Challenges in the areal measurement of surface roughness and shape at the micro and nanoscale

Challenges in the areal measurement of surface roughness and shape at the micro and nanoscale

P C Montgomery[1*], M Guellil[1], P Pfeiffer[1], B Serio[2], F Anstotz[1], L Pramatarova[3] and S Roques[1]

[1] *Laboratoire des Sciences de l'Ingénieur, de l'Informatique et de l'Imagerie (ICube), Unistra-CNRS, UMR 7357, 23 rue du Loess, 67037 Strasbourg cedex, France.*
[2] *Laboratoire Energétique Mécanique Electromagnétisme (LEME) - EA 4416, 50 rue de Sèvres, 92410 Ville d'Avray, France.*
[3] *Institute of Solid State Physics, Bulgarian Academy of Sciences, 72 Tzarigradsko Chaussee Blvd., 1784 Sofia, Bulgaria.*
E-mail: paul.montgomery@unistra.fr

Abstract. Measuring surface roughness accurately at the micro and nano scale presents several challenges. While optical techniques can be used to rapidly measure large areas, significant variations can be found between results from different techniques on similar samples. In the present work, a comparison has been made between the results of two different systems using interference microscopy and AFM to make measurements at the same place on the same sample. Two samples were prepared on silicon wafers by marking them with a multi-scale pattern using a photoresist process of lithography from an optical mask, followed by reactive ion etching. One was left bare and the other was prepared with a rough layer of hydroxyapatite before measuring at the chosen positions. Comparison of the results showed that while the general shapes of the measured surface microstructures were similar, several differences were found. For example, there was a variation of up to 7 % between techniques in the measurement of the depths of the etched features and artefacts were also visible at square edges. These results show the need to pay careful attention to instrument calibration and probe/surface interactions in order to improve the accuracy of surface characterization of surface roughness and topography.

1. Introduction

Measuring surface roughness accurately at the micro and nano scale presents several challenges concerning the lateral resolution, the absolute height measurement, the accuracy and repeatability. Up until recently, the stylus profiler has been used to provide the most reliable measurements of surface roughness, due to its simplicity combined with the many advanced standards that now exist [1]. These ensure the uniformity of measurements and an independence of the measurements from the machine and the user. Over the past three decades, many new optical techniques as well as near field scanning techniques have been developed. Optical methods are of particular interest because of their rapidity of measurement over large areas [2]. One emerging difficulty though, is the variation in the results from stylus, near field and optical techniques when measuring similar samples due to technique dependent artefacts and probe/surface interactions. The recent ISO/CD 25178-600 standard on areal measurement has begun to address this problem [3]. The use of carefully chosen measurement standards that can be traced back to the metre is an important part of calibrating surface profilers [4].

Comparing the measurements made using different techniques on real samples is another useful way for benchmarking, to help in the understanding of the performance of each technique and to improve the overall measurement precision in real life applications. Such a study that was carried out within a European project to characterize microlens arrays revealed some significant disparities between results from different techniques [5]. This is all the more true when the surface structure deviates from a homogeneous and flat surface to one that is heterogeneous and rough. Comparative studies of silicon and rough poly-Si have been useful in revealing different artefacts and algorithm related variations in measurements between AFM and interference microscopy [6].

Coherence scanning interferometry (CSI) or white light scanning interferomety (WLSI) as it is also known, is a powerful technique for areal surface roughness measurement today because of its high axial resolution, its versatility on different types of samples and its ease of use [7, 8]. Being an optical technique, CSI also has its limits, making it necessary to compare measurements with those from other techniques to help in the understanding of the different sources of errors such as with SEM and AFM [9]. For example, it is well known that CSI can produce artefacts in the measurement of rough layers due to the steep surfaces present [10].

Where the results of the characterization of surface topography of materials are presented in the literature using several techniques on the same sample, comparative measurements are more often made in different places [6, 11]. Taking measurements from exactly the same place on the same sample with different techniques in practice is not easy. Nonetheless, such measurements are of great importance in order to be able to characterize and to understand important new materials today such as in nano- and biomaterials. It is also important for the characterization of etching processes in micro and nanotechnologies for which the homogeneity and the aspect ratio often depends on the position of the sample in the etching device [12]. While it is already difficult to make comparative measurements for µm sized structures, the challenges increase even more when the details required are sub-µm and nanometric in size.

