfaces have been studied by means of AFM and SEM. This gathering of data was in anticipation of the *in situ* reflectance measurements. The sample-sets have, again, consisted of structures which can be said to be "snapshots" of a layered MOCVD growth run with respect to the surface roughness, and has therefore been of use when trying to model the real-time surface scattering signal

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development. An in situ scattering and reflectance testbed was constructed in order to research this. Here, a single laser, situated normal to a sample, illuminates the surface, creating a Newton's ring interference pattern. The reflection and scattering of this interference pattern could be analysed for information of the developments of the film, where three parameters can be said to be important. The first two are the standard interferometry growth and laser light scattering parameters, as specified in the project proposal. The third parameter would, at least in theory, be the monitoring of film stress. Just as the change in the tail-off of the combined Gaussian/Yang function, section 4.4.3, can be used for monitoring roughness, any change in the Gaussian constant of the LLP distribution could be used for monitoring stress; any change in distribution would be due to a focusing/ defocusing effect of the surface, which can only be due to a change in the curvature of the film and substrate, because of tensile or compressive stress. The structure would thus work as a lens. This, of course, only applies to uniform films. This was not investigated further, as it was considered to fall outside the boundaries of the initial proposal.

The photodetector in the original interferometry setup was replaced with a standard CCD-camera, to reduce the need for additional detectors, filters and optics, and in order to detect the spot profiles. This also enhanced signal to noise ratio. The benefit of the switch is obvious, as this enables monitoring of three different wavelengths simultaneously in the same detector, simultaneous with regular CCD imaging. The laser wavelengths would have to be matched to the maxima in spectral responses of the different pixel types of the CCD-array, which is something that happened in the third year. Only the green and red pixel wavelength were ever used, but the instrument could just as easily have been based on any matched combination of red, green and blue. The further red the

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wavelength is shifted, the less the interference signal will be disturbed by surface scattering. The further into the blue spectrum the laser is, the faster the response would be. Ideally, the combination of an infra-red and an ultra-violet laser would be the choice, but as a proof of concept the green and red wavelengths suffice. Infra-red and ultra-violet lasers are also substantially more expensive, and a matched CCD array would have to be specially developed for this. Furthermore, infrared and ultraviolet wavelengths have proven to be very difficult to work with. For example, alignment of such lasers is very time consuming.

The third year added the second wavelength laser to the setup. The Newton's ring pattern was also decided to be too hard to produce reliably, and, although convenient for the calculations, not necessary for achieving the targets. A regular dispersion lens was instead used for the incident light, and this has proved to be a good solution. With this second, dual wavelength, version of the combined interferometer/profiler, a number of growth runs were performed. The interferometry data was compared with data from a conventional interferometry system, and AFM data was correlated with these results. This material was later presented in the IC-MOVPE IX conference in Berlin, Germany and subsequently published³. Analysis of the data has proven to predict nicely the progress of the film growth *in situ* and a model has been suggested for real-time monitoring of the surface roughness.

The instrument has only been used for single wafer growths, and calculations of the speed of the devices used in the experiments have shown that these are too slow for multi-wafer monitoring. Fortunately, off-the-shelf, high-speed devices are available for drop in replacements. Again, as proof of concept, the design can be considered to be sound, and all targets set up by the proposal has been successfully accomplished.

5.3 Further Work.

Although the project can be said to be a success, there are several questions that have appeared while carrying out the project. The discussion will divided into the two different signals: interferometry and scattering.

5.3.1 Interferometry signal.

The interferometry signal is well researched already, but some issues have turned up during the project;

- Work would have to be done to try to correctly model the behaviour at the onset of growth. It is obvious that a short wavelength will give a faster response time, with a trade-off on the monitorable film thickness. A blue laser should therefore be added to the instrument.

- In conjunction to the above response time, the zero point for the onset of growth could be studied by extrapolating the response delay for the wavelengths used. The extrapolation would be lim t, as $\lambda \rightarrow 0$. Several more wavelengths would have to be investigated, possibly from a high power broadband source, with Fabry-Perot filters deciding the analysis wavelengths.

- The laser light is presently detected after it has been dispersed over a larger area. While this is not in itself a problem, the 8-bit nature of the CCD used can cause saturation. There are already several companies manufacturing everything from 10 to 16 bit CCD detectors, which would be an improvement.

- The laser stability is important for the quality of the data. During the dual wavelength experiments, the green YAG laser has performed flawlessly, while the red diode laser has had problems with mode switching and noise. While the diode laser probably can be stabilised by using a better power supply, the hi-grade supply that would be necessary, would be more expensive than starting

off with a better laser in the first place. This should be considered when continuing work on the instrument.

- A higher speed CCD device is needed if the instrument is going to work on a multi-wafer reactor. The current detector can work at 12 Hz without dropping frames, which is close to the theoretical bandwidth of the USB1.1 bus. However, there are commodity devices capable of 90 Hz at the same resolution. They of course instead use fire-wire, the IEEE1394 standard, as the bandwidth of this bus is about 20 times that of the USB1.1 bus. The reimplementation of the monitoring software should also be done, but with the generic device driver structure of any *nix operating system, this would be a minor adjustment.

5.3.2 Scattering signal.

When it comes to the scattering signal, this has still not been used much for monitoring, even if the theory is well known. The usage of the cross-correlated scattering signal is accurate, but the usage of the LLP would be preferable, as it provides unambiguousity. Some work will have to be done in order to enable better usage of this data.

- The dual wavelength LLP behaviour should be studied further, using the combined instrument prototype, and calibration of it will have to be made, using AFM roughness data.

- The quality of the LLP data is dependent on the resolution of the CCD detector. With the above discussion of the speed and depth of the device, a resolution of 1024x768 would be adequate.

- The noise level has to be substantially reduced in the LLP signal, by mathematically treating and removing the precession from the signal. The current computing setup manages to treat about one frame per 7 seconds, with

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its 1GHz Athlon processor, even if this could probably be done at a double rate, with proper optimisation to the code. To properly monitor the precession, the central beam would have to be followed closely, and at least 8 measurements would have to be done per revolution. As the rotation of a wafer in a reactor cell is normally held at about 1 revolution per second, the frames would have to be monitored at the rate of about 8Hz. This mean we would have to increase the processing power about 50 times, which would be impractical (at least for the time being, considering processing power of CPUs doubles every 18 months), as this would involve setting up a computing cluster of perhaps a couple of dozens of commodity machines. Maintaining the laser beam normal to the sample, which would eliminate this effect, could also be achieved by a mechanical setup that follows the wafer precession, but this would only be practical for a single wafer setup. This is not an option, as the instrument is ultimately to be used on multi wafer reactors. A finer mathematical treatment of the CCD-images with respect to the LLP distribution skewness would have to be the best option, even if more processing power would undoubtedly be required. But in the case of using this, the skewness contribution to the overall distribution would have to be researched and modelled.

There are also a few general issues that have to be solved:

- A working engineering prototype should be manufactured, and extensive testing of the instrument will still have to be made on a multi-wafer reactor.

- An adaptor for the interface between the Aixtron reactor optical port and the instrument has to be designed and manufactured.

- The programming code and computer monitor interface has to be cleaned up to be more user friendly. Similarly, this would have to be optimised. An integrated industrial solution with the new interface, with easy to use touch screens and feedback loops for process control would also have to be engineered.

5.4 Final remarks.

This project was started three years ago under the EPSRC project "Combined Interferometer and Laser Scattering Monitor for Multi-Wafer MOCVD", EPSRC proposal no. GR/M50034. The project was aimed at developing the theory, model the behaviour, and produce a testbed for an *in situ*, real-time MOCVD film growth and surface roughness monitor, which could only use a very narrow orifice for collecting the data. The theory was evaluated using *ex situ* ARS measurements, which provided the understanding to build a first, single wavelength interferometer, using proven components. This was made to evaluate a new, digital design to the standard normal incidence reflectance interferometer, where instead of using analogue detection electronics, a CCD-array was used. This gives numerous advantages, such as improved signal-to-noise ratio, and much simpler design.

As the single wavelength interferometer functioned as specified, the interferometer testbed was modified to use two lasers, with wavelengths matched to the CCD pixel response. The two wavelengths provide a higher accuracy to the growth monitor, while also providing redundancy. By using a long and a short wavelength, a combination of fast response and long term monitoring was achieved.

Using two wavelengths also enables cross-correlation of the different intensities, which can be used for detection of the scattered intensity. Thus, the surface roughness was monitorable, even if this approach cannot be said to

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achieve unambiguousity, as the signal and effect measured is the same as the interferometer signal. To achieve unambiguousity in the roughness monitoring, a second way to monitor the roughness was therefore researched. By using laser light profiling of the reflectance distribution in conjunction with cross-correlation, this was achieved.

Using the three different effects, dual wavelength reflectance interferometry, scattering cross-correlation and laser light profiling, the combination monitor design was finalised. The final instrument has proven to be accurate, robust, simple and reliable, and does fulfil the specifications that were outlined in the original proposal.

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6 Acknowledgements.

The author would like to acknowledge the support of Professor Stuart Irvine, under whose supervision this project was carried out. Furthermore, the discussions within the Optoelectronic Materials Chemistry group concerning different aspects of the project and all other help were greatly appreciated. Thanks to the technicians of the Chemistry Department, University of Wales, Bangor, for help developing and making the different pieces of equipment used. Finally, I would also like to thank my family for all support during these years.

The project "Combined Interferometer and Laser Scattering Monitor for Multi-Wafer MOCVD" was carried out with the financial support of EPSRC grant no. GR/M50034, which is gratefully acknowledged. Thanks to the Linux community for providing the basis for the software developed and used. No Microsoft products were used in this project.

