Towards real time 3D quantitative characterisation of \textit{in situ} layer growth using white light interference microscopy

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\textbf{Abstract.} Quantitative 3D characterisation of layer growth or modification \textit{in situ} in liquid systems is a challenge because of the changing morphology of the layer and the presence of the growth liquid. Because of the limited bandwidth of many surface profiling techniques, measurement of microscopic surface roughness is generally limited to that of static surfaces. The aim of the present work is to develop a new technique using high speed scanning interference microscopy combined with an adapted immersion head for use in liquid growth systems. For several years we have been developing a real time 3D surface analysis system based on a high speed camera and cabled logic processing, combined with continuous scanning white light interferometry. An optical measurement head is also being developed for use in liquid immersion conditions, with the view of measuring layer growth or modification in biomaterials. In this paper we report on the present status of the development of our second prototype 4D system and also of the optical immersion head for \textit{in situ} measurements, describing the achievements made and the difficulties still to be overcome.

1. Introduction

Many new areas of materials development such as organic electronics, microsystems, photonic crystals and biomaterials \cite{1} use some sort of layer growth and processing. Amongst the many characterisation techniques employed to study the different properties of the layers grown, 3D measurement of the surface morphology figures as a growing challenge, due to the high roughness and growing complexity of the layers. Knowledge of the roughness values, particle size, step heights, and structural shape of the layers plays a growing role in calibrating growth techniques and in understanding the functionality of the grown layers at different scales. Classical profilometry techniques such as stylus profiling and AFM generally require many seconds to many minutes or more to perform a single measurement of the 3D surface. Such a time constraint limits measurements to static samples, \textit{ex situ}.

A key aspect of achieving real time quantitative microscopy is the requirement of a very high bandwidth in the measurement system. Real time microscopy of several to tens of 2D image slices per second has been demonstrated in the area of confocal microscopy for biological specimens \cite{2} by increasing the scanning speed of the probe beam, but the 3D image acquisition time is several seconds.
or more due to the need to acquire several slices over the depth. By using probe tip resonance in AFM [3] and SNOM [4], 3D image acquisition can be increased to tens of images per second (i/s), but this is generally limited to small field sizes of 10 µm or less and with uncalibrated depth information. Several tens of i/s has been achieved in interference microscopy, but limited to the measurement of either small surface roughness (below λ/2) [5] or certain types of deeper sample structure [6]. More recently, in digital holography, a rate of 15 i/s has been demonstrated for measuring continuous smooth surfaces [7].

In interference microscopy, signal processing is generally carried out on the PC microprocessor, requiring a measurement time for a single 3D image of several seconds or minutes depending on the image size and scan depth [8]. A real time "smart pixel" CMOS camera with integrated dedicated hardware processing in pOCT (parallel optical coherence tomography) has demonstrated a 3D imaging rate of 14 i/s over a depth of 1.4 mm with a depth resolution of 4.5 µm [9]. While this technology is interesting for certain dedicated real time applications, it has the disadvantages of a small camera sensor size of 90x144 pixels, photodiodes with a very low fill factor of 10 % and lack of flexibility in terms of the choice of signal processing.

White light scanning interferometry (WLSI) is of great interest in quantitative real time microscopy because no physical scanning in the XY plane is necessary as in stylus profiling or near field microscopy, the measurement rate being limited only by the Z scan speed of the fringes, and the image acquisition and processing speeds. High depths can be measured (hundreds of µm) with an axial sensitivity of nm to tens of nm, depending on the surface roughness [8, 10]. For example, using a high speed CCD camera (250 i/s, 512x512 pixels full frame) synchronized with a continuously scanning piezo (10 Hz) and image processing carried out on a FPGA (Field Programmable Gate Array), we previously demonstrated a first prototype system for 3D measurement at a rate of 5 i/s over 3 µm [11]. In the present work we discuss the development of a second prototype based on a high speed CMOS camera (500i/s, 1280x1024 pixels full frame - 625 Mbytes/s) connected by a Cameralink bus to a PCI-X (64 bits, 66 Mhz) FPGA based vision processing board [12]. Results have been demonstrated on a laterally translated microchip.