For measuring the same place on the same sample with different microscopy techniques, there exist three main solutions:

1. Multi-mode measurement on the same microscope (the sample is not moved).
2. Position indexing using a calibrated *XY* table or sample holder [13] (the sample is moved between different microscopes).
3. Marking the sample (the sample is moved between different microscopes).

In the present work we are particularly interested in the third solution, in marking the sample. Many different types of techniques exist for marking, such as by using ink [14], mechanical or electrochemical etching, pulsed laser ablation [15], laser marking with a photonic jet [14], thin film deposition, and photolithographic etching. The choice of marking technique depends on several factors, such as the properties of the material (physical, chemical, topographic…), the resolution of marking required and attainable on a given sample and the size of the field of view necessary for the different characterization methods employed.

In the ICube laboratory we are interested in characterizing a wide variety of materials, ranging from semiconductor crystals and microelectronic components, various organic, optical and biomaterials through to cement pastes. Tests carried out using laser marking of cement paste materials and a thin film evaporated Al layer on a Si wafer, while allowing the identification of a particular zone, did not allow a high enough resolution to be attained to be able to make the same linear profile in exactly the same areas under the microscopes.

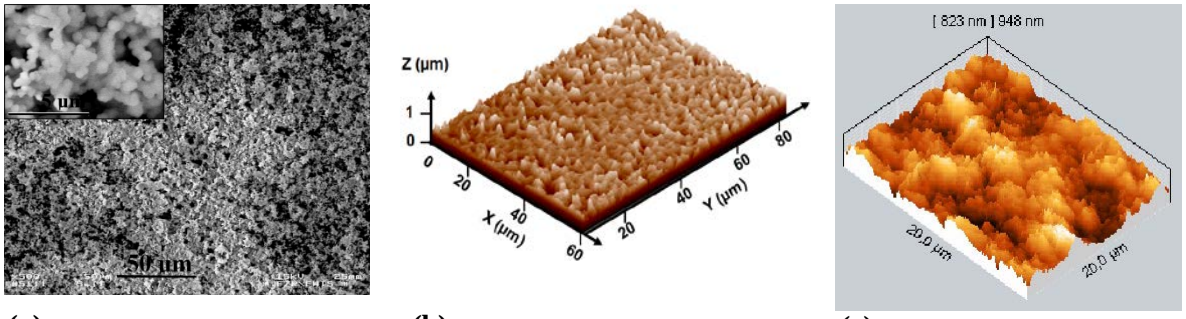
To attain a higher positional resolution, the photolithographic etching technique on silicon wafers was therefore chosen [16]. A multi-scale photolithographic mask was developed and used to mark two samples of silicon wafers with features etched to a depth of about 2.4 µm and 6.6 µm respectively. The first sample was left bare to provide a smooth surface with simple etched levels. The second was prepared with a partial layer of hydroxyapatite deposited from a solution of simulated body fluid (SBF) [17] to give a partially filled, rough layer on the smooth levels of the etched silicon surface. Using the numbered squares, the same area was then found and measured with two different systems using

interference microscopy (a Leitz-Linnik research microscope and a Zygo NewView 7200 microscope) and an AFM microscope (Park XE70). The samples were also imaged with ESEM (environmental SEM).

The measurements of the two dimensional cross sectional profiles and 3D views of the surface topography of the same area using the different techniques were then compared using the MountainsMap® 6 analytical measurement tool. The results of the comparisons are presented in order to reveal the similarities and the differences in the measurements between the different techniques. Particular attention is paid to average depth measurements of the etched features, the edge shapes and the presence of artefacts. A discussion is given concerning the different origins of the differences in measurements in terms of calibration, probe/surface interaction and the precision of placing the cursor in the data analysis software.

2. Measuring the same sample in different places

As mentioned previously, in materials characterization, comparative measurements of surface topography, if at all made are done so either on different samples that are similar or on the same sample in different areas [6, 9]. An example is given in figure 1 of the use of SEM, CSI and AFM to measure the roughness of a thick layer of HA on silicon substrate.



(a) **(b)** **(c)**

Figure 1. Comparative measurements of the surface topography of similarly prepared rough layers of hydroxyapatite on different Si substrates using (a) SEM, (b) the Leitz-Linnik microscope and (c) AFM.