7 Appendix.

7.1 Programming code.

Listed here are some C++ code, that was used in capturing and calculating the growth and roughness parameters. The code should conform to the ANSI C++ standard, and was compiled with the GNU gcc compiler.

7.1.1 readdata.cc.

readdata.cc, is the code for the very simple data collector in the ARS scatterometer. It basically opens a COMEDI device (in this case the Advantech 711b A/D card), reads the 12-bit data, and passes it to stdout. At the same time, it sends a pulse signal to the parallel port, which is connected to the stepper-motor driver, and hence controls it.

/* Must be compiled with -O for outb to be inlined, otherwise link error */ //#include <stdlib.h>

//#include <stdio.h>

#include <sys/io.h>

#include <linux/ppdev.h>

#include <linux/parport.h>

#include <time.h>

#include <sched.h>

#include <iostream.h>

#include <comedilib.h>

#include <unistd.h>

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int base=0x378;

int subdev = 0 ;	/* change this to your input subdevice */
int $chan = 0;$	/* change this to your channel */
int range = 0 ;	/* more on this later */

/* more on this later */

/*unsigned char steptable[16]=

int aref = AREF GROUND;

{0x0000,0x0002,0x0004,0x0020,0x0040,0x0006,0x0022,0x0042,0x0024,

0x0044,0x0060,0x0026,0x0046,0x0062,0x0064,0x0066};//test all channels

*/

unsigned char steptable1[10]=

 $\{0x0044, 0x0064, 0x0044, 0x0064, 0x0044, 0x00044, 0x0044, 0x0040, 0x0044, 0x0044, 0x0044, 0x0044, 0x0044, 0x0044, 0x0044, 0x0044, 0x0044, 0x0040, 0x004, 0x0044, 0x0044, 0x0044, 0x0044, 0x0044, 0x0044, 0x0044, 0x0$

0x0064,0x0044,0x0064,0x0044,0x0064};//half, backwards

unsigned char steptable2[10]=

{0x0040,0x0060,0x0040,0x0060,0x0040,

0x0060,0x0040,0x0060,0x0040,0x0060};//half, forward

unsigned char steptable3[10]=

{0x0004,0x0024,0x0004,0x0024,0x0004,

0x0024,0x0004,0x0024,0x0004,0x0024};//full, backwards

unsigned char steptable4[10]=

{0x0000,0x0020,0x0000,0x0020,0x0000,

0x0020,0x0000,0x0020,0x0000,0x0020};//full, forward

void verbose_outb(int port, int value)

{

printf("outb(%02X,%04X)\n",port,value);

// outb(port,value);

```
}
void readcard(int t,comedi_t *it)
{
lsamp1 t temp=0;
lsampl t data;
for (int i=0;i<10;i++){
          comedi_data_read(it,subdev,chan,range,aref,&data);
          temp+=data;
          }
cout << t << "\t" << temp << "\n";
}
void usdelay(unsigned long delay_in_us)
{
long
          i;
struct timespec
                       sleep data, dummy return;
struct sched param
                       normal priority data,
                                                 realtime priority data;
// Prepare the normal priority data structure:
normal priority data.sched priority = 0;
// Prepare the real-time priority data stucture:
realtime_priority_data.sched_priority = 1;
// Prepare the nanosleep data stucture for sleeping the reminder
// of the division of the desired delay time in microseconds by
// 1000. The result is formatted in nanoseconds.
sleep_data.tv_nsec = 1000000;
sleep_data.tv_sec = 0;
```
sleep_data.tv_nsec = (delay_in_us % 1000) * 1000;

// Set the current process to real-time priority scheduling:

sched_setscheduler (0, SCHED_RR, &realtime_priority_data);

// Sleep the millisecond-reminder time:

nanosleep (&sleep_data, &dummy_return);

// Prepare the nanosleep data structure for sleeping in portions

// of 1 millisecond each. The sleeping time is given in

// nanoseconds.

sleep_data.tv_nsec = 1000000;

// Perform the necessary number of delays of 1ms each

for (i = delay in us / 1000; i; i--)

{

// Set the current process to real-time priority scheduling:

sched_setscheduler (0, SCHED_RR, &realtime_priority_data);

// Sleep for 1ms:

nanosleep (&sleep_data, &dummy_return);

// Set the current process to normal priority scheduling so

// the other processes can breath:

sched_setscheduler (0, SCHED_OTHER, &normal_priority_data);

```
}
void base_line_pre(void)
{
int i=0;
```

comedi_t *it;

```
it=comedi_open("/dev/comedi0");
```

for (i=0;i<100;i++){

readcard(i,it);

usdelay(1250);

}

comedi_close(it);

}

```
void slow_sweep(void)
```

```
{
```

```
int i=0,j=0;
```

```
comedi_t *it;
```

it=comedi_open("/dev/comedi0");

for (i=0;i<1850;i++){

readcard(j*i+100,it);

```
for(j=0;j<100;j++){
```

outb(steptable2[j%10],base);

```
usdelay(1250);
```

}

}

```
comedi_close(it);
```

```
}
```

void sweep_back(void)

{

int i=0,j=0;

comedi_t *it;

```
it=comedi_open("/dev/comedi0");
```

for(i=0;i<1850;i++){ readcard(j*i+1850*100+100,it); for(j=0;j<100;j++){ outb(steptable1[j%10],base); usdelay(1250); } } comedi close(it); } void base line post(void) { int i=0; timespec sleep_data; struct //struct timespec dummy_r; sleep_data.tv_nsec = 1000000; comedi_t *it;

it=comedi_open("/dev/comedi0");

```
for (i=0;i<10000000;i++){
```

readcard(i,it);

usdelay(10000);

}

comedi_close(it);

}

int main(void)

{

iopl(3); /* Enable i/o (if root) */

// base_line_pre();

slow_sweep();

cout << "Forward OK\n";</pre>

// outb(0x0066,0x378);

usdelay(100000);

sweep_back();

// base_line_post();

cout << "Back OK\n";</pre>

getc(stdin);

return 1;

}

7.1.2 Regress.cc

11

11

11

Regress.cc was used for evaluating the LLP parameters. It takes a standard 8-bit ppm file, and uses its rgb data for a) calculating the TIS. b) calculating the middle point of the Gaussian distribution. c) calculating the gaussian regression. d) Calculating the Gaussian intercept.

```
//Main.c Regression of CCD-images
#include <stdio.h>
#include <stdlib.h>
#include <string.h>
#include <math.h>
int w=640;
int h=480;
double extract_line(int internal_reflection, unsigned int data[3][640][480]);
int calc_internal_reflection(unsigned int data[3][640][480]);
unsigned int data[3][640][480];
int main(void) {
bool found=0;
/* do not try previus used filenames from the same session */
static
         int capture_number=0,old capture number;
int file err=0;
```

FILE *tmp=NULL;

char *filename;

int i,k,j; //step variables

char buf[5120];

FILE *fp=NULL;

int debug=0;

double line=0.0;

int internal_reflection=0;

filename = (char *)malloc (strlen("/home")+strlen("SDL_picture")+5+strlen

(".pnm")+10); // Malloc error

do {

/* safe that it can't get too big */

sprintf(filename, "%s%05d%s","SDL_picture",capture_number,".pnm");

old_capture_number=capture_number;

if (debug > 0) printf("trying %s\n", filename);

tmp = fopen(filename, "r");

if (debug > 0) printf("Opened %s\n", filename);

if (tmp==NULL) {

found=1;

} else {

fclose(tmp);

if (debug > 0) printf("Closed %s\n", filename);

capture_number++;

if $(100000 \le \text{capture_number})$

/* if imposible to get an file name return */

```
printf("No filename found\n");
free(filename);
}
if (!(tmp=NULL)){
fp = fopen(filename,"r");
for (i=0; i<3;i++){ // Get rid of the ppm header</pre>
```

fgets(buf,sizeof(buf),fp);

}

for(j=0;j<h;j++){

for(k=0;k<w;k++){

for (i=0; i<3;i++){

fgets(buf,4,fp);

data[i][k][j]=atoi(buf);

fgetc(fp); //Get rid of tabulator

}

}

//Calculate the internal reflection plane (once, as scattering is zero at t=0)

internal_reflection=calc_internal_reflection(data);

//Calculate the gaussian

}

line=extract_line(capture_number,data);

}

} while(!(old_capture_number==capture_number));

fcloseall();

free(filename);

}

double extract_line(int internal_reflection, unsigned int data[3][640][480]) {
//Horisontal +step variables

int

j,i,k=0,cumulated_red_h=0,cumulated_green_h=0,largest_red_h=0,largest_green_ h=0,lredx_h=0,lgreenx_h=0;

//Vertical variables

int

cumulated_red_v=0,cumulated_green_v=0,largest_red_v=0,largest_green_v=0,lre dx_v=0,lgreenx_v=0;

//Negative diagonal variables, 2 sets

int

cumulated_red_dn1=0,cumulated_green_dn1=0,largest_red_dn1=0,largest_green _dn1=0,lredx_dn1=0,lgreenx_dn1=0;

int

```
cumulated_red_dn2=0,cumulated_green_dn2=0,largest_red_dn2=0,largest_green
dn2=0,lredx_dn2=0,lgreenx_dn2=0;
```

int largest_red_dn=0,largest_green_dn=0,lredx_dn=0,lgreenx_dn=0;

//Positive diagonal variables, 2 sets

int

```
cumulated_red_dp1=0,cumulated_green_dp1=0,largest_red_dp1=0,largest_green
_dp1=0,lredx_dp1=0,lgreenx_dp1=0;
```

int

cumulated_red_dp2=0,cumulated_green_dp2=0,largest_red_dp2=0,largest_green
_dp2=0,lredx_dp2=0,lgreenx_dp2=0;