A second difficulty of measuring layer growth is the physical growth conditions. Most of the previously mentioned techniques are generally used in dry, ex situ conditions. In situ measurement is made more complicated by the limited space available due to the growth chamber and the difficulty to approach the sample surface. In the case of growth in a liquid, the presence of the growth solution also limits the type of optics that can be used. A new optical measurement head is being developed that is suitable for use in liquid immersion conditions, with the view of measuring layer growth or modification in biomaterials [1]. In this paper we report on the present status of the development of our second prototype 4D system and also of the optical immersion head for in situ measurements, describing the achievements made and the difficulties still to be overcome.

2. Choice of algorithm for high speed processing with cabled logic
2.1. 3D measurement by fringe scanning

Optical interference microscopy makes use of the phase of the light reflected from the surface to be measured by comparing it with that of light reflected from a plane reference mirror. With the path lengths of each beam being nearly equal, a series of white light fringes are produced superimposed on an image of the surface in the plane of the camera [10]. Interference objectives typically used are of the Michelson or Mirau type that use a single objective for low to medium magnifications, or the Linnik type, that uses two separate objectives for higher magnifications [13]. The fringes are translated over the whole of the depth of the sample by varying the distance between the interference objective and the sample surface. A series of images is captured using a digital camera and the images processed to extract the fringe envelope at each pixel. The light intensity I measured in a white light interferometer that is spatially incoherent has the following form:

\[I = \frac{2}{\pi} \frac{\lambda}{d} \sin \frac{\pi d}{2} \sin \frac{\pi d}{2}\]
The $x$ and $y$ coordinates correspond to the image coordinates and the $z$ coordinate indicates the axial location of the sample surface. The quantity $I_0$ is an offset related to the reference and object beam intensities. The interferogram envelope function $\Gamma$ is related to the spectral profile of the white light and $R$ is the effect of the reflection coefficient of the sample. Detection of the peak of the fringe intensity or the fringe envelope at a pixel over the depth scanned determines the position of the surface at that point. Many different signal processing techniques for determining the envelope peak have been proposed in the literature, varying in mathematical complexity, computational cost, processing time, robustness and axial measurement uncertainty. Some of the more computationally costly algorithms include demodulation, correlation, determination of the barycenter of the envelope, Fourier, Hilbert and wavelet techniques [13]. These techniques are suitable for static measurement (or post-processing after acquisition at high speed) where a processing time above one second is sufficient. On the contrary, for real time on-line measurements in which processing needs to be carried out "on-the-fly" at less than a second per 3D image, the use of complex mathematical functions in a microprocessor in a PC, a DSP or a GPU is not possible at present above a 2D image rate of around 90 i/s.

Processing images from a high speed camera above 500 i/s requires the use of cabled logic processing, for example using a FPGA. The choice of the best algorithm is then mainly based on the possibility of integrating it into the FPGA, which reduces the availability of mathematical functions to basic operators such as adders, multipliers and comparators, together with memory requirement. Two of the simpler algorithms proposed in the literature, peak fringe scanning microscopy (PFSM) and the determination of the fringe visibility (FSA) are now described.

2.2. **PFSM algorithm**

The PFSM algorithm [14] is based on the detection of the highest intensity of the fringe signal along the optical axis, which corresponds to the peak of the central zero-order fringe as long as the background intensity is always less than this value. This algorithm requires a first memory to record the position of the maximum for each pixel (altitude image) and a second memory for the current value of the maximum intensity (which gives the reflection image). The interest of this algorithm lies in its simplicity, requiring only one comparator, a limited number of images stored in on board memory and thus a considerable gain in processing time and storage space. It is well adapted for implementation on a FPGA. The axial resolution depends on the displacement step between images, with typical values being between 20 nm and 80 nm.