Such comparative measurements are obviously useful, the different techniques often confirming the general topographic and structural characteristics of the sample surface. Nonetheless, it is clear that the results are slightly different. While the SEM image shows a higher lateral resolution and the detailed spongy nature of the HA layer together with the HA nanocrystals at the higher resolution, quantitative measurements can only be approximate. On the other hand, the CSI and AFM results provide quantitative data concerning the measured heights of the layer at different points and the roughness values [11]. Any quantitative data can only be representative of the layer. Problems arise when comparing measurements between different techniques since the values are often different due to differences between sample positions and instrument performance. Any artefacts present in the measurements would also be more difficult to detect.

By measuring the same place on the same sample, a more rigorous comparison can be carried out in the measurements between different techniques in terms of absolute values of roughness and height as well as the presence of different artefacts.

3. Measuring the same sample in the same place

In order to achieve measurement at the same place on the same sample using marking methods, various methods of marking were investigated. These included laser marking of cement paste samples, Al layer evaporation on a silicon wafer and the use of a photolithographic mask combined with etching on silicon wafers. The method used for growing the hydroxyapatite layers is also described. Details are given of the different topographic measurement techniques used, together with the analytical procedures employed to compare the different surface shape measurements.

3.1. Initial studies using laser and Al layer marking

Some first tests were carried out using laser marking on cement paste materials, consisting of a 1 mm numbered grid of 200 µm sided squares written with 30 µm wide lines (figure 2(a)). A thin film evaporated Al layer using a 1.5 mm square mask was also made on a Si wafer (figure 2(d)). While these two marking techniques allowed the successful identification of a particular zone under the optical and SEM microscopes, they could not be used to make comparisons between profiles. The resulting marks on each sample were too rough and inaccurate to be able to find the same place with sufficient precision to be able to compare the line profiles. This was more so the case for the AFM measurements on the Si sample with its limited field of view.