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```
int largest_red_dp=0,largest_green_dp=0,lredx_dp=0,lgreenx_dp=0;
```

//Negative 30 degree variables, 2 sets

int

```
cumulated_red_2dn1=0,cumulated_green_2dn1=0,largest_red_2dn1=0,largest_gr
een_2dn1=0,lredx_2dn1=0,lgreenx_2dn1=0;
```

 int

```
cumulated_red_2dn2=0,cumulated_green_2dn2=0,largest_red_2dn2=0,largest_gr
een_2dn2=0,lredx_2dn2=0,lgreenx_2dn2=0;
```

int largest_red_2dn=0,largest_green_2dn=0,lredx_2dn=0,lgreenx 2dn=0;

//Negative 30 degree variables, 2 sets

int

cumulated_red_2dp1=0,cumulated_green_2dp1=0,largest_red_2dp1=0,largest_gr een_2dp1=0,lredx_2dp1=0,lgreenx_2dp1=0;

int

```
cumulated_red_2dp2=0,cumulated_green_2dp2=0,largest_red_2dp2=0,largest_gr
een_2dp2=0,lredx_2dp2=0,lgreenx_2dp2=0;
```

int largest_red_2dp=0,largest_green_2dp=0,lredx_2dp=0,lgreenx 2dp=0;

//Negative 60 degree variables, 3 sets

int

```
cumulated_red_3dn1=0,cumulated_green_3dn1=0,largest_red_3dn1=0,largest_gr
een_3dn1=0,lredx_3dn1=0,lgreenx_3dn1=0;
```

int

cumulated_red_3dn2=0,cumulated_green_3dn2=0,largest_red_3dn2=0,largest_gr een_3dn2=0,lredx_3dn2=0,lgreenx_3dn2=0;

int

cumulated_red_3dn3=0,cumulated_green_3dn3=0,largest_red_3dn3=0,largest_gr

```
een_3dn3=0,lredx_3dn3=0,lgreenx_3dn3=0;
```

```
int largest_red_3dn=0,largest_green_3dn=0,lredx_3dn=0,lgreenx_3dn=0;
```

//Positive 60 degree variables, 3 sets

int

```
cumulated_red_3dp1=0,cumulated_green_3dp1=0,largest_red_3dp1=0,largest_gr
een_3dp1=0,lredx_3dp1=0,lgreenx_3dp1=0;
```

int

cumulated_red_3dp2=0,cumulated_green_3dp2=0,largest_red_3dp2=0,largest_gr een_3dp2=0,lredx_3dp2=0,lgreenx_3dp2=0;

int

```
cumulated\_red\_3dp3=0, cumulated\_green\_3dp3=0, largest\_red\_3dp3=0, largest\_grammarked\_green\_3dp3=0, largest\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_grammarked\_g
```

```
een_3dp3=0,lredx_3dp3=0,lgreenx_3dp3=0;
```

int largest_red_3dp=0,largest_green_3dp=0,lredx_3dp=0,lgreenx_3dp=0;

int larg_rg_pn_order_index[1][1][3];

int total_integral_r=0,total_integral_g=0;

//Main loop 640,480,640

//Loop for total integral.

for(k=0;k<640;k++){

for(j=0;j<480;j++){

total_integral_r+=data[0][k][j];

total_integral_g+=data[1][k][j];

```
}
```

}

// Loop for line scans

for(k=0;k<640;k++){

for(j=0;j<480;j++){

```
if (k==0 || k==j )
        for (i=0;i<640;i++){
                                 //Horisontally
                     if(k==0){
                     cumulated red h+=data[0][i][j];
                     cumulated green_h+=data[1][i][j];
                     if (i==639) {
                                              //Horsontal end
         if(cumulated red h > largest red h) {
                     lredx_h=j;
                     largest red_h=cumulated_red_h;
                     }
         if(cumulated green h > largest green h) {
                     lgreenx_h=j;
                     largest green h=cumulated green h;
                      }
         cumulated red h=0;
         cumulated green h=0;
                      }
         }
}
cumulated_red_v+=data[0][k][j]; // Vertically
cumulated_green_v+=data[1][k][j];
if (j==479) {
                     //Vertical end
if(cumulated red v > largest red v) {
         lredx_v=k;
```

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and the second of the second of the second

```
largest red v=cumulated_red_v;
            }
  if(cumulated green v > largest green v) {
            lgreenx v=k;
            largest green v=cumulated green v;
            }
  cumulated red v=0;
  cumulated green v=0;
  }
                        //Negative and Positive diagonal
  if(k<560){
  if((int)(k-j) \ge 0 \&\& (int)(k-j) \le 639)
            cumulated_red_dn1+=data[0][k-j][j]; // Negative diagonal first half
            cumulated_green_dn1+=data[1][k-j][j];
            }
  if((int)(k-j+559)>0 && (int)(k-j+559)<639){
            cumulated red dn2+=data[0][k-j+560][j];
                                                               // Negative
diagonal second half
            cumulated green dn2+=data[1][k-j+560][j];
            k, j, k-j+560, j);
            }
  if((int)(k+j-479) \ge 0 \&\& (int)(k+j-479) \le 639)
            cumulated_red_dp1+=data[0][k+j-479][j];
                                                               // Positive
diagonal first half
            cumulated green dp1+=data[1][k+j-479][j];
            k,j,k+j-479,j);
```

if((int)(k+j+80)>0 && (int)(k+j+80)<639){

```
cumulated_red_dp2+=data[0][k+j+81][j]; // Positive
```

diagonal second half

```
cumulated_green_dp2+=data[1][k+j+81][j];
k,j,k+j+81,j);
}
```

```
//30deg slope diagonals (2nd order)
```

if (k < 460){

if ((int)(rint((double)(k-(j/sqrt(3)))))>=0 $\$

&& (int)(rint((double)(k-(j/sqrt(3)))))<=639) {// -30 degrees, first

half

}

cumulated_red_2dn1+=data[0][(int) rint((double)(k-(j/sqrt(3))))][j]; cumulated_green_2dn1+=data[1][(int) rint((double)(k-(j/sqrt(3))))]

[j];

}

```
if ((int)(rint((double)(k-(j/sqrt(3)))))+459>=0 \
&& (int)(rint((double)(k-(j/sqrt(3)))))+459<639) {// -30 degrees, second half
cumulated_red_2dn2+=data[0][(int) rint((double)(k-(j/sqrt(3))+460))][j];
cumulated_green_2dn2+=data[1][(int) rint((double)(k-(j/sqrt(3))+460))][j];
}</pre>
```

if ((int)(rint((double)(k+(j/sqrt(3)-278))))>=0 $\$

&& (int)(rint((double)(k+(j/sqrt(3)-278))))<=639) {//+30 degrees, first half cumulated_red_2dp1+=data[0][(int) rint((double)(k+(j/sqrt(3))-278))][j];

```
cumulated_green_2dp1+=data[1][(int) rint((double)(k+(j/sqrt(3))-278))][j];
  }
  if ((int)(rint((double)(k+(j/sqrt(3)))))+459-278>=0 \
   && (int)(rint((double)(k+(j/sqrt(3)))))+459-278<639) {// +30 degrees, second
half
   cumulated red 2dp2 = data[0][(int) rint((double)(k+(i/sqrt(3))+460-278))][i];
   cumulated green 2dp2 = data[1][(int) rint((double)(k+(j/sqrt(3))+460-278))]
[j];
   }
   }
   //+-60 degrees diagonals (3nd order)
   if (k < 491)
   if ((int)(rint((double)(k-(j*sqrt(3))))) >= 0 \
   && (int)(rint((double)(k-(j*sqrt(3)))))<=639) {// -60 degrees, first third
   cumulated_red_3dn1+=data[0][(int) rint((double)(k-(j*sqrt(3))))][j];
   cumulated green 3dn1+=data[1][(int) rint((double)(k-(j*sqrt(3))))][j];
   }
   if ((int)(rint((double)(k-(j*sqrt(3)))))+491>=0 \
   && (int)(rint((double)(k-(j*sqrt(3)))))+491<=639) {// -60 degrees, seed third
   cumulated red 3dn2 = data[0][(int) rint((double)(k-(i*sqrt(3))))+492][i];
   cumulated_green_3dn2+= data[1][(int) rint((double)(k-(j*sqrt(3))))+492][j];
   }
   if ((int)(rint((double)(k-(j*sqrt(3)))))+984>=0 \
   && (int)(rint((double)(k-(j*sqrt(3)))))+984<=639) {// -60 degrees, third third
   cumulated_red_3dn3+=data[0][(int) rint((double)(k-(j*sqrt(3))))+984][j];
```

```
cumulated_green_3dn3+=data[1][(int) rint((double)(k-(j*sqrt(3))))+984][j];
```

```
if ((int)(rint((double)(k+(j*sqrt(3)-830))))>=0 \
&& (int)(rint((double)(k+(j*sqrt(3)-830))))<=639) {//+60 degrees, first third
cumulated_red_3dp1+=data[0][(int) rint((double)(k+(j*sqrt(3))))-830][j];
cumulated_green_3dp1+=data[1][(int) rint((double)(k+(j*sqrt(3))))-830][j];
}</pre>
```

```
if ((int)(rint((double)(k+(j*sqrt(3)))))+491-830>=0 \
```

```
&& (int)(rint((double)(k+(j*sqrt(3)))))+491-830<=639) {//+60 degrees,
```

second third