2.3. **FSA algorithm**

The five sample adaptive (FSA) algorithm [15] is developed from a generalized form of the well known five-step phase-shifting algorithm used in monochromatic interferometry. Rather than measuring the phase, the modulation of the fringes is determined by measuring a series of five consecutive points along the $Z$ axis at each pixel location. Based on the Hilbert approximation, an analytical expression of the modulation function can be established, given by the following expression:

$$M = C \sqrt{((I_2 - I_3)^2 - (I_1 - I_3)(I_3 - I_5))}$$

where C is a constant of standardization. This formula is exact when the phase shift between interferograms is $90^\circ$.

Using this relation, it is possible to measure the variation in fringe visibility, to detect the envelope of the interference fringes and to detect the peak value in order to determine the $Z$ position. Although
the FSA algorithm is inevitably more complex in terms of digital architecture than the PFSM algorithm, it is nonetheless far simpler than algorithms requiring complex mathematical functions. Like the PFSM algorithm, the FSA algorithm is well adapted to implementation in a FPGA. The axial resolution of the FSA algorithm in its simplest form is limited to the step height of 90°, typically around 80 nm.

3. High speed camera acquisition and processing system

3.1. Measurement system

The real time measurement system (figure 1) consists of an adapted Leica DMR-X microscope equipped with a halogen lamp and a set of Michelson and Mirau interference objectives. Axial displacement of the sample is carried out by a piezo actuator (PI) controlled by a closed loop controller using a high accuracy capacitive position detector to achieve a sensitivity of 1 nm in the static mode. Image acquisition is performed with a high speed CMOS camera (BASLER A504k) and a CORECO "Anaconda" acquisition and processing board. An on-board FPGA is used for the high speed processing.

3.2. System synchronization

Each element of the electronic system is synchronized by a clock signal originating from a function generator developed under LabView (figure 2). This clock signal is used to trigger the camera, to control the processing rate on the imaging board, to generate the ramp signal for the piezoelectric translator and to synchronize the image acquisition and processing on the PC. Complex low and high level software has been developed under LabView and C++ to initialize and to configure the different parts of the system indicated by the dotted lines in figure 2. By this means it is thus possible to define the image size, the image acquisition rate, the exposure level, the piezo scanning rate and depth, the transfer rates to the imaging board, the type of processing on the FPGA, the 3D image rate, and the type of storage on the PC hard disc. The timing signals for synchronization of the camera, the piezo translator and the calculated 3D image are illustrated in figure 3. A symmetrical square wave is used for the timing signal and a stepped ramp signal to control the piezo translator. A single 2D image from the camera is acquired on the rising edge of the camera trigger signal.
Figure 2. Block diagram of image acquisition, processing and synchronisation

The piezo translator is displaced one step on the falling edge of the camera trigger signal. In this way image acquisition occurs during the second half of the plateau of the stepped ramp signal to the piezo translator, minimizing axial movement of the translator and optimizing fringe contrast. The previously acquired image is sent to the imaging board on the falling edge of the camera trigger signal. These operations are repeated until a complete scan in one direction is completed (rising or falling).

Figure 3. Timing signals for synchronizing different parts of the system

At the beginning of the scan in the opposite direction, the two images containing the height measurement data and the maximum intensities (reflection image) are transferred to the RAM of the PC via the PCI-X bus. Because the image data rate is far lower than the camera acquisition rate (by a factor of 50 to 200, depending on the number of images scanned along the optical axis), the PC software can be used to perform image filtering and construction of the pseudo 3D image. The image data can also be stored on hard disk. Such a system design makes it possible to perform real on-line measurements.

3.3. Implementation of algorithms in cabled logic

The two fringe processing algorithms proposed were implemented in cabled logic, firstly by simulating them in VHDL (Very High Speed Integrated Circuit Hardware Description Language) and secondly by synthesizing the optimized VHDL code to the bit stream file specific to our FPGA circuit. Although the algorithms are fairly simple, much work was required to develop a suitable architecture for the cabled logic. Many difficulties had to be overcome in interfacing between the camera and the processing board via the CameraLink connection and between the FPGA processor and the PC via the PCI-X bus and the different connections used to control each part of the system. The development work for implementing the PFSM and FSA algorithms in cabled logic was initiated in the framework of the PhD by G. Johnson [12] and then taken further and optimized by J. Montagna during the present...
Oséo project. A second option of using the minimum of the signal (black fringe) as well as the maximum (white fringe) has also been added. A «ChipScope Pro» tool was used on the FPGA chip to monitor directly the different signals and to optimize the algorithms developed.