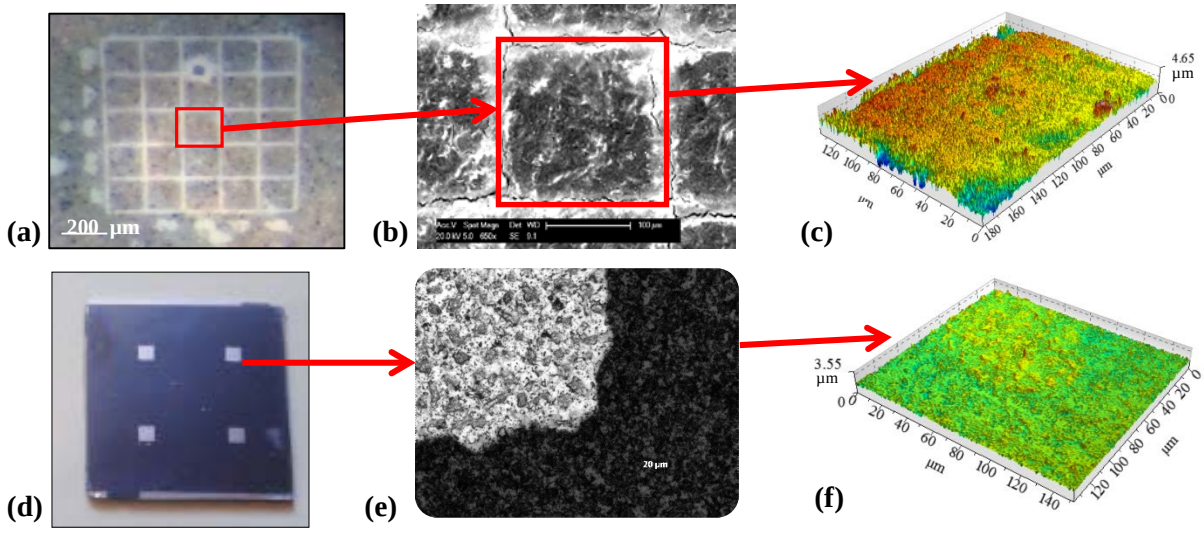


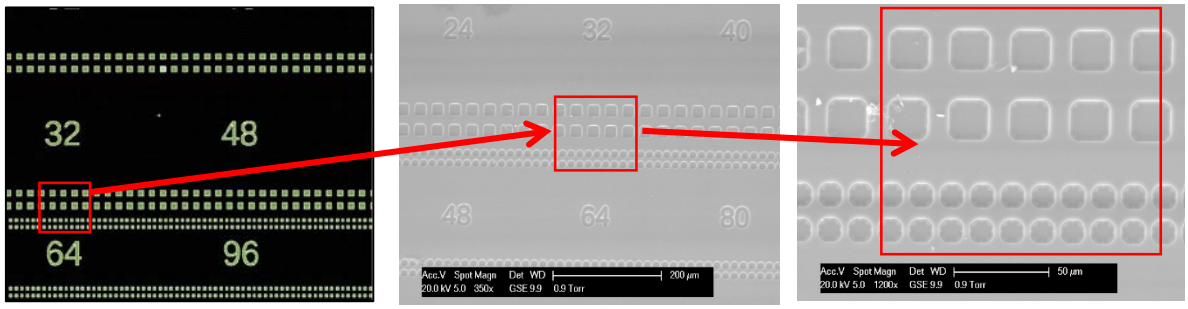
Figure 2. Comparison of results of marking using laser etching on cement (a) optical microscopy, (b) SEM, (c) the Leitz-Linnik microscope and Al layer on Si using (d) direct view, (e) optical microscopy and (f) the Leitz-Linnik microscope.

3.2. Preferred method chosen: multi-scale test pattern using photolithography

The preferred method chosen for marking was that using UV photolithography and RIE etching on smooth silicon wafers [6]. The challenge for positioning a sample under different microscopes is to have a test pattern with geometrical characteristics that allow the localization of different regions on a surface that may vary by several orders of magnitude in size, from 1 mm^2 to 1 μm^2 so that exactly the same zone can be identified.

To allow the rapid and efficient localization of a given position, a specific mask was developed based on a multi-scale geometry. The photomask was designed using LayoutEditor©, a computer-aided design software for editing MEMS designs and for use in integrated circuit fabrication. A special macro command in the C++ language was programmed to produce a multi-scale pattern of numbered squares with different geometries and sizes for a rapid and efficient localization of sample features from mm^2 to μm^2. The portion of interest is shown in figure 3(a). The test pattern was etched in two samples of silicon wafers to depths of 2.4 µm and 6.6 µm respectively. Standard photolithographic procedures were

employed, using a positive photoresist, direct contact in a mask aligner and RIE etching. The resulting etched mask pattern on one of the silicon wafers with the zone of interest is shown in figure 3(b) using ESEM.



(a) **(b)** **(c)**

Figure 3. Details of the test pattern developed, showing (a) the photolithographic test pattern developed in CAD and (b) the numbered square pattern etched in a Si wafer observed by SEM and (c) details of the exact zone measured (red square) observed by SEM.

The rough layer used consisted of hydroxyapatite (HA), an inorganic material that is chemically similar to the mineral component of bones, teeth and hard tissues in mammals ($Ca_{10}(PO_4)^6(OH)^2$). The study of synthetically grown layers of HA and composite materials incorporated with nanodiamonds, polymers, etc. is important today in the fields of human implants and new biological scaffolds [17]. Amongst the many parameters used to characterize these materials, the quantified measurement of the topology of these rough layers is a key aspect [11]. So as to be able to measure the same zone with the different techniques, the second silicon wafer etched with the square test pattern to an average depth of 6.6 µm was used as a substrate on which to grow a partial layer of HA, with enough HA to be measured while leaving spaces to be able to recognize the pattern.

The HA layer was deposited on the etched silicon using a supersaturated simulated body fluid (SBF), an aqueous solution that resembles the inorganic composition and concentration of human blood plasma. The solution was prepared by dissolving reagent-grade chemicals in doubly distilled water according to the SBF method [18]. The samples were immersed for 3 hours and 25 min in the solution under natural conditions (37°C, pH 7.4) while being mixed with a magnetic stirrer.