```
cumulated_red_3dp2+=data[0][(int) rint((double)(k+(j*sqrt(3))))+491-830][j];
cumulated_green_3dp2+=data[1][(int) rint((double)(k+(j*sqrt(3))))+491-830]
[j];
```

}

```
if ((int)(rint((double)(k+(j*sqrt(3)))))+982-830>=0 \
    && (int)(rint((double)(k+(j*sqrt(3))))+982-830<=639) {//+60 degrees,th third
    cumulated_red_3dp3+=data[0][(int) rint((double)(k+(j*sqrt(3))))+982-830][j];
    cumulated_green_3dp3+=data[1][(int) rint((double)(k+(j*sqrt(3))))+982-830]
[j];
    }
    }
    // for the + and - diagonals</pre>
```

```
if(cumulated_red_dn1 > largest_red_dn1) {
```

```
lredx_dn1=k;
```

```
largest_red_dn1=cumulated_red_dn1;
```

```
if(cumulated red_dn2 > largest_red_dn2) {
```

 $lredx_dn2=k+560;$

largest red_dn2=cumulated_red_dn2;

}

if(cumulated_green_dn1 > largest_green_dn1) {

lgreenx_dn1=k;

largest_green_dn1=cumulated_green_dn1;

}

if(cumulated_green_dn2 > largest_green_dn2) {

lgreenx_dn2=k+560;

largest_green_dn2=cumulated_green_dn2;

}

if(cumulated_red_dp1 > largest_red_dp1) {

lredx_dp1=k-479;

largest_red_dp1=cumulated_red_dp1;

}

if(cumulated_red_dp2 > largest_red_dp2) {

lredx_dp2=k+80;

largest_red_dp2=cumulated_red_dp2;

}

if(cumulated_green_dp1 > largest_green_dp1) {

lgreenx_dp1=k-479;

largest_green_dp1=cumulated_green_dp1;

```
}
```

if(cumulated_green_dp2 > largest_green_dp2) {

lgreenx_dp2=k+80;

largest_green_dp2=cumulated_green_dp2;

}

//Compare first and second half diagonals

```
if(largest_red_dn1>largest_red_dn2) {
```

largest_red_dn = largest_red_dn1;

lredx_dn=lredx_dn1;

} else {

largest_red_dn = largest_red_dn2;

lredx_dn=lredx_dn2;

}

if(largest_green_dn1>largest_green_dn2) {

largest_green_dn = largest_green_dn1;

lgreenx_dn=lgreenx_dn1;

} else {

largest_green_dn = largest_green_dn2;

lgreenx_dn=lgreenx_dn2;

}

if(largest_red_dp1>largest_red_dp2) {

largest_red_dp = largest_red_dp1;

lredx_dp=lredx_dp1;

} else {

largest_red_dp = largest_red_dp2;

```
lredx_dp=lredx_dp2;
```

```
}
```

```
if(largest_green_dp1>largest_green_dp2) {
```

```
largest_green_dp = largest_green_dp1;
```

lgreenx_dp=lgreenx_dp1;

} else {

```
largest_green_dp = largest_green_dp2;
```

lgreenx_dp=lgreenx_dp2;

}

// Set to zero for next loop

cumulated_red_dn1=0;

cumulated_red_dn2=0;

cumulated_red_dp1=0;

cumulated_red_dp2=0;

cumulated_green_dn1=0;

cumulated_green_dn2=0;

cumulated_green_dp1=0;

```
cumulated green dp2=0;
```

// And the 30degree slopes

```
if(cumulated_red_2dn1 > largest_red_2dn1) { //largest - 30 deg diagonals,
```

first half red

```
lredx_2dn1=k;
```

```
largest_red_2dn1=cumulated_red_2dn1;
```

}

if(cumulated_red_2dn2 > largest_red_2dn2) { //largest -30deg diagonals,

```
second half red
```

```
lredx 2dn2=k+460;
            largest red 2dn2=cumulated red 2dn2;
            }
  if(cumulated green 2dn1 > largest green 2dn1) {
                                                            //largest -30deg
diagonals, first half green
            lgreenx 2dn1=k;
            largest green 2dn1=cumulated green 2dn1;
            }
  if(cumulated green 2dn2 > largest green 2dn2) {
                                                            //largest -30deg
diagonals, first half green
            lgreenx 2dn2=k+460;
            largest green 2dn2=cumulated green 2dn2;
            }
  if(cumulated_red_2dp1 > largest_red_2dp1) { //largest +30deg diagonals,
first half red
            lredx 2dp1=k-278;
            largest_red_2dp1=cumulated_red_2dp1;
            }
  if(cumulated_red_2dp2 > largest_red_2dp2) { //largest +30deg diagonals,
second half red
            lredx 2dp2=k+460-278;
            largest red 2dp2=cumulated red 2dp2;
            }
  if(cumulated_green 2dp1 > largest_green 2dp1) {
                                                            //largest +30deg
diagonals, first half green
```

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```
lgreenx_2dp1=k-278;
largest_green_2dp1=cumulated_green_2dp1;
}
```

//largest +30deg

if(cumulated_green_2dp2 > largest_green_2dp2) {

diagonals, first half green

lgreenx_2dn2=k+460-278;

largest_green_2dp2=cumulated_green_2dp2;

}

// compare first and second half 30deg diagonals

if(largest_red_2dn1>largest_red_2dn2) {

largest_red_2dn = largest_red_2dn1;

lredx_2dn=lredx_2dn1;

} else {

largest_red_2dn = largest_red_2dn2;

lredx_2dn=lredx_2dn2;

}

if(largest_green_2dn1>largest_green_2dn2) {

largest_green_2dn = largest_green_2dn1;

lgreenx_2dn=lgreenx_2dn1;

} else {

largest_green_2dn = largest_green_2dn2; lgreenx_2dn=lgreenx_2dn2;

}

if(largest_red_2dp1>largest_red_2dp2) {

largest_red_2dp = largest_red_2dp1;

```
lredx_2dp=lredx_2dp1;
```

} else {

largest_red_2dp = largest_red_2dp2; lredx_2dp=lredx_2dp2;

}

if(largest_green_2dp1>largest_green_2dp2) {

largest_green_2dp = largest_green_2dp1;

lgreenx_2dp=lgreenx_2dp1;

} else {

largest_green_2dp = largest_green_2dp2;

lgreenx_2dp=lgreenx_2dp2;

}

// Set to zero for next loop

cumulated_red_2dn1=0;

cumulated_red_2dn2=0;

cumulated_green_2dn1=0;

cumulated_green_2dn2=0;

cumulated_red_2dp1=0;

cumulated_red_2dp2=0;

cumulated_green_2dp1=0;

cumulated_green_2dp2=0;

// And the 60degree slopes

if(cumulated_red_3dn1 > largest_red_3dn1) { //largest -60deg diagonals, first third red

lredx_3dn1=k;

```
largest_red_3dn1=cumulated_red_3dn1;
```

if(cumulated_red_3dn2 > largest_red_3dn2) { //largest -60deg diagonals, second third red

lredx_3dn2=k+492;

largest_red_3dn2=cumulated_red_3dn2;

}

if(cumulated_red_3dn3 > largest_red_3dn3) { //largest -60deg diagonals, third third red

lredx_3dn3=k+984;

largest_red_2dn2=cumulated_red_2dn2;

}

if(cumulated_green_3dn1 > largest_green_3dn1) { //largest -60deg

diagonals, first third green

lgreenx_3dn1=k;

largest_green_3dn1=cumulated_green_3dn1;

}

if(cumulated_green_3dn2 > largest_green_3dn2) { //largest -60deg

diagonals, second third green

lgreenx_3dn2=k+492;

largest_green_3dn2=cumulated_green_3dn2;

}

if(cumulated_green_3dn3 > largest_green_3dn3) { //largest -60deg

diagonals, third third green

lgreenx_3dn3=k+984;

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if(cumulated_red_3dp1 > largest_red_3dp1) { //largest +60deg diagonals, first third red

```
lredx_3dp1=k-830;
largest_red_3dp1=cumulated_red_3dp1;
```

}

if(cumulated_red_3dp2 > largest_red_3dp2) { //largest +60deg diagonals, second third red

```
lredx_3dp2=k+491-830;
```

```
largest_red_3dp2=cumulated_red_3dp2;
```

}

```
if(cumulated_red_3dp3 > largest_red_3dp3) { //largest +60deg diagonals, third third red
```

```
lredx_3dp3=k+982-830;
largest_red_3dp3=cumulated_red_3dp3;
}
```

```
if(cumulated_green_3dp1 > largest_green_3dp1) { //largest +60deg
```

diagonals, first half green

lgreenx_3dp1=k-830; largest_green_3dp1=cumulated_green_3dp1; }

if(cumulated_green_3dp2 > largest_green_3dp2) { //largest +60deg

diagonals, second half green

lgreenx_3dp2=k+491-830;

largest_green_3dp2=cumulated_green_3dp2;

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```
}
```

```
//largest +60deg
```

diagonals, third third green

```
lgreenx_3dp3=k+982-830;
```

if(cumulated green 3dp3 > largest green 3dp3) {

largest_green_3dp3=cumulated_green_3dp3;

}

// compare first, second and third thirds of 60deg diagonals

```
if (largest_red_3dn1>largest_red_3dn2) {
```

if (largest_red_3dn1>largest_red_3dn3) {

largest_red_3dn = largest_red_3dn1;

lredx_3dn=lredx_3dn1;

} else {

```
largest_red_3dn = largest_red_3dn3;
lredx_3dn=lredx_3dn3;
}
```

} else {

```
if (largest_red_3dn2 > largest_red_3dn3) {
```

largest_red_3dn = largest_red_3dn2;

lredx_3dn=lredx_3dn2;

} else {

}