3.4. System performance

In this section we present the practical performance of the acquisition and processing part of the system in terms of the on-line 3D image rate as a function of the scanning depth. The calculation of the performance depends on many different parameters and is complex, but the main limitation is due to data transfer rates to the FPGA and its local environment of buffers and memories.

![Graph showing 3D image rate vs. depth for different image sizes](image)

**Figure 4.** Comparison of performance of two algorithms in terms of 3D image rate as a function of scan depth and image size for a step size of 76 nm

The calculated results for the PFSM and FSA algorithms are given in figure 4 for different image sizes over an axial scan range of 1-10 µm and a step value of 76 nm between the acquired fringe images. Comparing the performance of the two algorithms, it can be noted that the 3D image rates are slightly higher for the PFSM algorithm for the same image sizes and scan depths. This improvement is due to the greater simplicity of the PFSM algorithm. For example, for a scan depth of 3 µm and an image size of 640x1024 pixels, the 3D image rate is 4.94 i/s for the FSA algorithm and 9.75 i/s for the PFSM algorithm. For the smaller image size of 160x128 pixels, the 3D image rate increases to 49.40 i/s for the FSA algorithm and 50.67 i/s for the PFSM algorithm. Whereas the step size is fixed for the FSA algorithm, for the PFSM algorithm it can be varied over a practical range of 20-80 nm, with a corresponding increase in processing time and reduction in 3D image rate for steps smaller than the example of 76 nm given.

4. Results of on-line measurements on laterally moving samples

Whilst the real time measurement system is being developed for measuring in situ layer growth, at this stage, it has been found that translating a microelectronic chip laterally using the motorized tables at different speeds is quite satisfactory for the purpose of testing the system under development. The chip used was a microfluxgate sensor, an integrated version of the fluxgate magnetic probe [16] fabricated using a process derived from a standard Bipolar process. The surface of the chip is covered with a 0.8 µm to 1.6 µm thick transparent passivation layer. The measurements in WLSI were performed on the buried interface consisting of the Silicon substrate and the metal connections.
4.1. Measurements with the PFSM algorithm

Results of measuring the microfluxgate with the PFSM algorithm for large and small image sizes for an axial step size of 40 nm are given in figure 5.

(i) \( I_0 \) at \( t = 0 \) s, \( dx = 0 \) µm

(ii) \( I_8 \) at \( t = 4.2 \) s, \( dx = 84 \) µm

(a) Large image (640x1024 pixels, x5 Michelson objective), 40 nm step over 5 µm, lateral speed 20 µm/s, sampling rate 240 i/s and a 3D image rate of 1.9 i/s

(b) Small image (320x256 pixels, x10 Mirau objective), 40 nm step over 4 µm, a lateral speed of 20 µm/s, sampling rate of 1010 i/s and a 3D image rate of 10 i/s

**Figure 5.** Two sequences of measurements for different image sizes and sampling rates with the PFSM algorithm of the microfluxgate chip being displaced sideways at different speeds

Two images at different times are presented for each size, to show the advancing chip. The full results exist in the form of a video clip. The x5 Michelson objective was used for the large image size (640x1024 pixels, 1.5x2.4 mm) with a fringe image acquisition rate of 240 i/s over a scan range of 5 µm, giving a 3D image rate of 1.9 i/s. For the small image size (320x256 pixels, 375x300 µm), the x10 Mirau objective was used with a fringe image acquisition rate of 1010 i/s over a scan range of 4 µm, giving a 3D image rate of 10 i/s. The results contained much less noise and artifacts than the measurements made with the first prototype [5]. The most significant artifacts remaining were scattered bumps \( \lambda/2 \) in height due to secondary fringe detection (visible in figure 5(b)(ii)), mainly arising from the reduced signal to noise ratio in measuring the buried interface. The results would be "cleaner" on a bare chip without a passivation layer. Otherwise, the axial measurement uncertainty is limited by the scanning step, which is 40 nm in these examples. Slope artifacts are also visible on leading and trailing square step edges due to motion blur.