3.3. Growth technique used for hydroxyapatite layers

The different microscope techniques used to characterize the samples are the following:

i) Leitz-Linnik microscope

The CSI interference microscopy system used was developed at ICube, based on the principle of white light scanning interferometry and described in [6, 11]. The system is based on a Leitz-Linnik microscope equipped with a Linnik type objective (x50, NA = 0.85), giving a lateral resolution of R_{lat} = 0.43 µm in visible light, an axial resolution of R_{ax} = 1 to 15 nm depending on the algorithm used and nature of the surface to be measured and a maximum field of view of 180 µm x 140 µm with a Prosilica CE1380 camera (1360x1024 pixels). The software used (CPM 2.2) for the control, acquisition and processing was developed in-house using LabView. The algorithm used for fringe processing and surface roughness characterization was an improved version of the visibility measurement technique [6] derived from Teager-Kaiser energy operators [19]. Rapid image acquisition was used (sequence of successive images) with axial steps of 90 nm, slight noise reduction (low pass 3x3 Gaussian filter) and envelope peak interpolation using second order spline fitting.

ii) Zygo NewView 7200 microscope

The Zygo NewView 7200 interference microscope used was a system equipped with x50 Mirau interference objective, a 150 µm piezo-electric vertical stage, a digital B&W camera (640x480 pixels) and Zygo proprietary image analysis software. The field of view is 140 x 110 µm and the system has a quoted axial resolution of ~ 0.1 nm with a precision of < 0.75 % over 150 µm. The lateral resolution is R_{lat} = 0.52 µm.

iii) AFM (Park XE70) microscope

The AFM microscope used was a Park XE 70 model working in the non-contact mode. The tip used was a non-contact high frequency point probe. The field size used was 45x45 µm for 512x512 pixels, giving a lateral resolution of 90 nm. This value can be improved by scanning over a smaller area with a higher number of pixels. For example, scanning over 2x2 µm for 1000x1000 pixels gives a lateral resolution of 2 nm.

iv) ESEM environmental microscope

The electron microscope employed was an ESEM environmental system under a pressure of 5 Torr.

3.4. Analytical procedures for roughness analysis

For analyzing and comparing the measurements from the different microscopes, MountainsMap® 6 software (from Digital Surf) was used. This enables false color images of the height data, 3D views, 2D surface line profiles and height and roughness measurements to be made in a uniform way from the different measurement sources. For comparing the line profiles from the same place, a line profile 45 µm in length, corresponding to the width of the AFM measurements, was taken from the middle of the same etched square in the measurements from each of the microscopes. The square was identified using the numbering system.

4. Results of surface roughness measurements

The results of the surface topography measurements made with the different microscopes on the Si sample and HA on Si are now presented.

4.1. Results on patterned silicon wafer

A first comparison of the three sets of 3D measurements on the same area of bare patterned Si wafer (figure 4) shows certain broad similarities in the results.

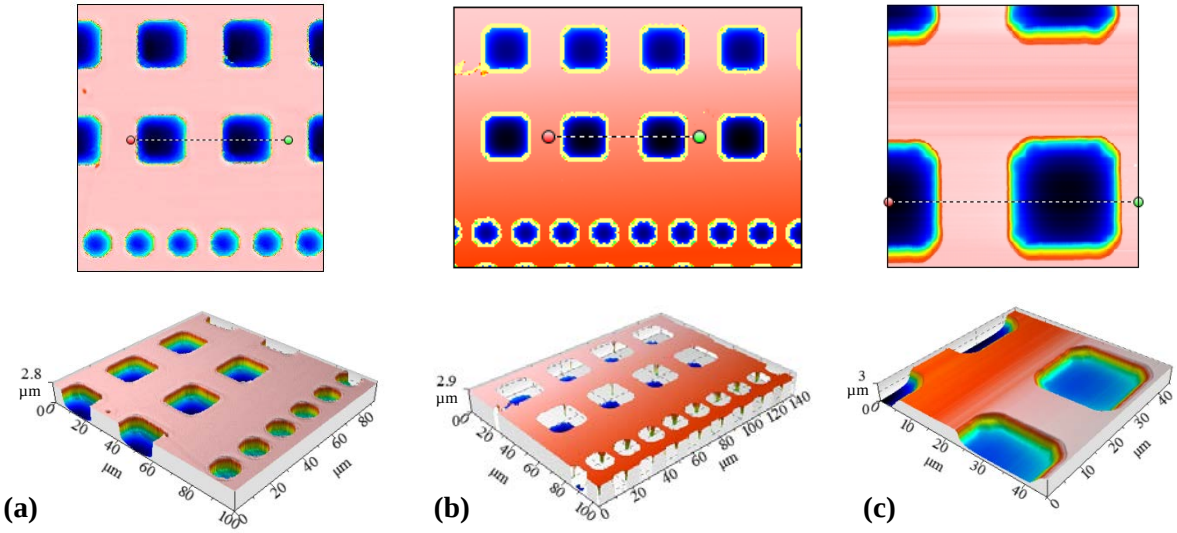


Figure 4. Comparison of measurements of the same etched squares in Si using (a) the Leitz-Linnik microscope, (b) the Zygo NewView 7200 microscope and (c) the Park XE70 AFM microscope.