```
largest_red_3dn = largest_red_3dn3;
lredx_3dn=lredx_3dn3;
}
```

if(largest_green_3dn1>largest_green_3dn2) {

if(largest_green_3dn1>largest_green_3dn3) {

largest_green_3dn = largest_green_3dn1;

lgreenx_3dn=lgreenx_3dn1;

} else {

```
largest_green_3dn = largest_green_3dn3;
lgreenx_3dn=lgreenx_3dn3;
}
```

} else {

```
if(largest_green_3dn2>largest_green_3dn3) {
    largest_green_3dn = largest_green_3dn2;
    lgreenx_3dn=lgreenx_3dn2;
    } else {
    largest_green_3dn = largest_green_3dn3;
    lgreenx_3dn=lgreenx_3dn3;
}
```

}

```
if (largest_red_3dp1>largest_red_3dp2) {
```

```
if (largest_red_3dp1>largest_red_3dp3) {
```

largest_red_3dp = largest_red_3dp1;

lredx_3dp=lredx_3dp1;

} else {

largest_red_3dp = largest_red_3dp3; lredx_3dp=lredx_3dp3; }

} else {

if (largest_red_3dp2 > largest_red_3dp3) {

largest_red_3dp = largest_red_3dp2;

lredx_3dp=lredx_3dp2;

} else {

```
largest_red_3dp = largest_red_3dp3;
lredx_3dp=lredx_3dp3;
}
```

}

if(largest_green_3dp1>largest_green_3dp2) {

if(largest_green_3dp1>largest_green_3dp3) {

largest green_3dp = largest_green_3dp1;

lgreenx_3dp=lgreenx_3dp1;

} else {

largest_green_3dp = largest_green_3dp3;
lgreenx_3dp=lgreenx_3dp3;
}

} else {

if(largest_green_3dp2>largest_green_3dp3) {

largest green_3dp = largest_green_3dp2;

lgreenx 3dp=lgreenx 3dp2;

} else {

}

```
largest_green_3dp = largest_green_3dp3;
lgreenx_3dp=lgreenx_3dp3;
}
```

// Set to zero for next loop

cumulated_red_3dn1=0;

cumulated_red_3dn2=0;

cumulated_red_3dn3=0;

cumulated_green_3dn1=0;

cumulated_green_3dn2=0;

cumulated_green_3dn3=0;

cumulated_red_3dp1=0;

cumulated_red_3dp2=0;

cumulated_red_3dp3=0;

cumulated_green_3dp1=0;

cumulated_green_3dp2=0;

cumulated_green_3dp3=0;

}

// Calculate middle.

// Density middle

int coord_hvd36_hvd36_pn_rg_xy[5][5][2][2][2];

const double sqrt3=sqrt(3);

//Red

// H,V

coord_hvd36_hvd36_pn_rg_xy[0][1][0][0][0]=lredx_v; coord_hvd36_hvd36_pn_rg_xy[0][1][0][0][1]=lredx_h; // H,+D coord_hvd36_hvd36_pn_rg_xy[0][2][0][0][0]=lredx_h+lredx_dp; coord_hvd36_hvd36_pn_rg_xy[0][2][0][0][1]=lredx_h;

// H,-D

coord_hvd36_hvd36_pn_rg_xy[0][2][1][0][0]=lredx_dn-lredx_h; coord_hvd36_hvd36_pn_rg_xy[0][2][1][0][1]=lredx_h;

// H,+30

coord_hvd36_hvd36_pn_rg_xy[0][3][0][0][0]=(int)rint

(lredx_2dp+lredx_h/sqrt3);

coord_hvd36_hvd36_pn_rg_xy[0][3][0][0][1]=lredx_h;

// H,-30

coord_hvd36_pn_rg_xy[0][3][1][0][0]=(int)rint(lredx_2dn-

lredx_h/sqrt3);

coord_hvd36_pn_rg_xy[0][3][1][0][1]=lredx_h;

// H,+60

coord_hvd36_hvd36_pn_rg_xy[0][4][0][0][0]=(int)rint

(lredx_3dp+lredx_h*sqrt3);

coord_hvd36_hvd36_pn_rg_xy[0][4][0][0][1]=lredx_h;

// H,-60

coord_hvd36_pn_rg_xy[0][4][1][0][0]=(int)rint(lredx_3dn-

lredx_h*sqrt3);

coord_hvd36_hvd36_pn_rg_xy[0][4][1][0][1]=lredx_h;

// V,+D

coord_hvd36_hvd36_pn_rg_xy[1][2][0][0][0]=lredx_v;

coord_hvd36_pn_rg_xy[1][2][0][0][1]=lredx_v-lredx_dp;

// V,-D

coord_hvd36_pn_rg_xy[1][2][1][0][0]=lredx_v;

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coord_hvd36_pn_rg_xy[1][2][1][0][1]=lredx_dn-lredx_v;

// V,+30

coord_hvd36_hvd36_pn_rg_xy[1][3][0][0][0]=lredx_v;

coord_hvd36_hvd36_pn_rg_xy[1][3][0][0][1]=(int)rint(sqrt3*(lredx_v-

lredx_2dp));

// V,-30

coord_hvd36_hvd36_pn_rg_xy[1][3][1][0][0]=lredx_v;

coord_hvd36_hvd36_pn_rg_xy[1][3][1][0][1]=(int)rint(sqrt3*(lredx_2dn-

lredx_v));

// V,+60

coord_hvd36_hvd36_pn_rg_xy[1][4][0][0][0]=lredx_v;

coord_hvd36_hvd36_pn_rg_xy[1][4][0][0][1]=(int)rint((lredx_v-lredx_3dp)/
sqrt3);

// V,-60

```
coord_hvd36_hvd36_pn_rg_xy[1][4][1][0][0]=lredx_v;
```

coord_hvd36_hvd36_pn_rg_xy[1][4][1][0][1]=(int)rint((lredx_3dn-lredx_v)/ sqrt3);

// +D,-D

coord_hvd36_pn_rg_xy[2][2][1][0][0]=(int)rint((lredx_dn-lredx_dp)/ 2+lredx_dp);

coord_hvd36_hvd36_pn_rg_xy[2][2][1][0][1]=(int)rint((lredx_dn-lredx_dp)/

2);

// +D,-30

coord_hvd36_pn_rg_xy[2][3][1][0][0]=(int)rint

((lredx_dp+sqrt3*lredx_2dn)/(1+sqrt3));

coord_hvd36_hvd36_pn_rg_xy[2][3][1][0][1]=(int)rint((lredx_2dn-lredx_dp)/

- 173 -

(1+1/sqrt3));

// +D,-60

coord_hvd36_hvd36_pn_rg_xy[2][4][1][0][0]=(int)rint

((lredx_3dn+sqrt3*lredx_dp)/(1+sqrt3));

coord_hvd36_hvd36_pn_rg_xy[2][4][1][0][1]=(int)rint((lredx_3dn-lredx_dp)/

(1+sqrt3));

// -D,+30

coord_hvd36_hvd36_pn_rg_xy[2][3][0][0][0]=(int)rint

((lredx_dn+sqrt3*lredx_2dp)/(1+sqrt3));

coord_hvd36_hvd36_pn_rg_xy[2][3][0][0][1]=(int)rint((lredx_dn-lredx_2dp)/

(1+1/sqrt3));

// -D,+60

coord_hvd36_hvd36_pn_rg_xy[2][4][0][1][0]=(int)rint

((sqrt3*lredx_dn+lredx_3dp)/(1+sqrt3));

coord_hvd36_hvd36_pn_rg_xy[2][4][0][1][1]=(int)rint((lredx_dn-lredx_3dp)/

(1+sqrt3));

// +30,-30

coord_hvd36_hvd36_pn_rg_xy[3][3][1][1][0]=(int)rint((lredx_2dn-lredx_2dp)/ 2+lredx_2dp);

coord_hvd36_hvd36_pn_rg_xy[3][3][1][1][1]=(int)rint(sqrt3/2*(lredx_2dnlredx_2dp));

// +30,-60

coord_hvd36_hvd36_pn_rg_xy[3][4][1][1][0]=(int)rint

 $((lredx_3dn+3*lredx_2dp)/4);$

coord_hvd36_hvd36_pn_rg_xy[3][4][1][1][1]=(int)rint((lredx_3dn-lredx_2dp)/ (sqrt3+1/sqrt3));

- 174 -

// -30,+60

coord_hvd36_pn_rg_xy[3][4][0][1][0]=(int)rint

 $((3*lredx_2dn+lredx_3dp)/4);$

coord_hvd36_hvd36_pn_rg_xy[3][4][0][1][1]=(int)rint((lredx_2dn-lredx_3dp)/ (sqrt3+1/sqrt3));

// +60,-60

coord_hvd36_hvd36_pn_rg_xy[4][4][1][1][0]=(int)rint((lredx_3dn-lredx_3dp)/ 2+lredx_3dp);

coord_hvd36_hvd36_pn_rg_xy[4][4][1][1][1]=(int)rint((lredx_3dn-lredx_3dp)/(2*sqrt3));

//Green

// H,V

coord_hvd36_hvd36_pn_rg_xy[0][1][0][1][0]=lgreenx_v;

coord_hvd36_hvd36_pn_rg_xy[0][1][0][1][1]=lgreenx_h;

// H,+D

coord_hvd36_hvd36_pn_rg_xy[0][2][0][1][0]=lgreenx_h+lgreenx_dp;

coord_hvd36_pn_rg_xy[0][2][0][1][1]=lgreenx_h;

// H,-D

coord_hvd36_pn_rg_xy[0][2][1][1][0]=lgreenx_dn-lgreenx_h;

coord_hvd36_hvd36_pn_rg_xy[0][2][1][1][1]=lgreenx_h;

// H**,**+30

coord_hvd36_pn_rg_xy[0][3][0][1][0]=(int)rint

(lgreenx_2dp+lgreenx_h/sqrt3);

coord_hvd36_hvd36_pn_rg_xy[0][3][0][1][1]=lgreenx_h;

// H,-30

coord_hvd36_pn_rg_xy[0][3][1][1][0]=(int)rint(lgreenx_2dn-

- 175 -

lgreenx_h/sqrt3);

coord_hvd36_hvd36_pn_rg_xy[0][3][1][1][1]=lgreenx_h;

// H,+60

coord_hvd36_hvd36_pn_rg_xy[0][4][0][1][0]=(int)rint

(lgreenx_3dp+lgreenx_h*sqrt3);

coord_hvd36_pn_rg_xy[0][4][0][1][1]=lgreenx_h;

// H,-60

coord_hvd36_pn_rg_xy[0][4][1][1][0]=(int)rint(lgreenx_3dn-

lgreenx_h*sqrt3);

coord_hvd36_pn_rg_xy[0][4][1][1][1]=lgreenx_h;

// V,+D

coord_hvd36_hvd36_pn_rg_xy[1][2][0][1][0]=lgreenx_v;

coord_hvd36_pn_rg_xy[1][2][0][1][1]=lgreenx_v-lgreenx_dp;

// V,-D

coord_hvd36_hvd36_pn_rg_xy[1][2][1][1][0]=lgreenx_v;

coord_hvd36_pn_rg_xy[1][2][1][1]=lgreenx_dn-lgreenx_v;

// V,+30

coord_hvd36_hvd36_pn_rg_xy[1][3][0][1][0]=lgreenx_v;

coord_hvd36_hvd36_pn_rg_xy[1][3][0][1][1]=(int)rint(sqrt3*(lgreenx_vlgreenx_2dp));

// V,-30

coord_hvd36_hvd36_pn_rg_xy[1][3][1][1][0]=1greenx_v;

coord_hvd36_hvd36_pn_rg_xy[1][3][1][1][1]=(int)rint(sqrt3*(lgreenx_2dnlgreenx_v));

// V,+60

coord_hvd36_hvd36_pn_rg_xy[1][4][0][1][0]=lgreenx_v;

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coord_hvd36_pn_rg_xy[1][4][0][1][1]=(int)rint((lgreenx_v-

lgreenx_3dp)/sqrt3);

// V,-60

coord_hvd36_hvd36_pn_rg_xy[1][4][1][1][0]=lgreenx_v;

coord_hvd36_hvd36_pn_rg_xy[1][4][1][1]=(int)rint((lgreenx_3dnlgreenx_v)/sqrt3);

// +D,-D

coord_hvd36_hvd36_pn_rg_xy[2][2][1][1][0]=(int)rint((lgreenx_dnlgreenx_dp)/2+lgreenx_dp);

coord_hvd36_hvd36_pn_rg_xy[2][2][1][1]=(int)rint((lgreenx_dnlgreenx_dp)/2);

// +D,-30

coord_hvd36_pn_rg_xy[2][3][1][1][0]=(int)rint

((lgreenx_dp+sqrt3*lgreenx_2dn)/(1+sqrt3));

coord_hvd36_pn_rg_xy[2][3][1][1]=(int)rint((lgreenx_2dn-