4.2. Measurements with the FSA algorithm

Results of measuring the microfluxgate with the FSA algorithm for large and small image sizes for an axial step size of 76 nm are given in figure 6.
Figure 6. Two sequences of measurements for different images sizes and sampling rates with the FSA algorithm of the microfluxgate chip being displaced sideways at different speeds (x5 Michelson objective, axial range of 5 µm)

For both image sizes, the x5 Michelson objective was used with a 5 µm scan. A fringe acquisition image rate of 190 i/s was used for the large image size (640x1024 pixels, 1.5x2.4 mm), giving a 3D image rate of 2.96 i/s. For the small image size (320x256 pixels, 750x600 µm), a fringe image acquisition rate of 924 i/s was used, giving a 3D image rate of 20.52 i/s. The measurements contained less axial noise than those made with the PFSM algorithm. This is to be expected as the FSA algorithm is more robust than the PFSM algorithm. The axial measurement uncertainty is again limited by the scanning step, which in this case is 76 nm. Slope artifacts are also visible on leading and trailing square step edges due to motion blur.

4.3. Immersion head for in situ growth measurement

Hydroxyapatite (HA) is the mineral part of bones, teeth and shells. Growth of thick layers (1-20 µm) of synthetic HA is important for medical implants and biosensors in the field of new biomaterials. The ISSP of the Bulgarian Academy of Sciences has developed a novel growth method (laser-liquid-solid interaction) in which substrates (steel, Si, glass, polymers...) are immersed in a simulated body fluid solution for layer growth. The effects of laser illumination, surface nano-structuring and the addition of diamond nanoparticles are being studied [1]. These layers are characterized using a series of
classical techniques (SEM, FTIR, AFM, XRD…). The surface roughness is measured at InESS using WLSI that has been optimized for these particular layers that are thick, rough and translucent (figure 7(b)). The aspect is very similar to that found with MEB and AFM, the layer consisting of micron sized sphere-like nodules.

In order to be able to measure the growth of such layers in situ, an immersion head is being specifically developed, shown in figure 7(a). A pair of immersion objectives is used in a Linnik configuration. The sample observation objective will be placed in a protective head containing distilled water and a window that will be immersed in the SBF solution. In this way, the observation window can be cleaned or replaced between growth experiments. The head is still under development. The combination of the real time system and immersion head will enable the study of the growth of HA layers in situ.

**Figure 7.** Future adapted immersion Linnik interferometer head (a) for characterizing in situ layer growth in liquid conditions (b)

5. Conclusions

The latest results of a new system for measuring real time 3D surface relief for layer growth in situ using continuously scanned WLSI has been presented. The system consists of a high speed CMOS camera and cabled logic image processing system.

Two different algorithms for fringe processing have been implemented on the FPGA based board and characterized in terms of performance. The fastest algorithm is by simple detection of the peak intensity (PFSM), giving 3D image rates of 2.93 i/s to 152 i/s for image sizes of between 640x1024 pixels and 160x128 pixels and scan depths of 1 to 10 µm. For the same sampling step of 76 nm, an algorithm based on the detection of the fringe visibility (FSA) gives slightly slower 3D image rates of 1.48 i/s to 148.20 i/s for similar conditions. Tests performed on a microfluxgate chip being translated sideways between speeds of 20 µm/s and 200 µm/s demonstrated successful on-line measurements over a scan depth of 4-5 µm and 3D image rates of between 1.9 i/s and 20.52 i/s. The FSA algorithm proved to be more robust, with less artifacts than the PFSM algorithm.

Future work will consist of completing the immersion head and testing the combined system on in situ layer growth of HA layers. An improved real time camera and processor is also being developed to improve the performance rates still further.

**Acknowledgements**

This work has been supported by the Oséo Conectus project "CAM4D" and the CNRS PICS project N° 4848. Thanks are also extended to Luc Hébrard for providing the microfluxgate chip.
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