As would be expected, square depressions of a similar size were found in each case. While the 2D surface profiles (figure 5) confirm the resemblance between the three measurements, on closer study, several differences can be observed. The most significant difference is the variation between the values of the average depths of the etched squares found by each measurement technique (Table 1).

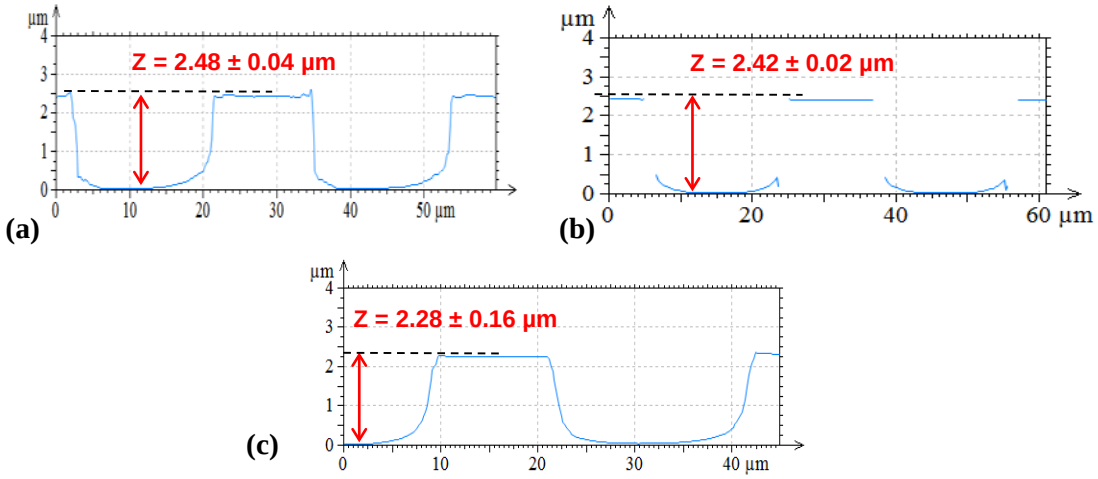


Figure 5. Comparison of 2D profiles of the same etched squares in Si measured using (a) the Leitz-Linnik microscope, (b) the Zygo NewView 7200 microscope and (c) the Park XE70 AFM microscope.

Table 1. Comparison of depth measurements of etched squares (figure 5) using each technique.

Technique	Average measured depth	Estimated depth uncertainty	Estimated percentage uncertainty
Leitz-Linnik	2.48 µm	± 0.04 µm	± 1.6 %
Zygo NewView 7200	2.42 µm	± 0.02 µm	± 0.75 %
Park XE70 AFM	2.28 µm	± 0.16 µm	± 7 %

The estimations of the uncertainties of measurements for each technique were made in the following way. For the Leitz-Linnik measurements, the main contributions to the uncertainty come from errors in the piezo step positioning, the envelope determination algorithm and the reference mirror flatness, resulting in an overall uncertainty of ± 0.04 µm (± 1.6 %). Concerning the Zygo NewView 7200, the uncertainty of ± 0.02 µm is calculated from the quoted accuracy of ± 0.75 % of the depth measurement.

For the AFM measurement the main contributions to the measurement uncertainty come from the non-linearity at depths greater than a few hundred nm together with noise from acoustic vibrations, leading to an estimated uncertainty of ± 0.16 µm (± 7 %). The uncertainty of the AFM measurements could be improved by optimizing the choice of the control parameters for the measurement. Since these measurements were made, the AFM microscope has been placed in an acoustic enclosure, which also improves the uncertainty values.