```
lgreenx_dp)/(1+1/sqrt3));
```

// +D,-60

coord_hvd36_pn_rg_xy[2][4][1][1][0]=(int)rint

((lgreenx_3dn+sqrt3*lgreenx_dp)/(1+sqrt3));

coord_hvd36_hvd36_pn_rg_xy[2][4][1][1]=(int)rint((lgreenx_3dn-lgreenx_dp)/(1+sqrt3));

// -D,+30

coord_hvd36_hvd36_pn_rg_xy[2][3][0][1][0]=(int)rint

((lgreenx_dn+sqrt3*lgreenx_2dp)/(1+sqrt3));

coord_hvd36_hvd36_pn_rg_xy[2][3][0][1][1]=(int)rint((lgreenx_dnlgreenx_2dp)/(1+1/sqrt3)); // -D,+60

coord_hvd36_hvd36_pn_rg_xy[2][4][0][1][0]=(int)rint

((sqrt3*lgreenx_dn+lgreenx_3dp)/(1+sqrt3));

coord_hvd36_hvd36_pn_rg_xy[2][4][0][1][1]=(int)rint((lgreenx_dn-

lgreenx_3dp)/(1+sqrt3));

// +30,-30

coord_hvd36_pn_rg_xy[3][3][1][1][0]=(int)rint((lgreenx_2dn-

lgreenx_2dp)/2+lgreenx_2dp);

coord_hvd36_hvd36_pn_rg_xy[3][3][1][1][1]=(int)rint(sqrt3/2*(lgreenx_2dn-

lgreenx_2dp));

// +30,-60

coord_hvd36_hvd36_pn_rg_xy[3][4][1][1][0]=(int)rint

```
((lgreenx_3dn+3*lgreenx_2dp)/4);
```

coord_hvd36_pn_rg_xy[3][4][1][1]=(int)rint((lgreenx_3dn-

lgreenx_2dp)/(sqrt3+1/sqrt3));

// -30,+60

coord_hvd36_pn_rg_xy[3][4][0][1][0]=(int)rint

```
((3*lgreenx_2dn+lgreenx_3dp)/4);
```

coord_hvd36_pn_rg_xy[3][4][0][1][1]=(int)rint((lgreenx_2dn-