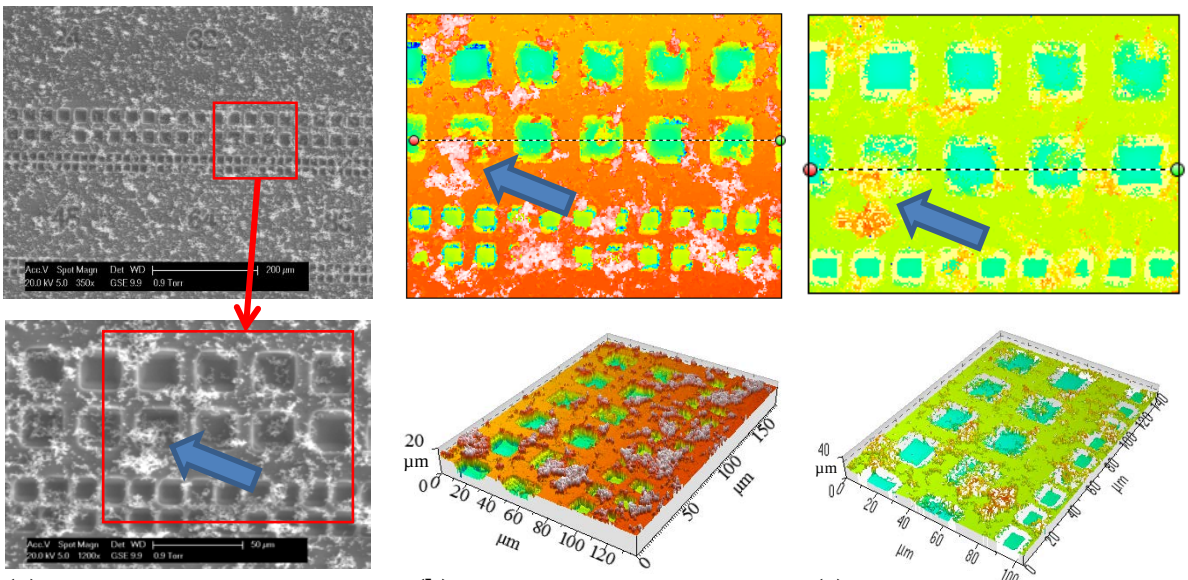
Another significant difference between the 2D profiles can be observed near to the measurements of the edges in all three cases. For both of the interferometric measurements, edge effects are visible. For the Leitz-Linnik this error is in the form of an over estimation of the top of the edge (figure 5(a)) and for the Zygo it is in the form of an under estimation at the bottom of the edge (figure 5(b)). These errors appear to be similar to the well-known "batwing" artefacts when measuring a step height near to the coherence length of the light used due to mixing of signals coming from the top and bottom of the edge [20]. In addition, for the Zygo results (figure 5(b)), the missing measurement points between the top and

bottom of the edges are due to the lack of fringe signals on the steep slopes because of the limited numerical aperture of the objective [10]. Finally, for the AFM measurements, the edges can be seen to be rounded off, which is a result of the convolution between the tip shape and the edge of the etched square. The degree of rounding off was also found to vary for different tip scanning speeds and sample orientations.

4.2. Results on the HA layer on patterned silicon wafer

For these particular samples of HA layers on etched silicon patterns, measurements were not possible using AFM because of the high layer thickness and depth of the etched squares (6.6 µm) and the limited dynamic range of 7 µm of the instrument. It is well known that HA is very difficult to characterize with AFM [11].

A first comparison of the different 3D measurements on the same region (figure 6) again shows certain broad similarities in the results, indicating a partially rough layer on square depressions. The presence of individually recognizable large clumps of HA, such as the one indicated by the large arrow in figure 6, confirms that exactly the same area had indeed been measured by each technique and the success of the numbered multi-scale square pattern used.



(a) **(b)** **(c)**

Figure 6. Comparison of the surface shape of the same place of hydroxyapatite layer deposited on etched squares in Si measured using (a) SEM, (b) the Leitz-Linnik microscope and (c) the Zygo NewView 7200 microscope. Blue arrows indicate same clump of HA.

A comparison of the 2D line profiles made at the same place (figure 7) between the measurements on the Leitz-Linnik and Zygo NewView 7200 also shows broad similarities as well as the presence of differences and artefacts. An exact comparison of the height measurements of the HA layer between the two systems is more difficult than just with the bare etched silicon squares due to the greater variation in surface height with axial position of the rough HA layer.