```
lgreenx_3dp)/(sqrt3+1/sqrt3));
```

// +60,-60

coord_hvd36_pn_rg_xy[4][4][1][1][0]=(int)rint((lgreenx_3dn-

lgreenx_3dp)/2+lgreenx_3dp);

coord_hvd36_hvd36_pn_rg_xy[4][4][1][1][1]=(int)rint((lgreenx_3dnlgreenx_3dp)/(2*sqrt3));

//Calc mean

int ccumumean_xy_rg[2][2];

int cmean_xy_rg[2][2];

ccumumean_xy_rg[0][0]=0;

cmean_xy_rg[0][0]=0;

ccumumean_xy_rg[0][1]=0;

cmean_xy_rg[0][1]=0;

ccumumean_xy_rg[1][0]=0;

cmean_xy_rg[1][0]=0;

ccumumean_xy_rg[1][1]=0;

cmean_xy_rg[1][1]=0;

//Red

// H,V

ccumumean_xy_rg[0][0]+=coord_hvd36_hvd36_pn_rg_xy[0][1][0][0][0]; ccumumean_xy_rg[1][0]+=coord_hvd36_hvd36_pn_rg_xy[0][1][0][0][1]; // H,+D

ccumumean_xy_rg[0][0]+=coord_hvd36_hvd36_pn_rg_xy[0][2][0][0][0]; ccumumean_xy_rg[1][0]+=coord_hvd36_hvd36_pn_rg_xy[0][2][0][0][1]; // H,-D

ccumumean_xy_rg[0][0]+=coord_hvd36_hvd36_pn_rg_xy[0][2][1][0][0]; ccumumean_xy_rg[1][0]+=coord_hvd36_hvd36_pn_rg_xy[0][2][1][0][1]; // H,+30

ccumumean_xy_rg[0][0]+=coord_hvd36_hvd36_pn_rg_xy[0][3][0][0][0]; ccumumean_xy_rg[1][0]+=coord_hvd36_hvd36_pn_rg_xy[0][3][0][0][1]; // H,-30

ccumumean_xy_rg[0][0]+=coord_hvd36_hvd36_pn_rg_xy[0][3][1][0][0];

ccumumean_xy_rg[1][0]+=coord_hvd36_hvd36_pn_rg_xy[0][3][1][0][1]; // H,+60

ccumumean_xy_rg[0][0]+=coord_hvd36_hvd36_pn_rg_xy[0][4][0][0][0]; ccumumean_xy_rg[1][0]+=coord_hvd36_hvd36_pn_rg_xy[0][4][0][0][1]; // H,-60

ccumumean_xy_rg[0][0]+=coord_hvd36_hvd36_pn_rg_xy[0][4][1][0][0]; ccumumean_xy_rg[1][0]+=coord_hvd36_hvd36_pn_rg_xy[0][4][1][0][1]; // V,+D

ccumumean_xy_rg[0][0]+=coord_hvd36_hvd36_pn_rg_xy[1][2][0][0][0]; ccumumean_xy_rg[1][0]+=coord_hvd36_hvd36_pn_rg_xy[1][2][0][0][1]; // V,-D

ccumumean_xy_rg[0][0]+=coord_hvd36_hvd36_pn_rg_xy[1][2][1][0][0]; ccumumean_xy_rg[1][0]+=coord_hvd36_hvd36_pn_rg_xy[1][2][1][0][1]; // V,+30

ccumumean_xy_rg[0][0]+=coord_hvd36_hvd36_pn_rg_xy[1][3][0][0][0]; ccumumean_xy_rg[1][0]+=coord_hvd36_hvd36_pn_rg_xy[1][3][0][0][1]; // V,-30

ccumumean_xy_rg[0][0]+=coord_hvd36_hvd36_pn_rg_xy[1][3][1][0][0]; ccumumean_xy_rg[1][0]+=coord_hvd36_hvd36_pn_rg_xy[1][3][1][0][1]; // V,+60

ccumumean_xy_rg[0][0]+=coord_hvd36_hvd36_pn_rg_xy[1][4][0][0][0]; ccumumean_xy_rg[1][0]+=coord_hvd36_hvd36_pn_rg_xy[1][4][0][0][1]; // V,-60

ccumumean_xy_rg[0][0]+=coord_hvd36_hvd36_pn_rg_xy[1][4][1][0][0]; ccumumean_xy_rg[1][0]+=coord_hvd36_hvd36_pn_rg_xy[1][4][1][0][1];
// +D,-D

ccumumean_xy_rg[0][0]+=coord_hvd36_hvd36_pn_rg_xy[2][2][1][0][0]; ccumumean_xy_rg[1][0]+=coord_hvd36_hvd36_pn_rg_xy[2][2][1][0][1]; //+D,-30

ccumumean_xy_rg[0][0]+=coord_hvd36_hvd36_pn_rg_xy[2][3][1][0][0]; ccumumean_xy_rg[1][0]+=coord_hvd36_hvd36_pn_rg_xy[2][3][1][0][1]; //+D,-60

ccumumean_xy_rg[0][0]+=coord_hvd36_hvd36_pn_rg_xy[2][4][1][0][0]; ccumumean_xy_rg[1][0]+=coord_hvd36_hvd36_pn_rg_xy[2][4][1][0][1]; // -D,+30

ccumumean_xy_rg[0][0]+=coord_hvd36_hvd36_pn_rg_xy[2][3][0][0][0]; ccumumean_xy_rg[1][0]+=coord_hvd36_hvd36_pn_rg_xy[2][3][0][0][1]; // -D,+60

ccumumean_xy_rg[0][0]+=coord_hvd36_hvd36_pn_rg_xy[2][4][0][0][0]; ccumumean_xy_rg[1][0]+=coord_hvd36_hvd36_pn_rg_xy[2][4][0][0][1]; //+30,-30

ccumumean_xy_rg[0][0]+=coord_hvd36_hvd36_pn_rg_xy[3][3][1][0][0]; ccumumean_xy_rg[1][0]+=coord_hvd36_hvd36_pn_rg_xy[3][3][1][0][1]; //+30,-60

ccumumean_xy_rg[0][0]+=coord_hvd36_hvd36_pn_rg_xy[3][4][1][0][0]; ccumumean_xy_rg[1][0]+=coord_hvd36_hvd36_pn_rg_xy[3][4][1][0][1]; // -30,+60

ccumumean_xy_rg[0][0]+=coord_hvd36_hvd36_pn_rg_xy[3][4][0][0][0]; ccumumean_xy_rg[1][0]+=coord_hvd36_hvd36_pn_rg_xy[3][4][0][0][1]; //+60,-60 ccumumean_xy_rg[0][0]+=coord_hvd36_hvd36_pn_rg_xy[4][4][1][0][0]; ccumumean_xy_rg[1][0]+=coord_hvd36_hvd36_pn_rg_xy[4][4][1][0][1]; cmean_xy_rg[0][0]=(int)rint(ccumumean_xy_rg[0][0]/22); cmean_xy_rg[1][0]=(int)rint(ccumumean_xy_rg[1][0]/22);

//Green

// H,V

ccumumean_xy_rg[0][1]+=coord_hvd36_hvd36_pn_rg_xy[0][1][0][1][0]; ccumumean_xy_rg[1][1]+=coord_hvd36_hvd36_pn_rg_xy[0][1][0][1][1]; // H,+D

ccumumean_xy_rg[0][1]+=coord_hvd36_hvd36_pn_rg_xy[0][2][0][1][0]; ccumumean_xy_rg[1][1]+=coord_hvd36_hvd36_pn_rg_xy[0][2][0][1][1]; // H,-D

ccumumean_xy_rg[0][1]+=coord_hvd36_hvd36_pn_rg_xy[0][2][1][1][0]; ccumumean_xy_rg[1][1]+=coord_hvd36_hvd36_pn_rg_xy[0][2][1][1][1]; // H,+30

ccumumean_xy_rg[0][1]+=coord_hvd36_hvd36_pn_rg_xy[0][3][0][1][0]; ccumumean_xy_rg[1][1]+=coord_hvd36_hvd36_pn_rg_xy[0][3][0][1][1]; // H,-30

ccumumean_xy_rg[0][1]+=coord_hvd36_pn_rg_xy[0][3][1][1][0]; ccumumean_xy_rg[1][1]+=coord_hvd36_hvd36_pn_rg_xy[0][3][1][1][1]; // H,+60

ccumumean_xy_rg[0][1]+=coord_hvd36_hvd36_pn_rg_xy[0][4][0][1][0]; ccumumean_xy_rg[1][1]+=coord_hvd36_hvd36_pn_rg_xy[0][4][0][1][1]; // H,-60 ccumumean_xy_rg[0][1]+=coord_hvd36_hvd36_pn_rg_xy[0][4][1][1][0]; ccumumean_xy_rg[1][1]+=coord_hvd36_hvd36_pn_rg_xy[0][4][1][1][1]; // V,+D

ccumumean_xy_rg[0][1]+=coord_hvd36_hvd36_pn_rg_xy[1][2][0][1][0]; ccumumean_xy_rg[1][1]+=coord_hvd36_hvd36_pn_rg_xy[1][2][0][1][1]; // V,-D

ccumumean_xy_rg[0][1]+=coord_hvd36_hvd36_pn_rg_xy[1][2][1][1][0]; ccumumean_xy_rg[1][1]+=coord_hvd36_hvd36_pn_rg_xy[1][2][1][1][1]; // V,+30

ccumumean_xy_rg[0][1]+=coord_hvd36_hvd36_pn_rg_xy[1][3][0][1][0]; ccumumean_xy_rg[1][1]+=coord_hvd36_hvd36_pn_rg_xy[1][3][0][1][1]; // V,-30

ccumumean_xy_rg[0][1]+=coord_hvd36_hvd36_pn_rg_xy[1][3][1][1][0]; ccumumean_xy_rg[1][1]+=coord_hvd36_hvd36_pn_rg_xy[1][3][1][1][1]; // V,+60

ccumumean_xy_rg[0][1]+=coord_hvd36_hvd36_pn_rg_xy[1][4][0][1][0]; ccumumean_xy_rg[1][1]+=coord_hvd36_hvd36_pn_rg_xy[1][4][0][1][1]; // V,-60

ccumumean_xy_rg[0][1]+=coord_hvd36_hvd36_pn_rg_xy[1][4][1][1][0]; ccumumean_xy_rg[1][1]+=coord_hvd36_hvd36_pn_rg_xy[1][4][1][1][1]; // +D,-D

ccumumean_xy_rg[0][1]+=coord_hvd36_hvd36_pn_rg_xy[2][2][1][1][0]; ccumumean_xy_rg[1][1]+=coord_hvd36_hvd36_pn_rg_xy[2][2][1][1][1]; //+D,-30

ccumumean_xy_rg[0][1]+=coord_hvd36_hvd36_pn_rg_xy[2][3][1][1][0];

ccumumean_xy_rg[1][1]+=coord_hvd36_hvd36_pn_rg_xy[2][3][1][1][1]; //+D,-60

ccumumean_xy_rg[0][1]+=coord_hvd36_hvd36_pn_rg_xy[2][4][1][1][0]; ccumumean_xy_rg[1][1]+=coord_hvd36_hvd36_pn_rg_xy[2][4][1][1][1]; // -D,+30

ccumumean_xy_rg[0][1]+=coord_hvd36_hvd36_pn_rg_xy[2][3][0][1][0]; ccumumean_xy_rg[1][1]+=coord_hvd36_hvd36_pn_rg_xy[2][3][0][1][1]; // -D,+60

ccumumean_xy_rg[0][1]+=coord_hvd36_hvd36_pn_rg_xy[2][4][0][1][0]; ccumumean_xy_rg[1][1]+=coord_hvd36_hvd36_pn_rg_xy[2][4][0][1][1]; //+30,-30

ccumumean_xy_rg[0][1]+=coord_hvd36_hvd36_pn_rg_xy[3][3][1][1][0]; ccumumean_xy_rg[1][1]+=coord_hvd36_hvd36_pn_rg_xy[3][3][1][1][1]; // +30,-60

ccumumean_xy_rg[0][1]+=coord_hvd36_hvd36_pn_rg_xy[3][4][1][1][0]; ccumumean_xy_rg[1][1]+=coord_hvd36_hvd36_pn_rg_xy[3][4][1][1][1]; // -30,+60

ccumumean_xy_rg[0][1]+=coord_hvd36_hvd36_pn_rg_xy[3][4][0][1][0]; ccumumean_xy_rg[1][1]+=coord_hvd36_hvd36_pn_rg_xy[3][4][0][1][1]; //+60,-60

ccumumean_xy_rg[0][1]+=coord_hvd36_hvd36_pn_rg_xy[4][4][1][1][0]; ccumumean_xy_rg[1][1]+=coord_hvd36_hvd36_pn_rg_xy[4][4][1][1][1]; cmean_xy_rg[0][1]=(int)rint(ccumumean_xy_rg[0][1]/22); cmean_xy_rg[1][1]=(int)rint(ccumumean_xy_rg[1][1]/22);

//Discard obvioslly faulty points (can sometimes appear due to internal

reflections

//FIXME: setting red midpoint to green midpoint for now cmean_xy_rg[0][0]=(int)rint(ccumumean_xy_rg[0][1]/22); cmean_xy_rg[1][0]=(int)rint(ccumumean_xy_rg[1][1]/22); //fprintf(stdout,"Midpoint Green:\t %03i %03i\n",\

cmean xy_rg[0][0], $\$

 $cmean_xy_rg[1][0]$

);

//fprintf(stdout,"Midpoint Red :\t %03i %03i\n",\

cmean_xy_rg[0][1],\

cmean_xy_rg[1][1]

);

//Calc which side is closest to center for deciding (half)side of cube.

int cubeside_xy_rg[2][2];

int cube_half_side_r=0;

int cube_half_side_g=0;

if $(\operatorname{cmean}_{xy}_{rg}[0][1] \le 640 - \operatorname{cmean}_{xy}_{rg}[0][1])$

cubeside_xy_rg[0][1]=cmean_xy_rg[0][1];

if $(\text{cmean}_xy_rg[0][1] > 640\text{-cmean}_xy_rg[0][1])$

cubeside_xy_rg[0][1]=640-cmean_xy_rg[0][1];

if (cmean_xy_rg[1][1] <= 480-cmean_xy_rg[1][1])

cubeside_xy_rg[1][1]=cmean_xy_rg[1][1];

if $(\text{cmean}_xy_rg[1][1] > 480\text{-cmean}_xy_rg[1][1])$

cubeside_xy_rg[1][1]=480-cmean_xy_rg[1][1];

if (cubeside_xy_rg[1][1] <= cubeside_xy_rg[0][1])

cube_half_side_g=cubeside_xy_rg[1][1];

if (cubeside_xy_rg[1][1] > cubeside_xy_rg[0][1])
 cube_half_side_g=cubeside_xy_rg[0][1];

//fprintf(stdout,"cube(half)side:\t %03i\n",\

cube_half_side\

);

//Transfer data array to smaller array using quadrant symmetry

//fprintf(stdout,"start x:\t%03i end x:\t%03i start y:\t%03i end y:\t%03i\n",\

cmean_xy_rg[0][1],cube_half_side+cmean_xy_rg[0][1],\

cmean_xy_rg[1][1],cube_half_side+cmean_xy_rg[1][1]\

);

//Save a copy of the original array to data_orig

//unsigned int data_orig[3][640][480];

//memcpy(data_orig,data,sizeof(data));

// Add quadrant 1 to 4 and 2 to 3 (and mirror array in yplane and set x_min=0
int temp_array_1[2][cube_half side g][2*cube half side g];

for(j=cmean_xy_rg[1][1]-cube_half_side_g+1;j<cmean_xy_rg[1][1]

+cube_half_side_g;j++){

 $for(k=cmean_xy_rg[0][1];k<cmean_xy_rg[0][1]+cube_half_side_g;k++){$

data[1][k][j]+=data[1][2*cmean_xy_rg[0][1]-k][j];

temp_array_1[1][k-cmean_xy_rg[0][1]][j-cmean_xy_rg[1][1]

+cube_half_side_g-1]=data[1][k][j];

data[0][k][j]+=data[0][2*cmean_xy_rg[0][0]-k][j];

temp_array_1[0][k-cmean_xy_rg[0][0]][j-cmean_xy_rg[1][0]

```
+cube_half_side_g-1]=data[0][k][j];
```

```
//fprintf(stdout,"x y :\t %03i %03i %03i %03i %03i %03i %06i %06i\n",\
```

k,j,∖

```
2*cmean_xy_rg[0][1]-k,j,
```

```
k-cmean_xy_rg[0][1],j-cmean_xy_rg[1][1]+cube_half_side-1,\
```

temp_array_1[1][k-cmean_xy_rg[0][1]][j-cmean_xy_rg[1][1]+cube_half_side-1],\

```
data[1][k][j]
    );
}
//getc(stdin);
}
//Do the same for y
int temp_array_2[2][cube_half_side_g][cube_half_side_g];
for(k=0;k<cube_half_side_g;k++){
for(j=cube half side g;j<2*cube half side g;j++){
temp_array_1[1][k][j-1]+=temp_array_1[1][k][2*cube half side g-j-1];
temp_array_2[1][k][j-cube_half_side_g]=temp_array_1[1][k][j-1];
temp_array_1[0][k][j-1]+=temp_array_1[0][k][2*cube_half_side_g-j-1];
temp_array_2[0][k][j-cube_half_side_g]=temp_array_1[0][k][j-1];
//fprintf(stdout,"x y :\t %03i %03i %03i %03i %03i %03i %06i %06i\n",\
k,j-1,\
k,2*cube_half_side-j-1,\
k,j-cube half side,
```

temp_array_2[1][k][j-cube_half_side],\

```
temp array 1[1][k][j-1]
    );
}
//getc(stdin);
}
//Calculate vectors from middle
double max y r,max y;
max_y=0;
max y r=0;
for(j=0;j<cube half side g;j++)
         for(k=0;k<cube half side g;k++){</pre>
         if (pow(k,2)+pow(j,2)<pow((1+j),2)){
                      temp_array_2[1][k][j]=+temp_array_2[1][j][k];
                      temp_array_2[0][k][j]=+temp_array_2[0][j][k];
                      }
         if((double)(log(temp_array_2[1][k][j])) > max y)
                      max_y = (double)(log(temp_array_2[1][k][j]));
         if((double)(log(temp_array_2[0][k][j])) > max_y_r)
                      \max_y_r = (double)(log(temp_array_2[0][k][j]));
          }
//Vectorwise mean
double cumul mean;
int vektor_x;
double vektor_y,vektor_l,slope_r,final_slope_g,final_slope_r;
double vektor_x_r,vektor_y_r;
```

vektor_x=0;

vektor_y=0;

vektor_x_r=0;

vektor_y_r=0;

vektor_l=0;

cumul_mean=0;

int max_x;

max_x=2*(int)pow(cube_half_side_g,2);

int larger_than_regression;

int smaller_than_regression;

larger_than_regression=0;

smaller_than_regression=0;

int larger_than_regression_r;

int smaller_than_regression r;

larger_than_regression_r=0;

smaller_than_regression_r=0;

slope=max_y/max_x; //initial guess

slope_r=max_y_r/max_x; //initial guess

double slope_delta=slope/2;

double slope_delta_r=slope_r/2;

int tt=0;

do{

for(j=0;j<cube_half_side_g;j++){

for(k=0;k<cube_half_side_g;k++){

if((pow(k,2)+pow(j,2)) < pow(j+1,2)){

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//linear regression is true here as half of observations is above and half is below

// set slope to max_y/max_x and then interval half iterations

//Count through the array

vektor_x=(int)pow(k,2)+(int)pow(j,2);

vektor_y=(double)(max_y-log(temp_array_2[1][k][j]+1));

vektor_x_r=pow(k,2)+pow(j,2);

vektor_y_r=(double)(max_y-log(temp_array_2[0][k][j]+1));

```
//vektor_l=sqrt(pow(pow(k,2)+pow(j,2),2)+
```

pow((double)(max_y-log(temp_array_2[1][k][j])),2));

if(vektor_y >= slope*vektor_x)larger_than_regression++;

if(vektor_y < slope*vektor_x)smaller_than_regression++;

if(vektor_y_r >= slope_r*vektor_x_r)larger_than_regression_r++;

if(vektor_y_r < slope_r*vektor_x_r)smaller_than_regression_r++;

```
if (tt==9) {
```

}

final_slope_g=slope;

}

}

}

if (tt==9) {

final_slope_r=slope_r;

}

if (larger_than_regression > smaller_than_regression) slope +=slope_delta;

if (larger_than_regression < smaller_than_regression) slope -= slope_delta;

if (larger_than_regression_r > smaller_than_regression_r) slope_r

+=slope_delta_r;

if (larger_than_regression_r < smaller_than_regression_r) slope_r

-=slope_delta_r;

slope_delta=slope_delta/2;

larger_than_regression=0;

smaller_than_regression=0;

slope_delta_r=slope_delta_r/2;

larger_than_regression_r=0;

smaller_than_regression_r=0;

tt++;

} while(tt<10);

int m,n,hypo,step=0;

double gaussian_integral_g=0.0;

double gaussian_integral_r=0.0;

double iLLPr=10, iLLP_lastr=15, iLLP_stepr=iLLPr/2;

double iLLPg=10, iLLP_lastg=15, iLLP_stepg=iLLPg/2;

cmean_xy_rg[0][0]+cube_half_side_g; n++) { // Same x

do { //Calculate red iLLP

gaussian_integral_r=0.0;

for(m =0; m < h; m++) {// For red y

for(n = 0; n < w; n++) { // Same x

hypo=(int)rint(sqrt(pow(cmean_xy_rg[0][0]-n,2)+ $\$

pow(cmean_xy_rg[1][0]-m,2)));

```
gaussian_integral_r+=exp(-final_slope_r*pow(hypo,2)+iLLPr);
         }
}
if (gaussian integral r-total integral r > 0)iLLPr-=iLLP_stepr;
if (gaussian_integral_r-total_integral_r < 0)iLLPr+=iLLP_stepr;
iLLP_stepr=iLLP_stepr/2;
iLLP lastr=iLLPr;
}while(!(total integral r==(int)rint(gaussian_integral_r)));
do {
gaussian integral g=0.0;
for(m =0; m < h; m++) {// For green y
          for(n = 0; n < w; n++) { // Same x
hypo=(int)rint(sqrt(pow(cmean xy rg[0][1]-n,2)+
          pow(cmean_xy_rg[1][1]-m,2)));
}
if (gaussian integral g-total integral g > 0)iLLPg-=iLLP stepg;
if (gaussian_integral_g-total_integral_g < 0)iLLPg+=iLLP_stepg;
iLLP_stepg=iLLP_stepg/2;
iLLP_lastg=iLLPg;
}while(!(total integral g=(int)rint(gaussian integral g)));
fprintf(stdout,"%06i\t%6.6f\t%6.6f\n",internal reflection,iLLPr,iLLPg);
return 1;
}
```

int calc_internal_reflection(unsigned int data[3][640][480]) { return 1; }