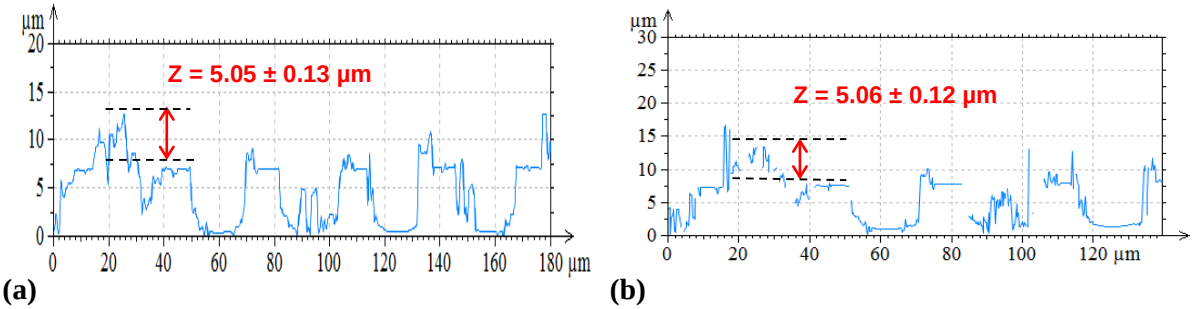


Figure 7. Comparison of 2D profiles of hydroxyapatite layer deposited on etched squares in Si measured using (a) the Leitz-Linnik microscope and (b) the Zygo NewView 7200 microscope.

Nonetheless, by careful positioning of the cursor lines in the two measurements, the two profiles obtained (figure 7) can be seen to be fairly similar. A comparison of the heights at the places indicated is shown in Table 2. The uncertainties in measurement were calculated as in section 4.1.

Table 2. Comparison of depth measurements of etched squares (figure 7) using each technique.

Technique	Average measured depth	Estimated depth uncertainty	Estimated percentage uncertainty
Leitz-Linnik	5.05 µm	± 0.20 µm	± 4 %
Zygo NewView 7200	5.06 µm	± 0.20 µm	± 4 %

The higher values of uncertainty in measurement on the HA layer compared with the results on the bare silicon are due to the inaccuracy from the placing of the measurement cursor in the image and the large lateral variation in roughness of the HA.

5. Conclusions

The aim of this work was to develop a technique of sample marking that enabled the topographic surface measurement of the same place on a given sample using different microscopes. Such a method allows the comparison of measurements at the same place so as to be able to reveal the specific artefacts and performance limitations of each microscopy method so as to be able to improve their measurement precision. After studying the laser marking of cement samples, the use of evaporated Al marks on silicon and photolithographic etching of a numbered, multi-scale square pattern on silicon, the latter technique was chosen for the purposes of the study. The etch pattern allowed the successful identification of the same zone of analysis under different microscopes for surface measurement. The microscopes used were interference microscopy (of different types), AFM and ESEM.

Two silicon samples were prepared with the etched patterns and one was covered with a partial layer of HA. Measurement of the same zone of 20 µm and 10 µm sided squares and comparison of the the 3D measurements and 2D line profiles showed a broad similarity in the results and confirmation that the same zone had been identified under each microscope. A more detailed study showed variations in the measurement of step heights between the different techniques, of up to 1.6 % between the interference techniques on the bare silicon sample, mainly due to errors from the piezo positioning, the envelope algorithm and the reference mirror flatness. This uncertainty increased to 7 % for the AFM measurements, due to the non-linearity over the depth range measured and noise from acoustic vibrations.

The difference between the results from the two interference techniques increased to 4 % for measurements on the rough HA layer, mainly due to the additional errors from the lateral positioning of the measurement cursor on a rough surface. Edge effects are also clearly visible due to probe/material interactions in both interferometry (optical effects) and AFM (convolution with the probe tip shape).

The overall conclusions of this work are that while topographic surface measurements using different techniques of microscopy give broadly similar results, variations exist between the exact details and roughness values. Differences can be observed between the absolute depth measurement values and between the details on step edges and rough surfaces. The use of different techniques on exactly the same zone in areal measurement is a useful solution for revealing the performance, artefacts and limits of each technique in real life applications. We have thus shown that great care needs to be taken in choosing the right technique for measuring different types of samples as well as in the correct setup of the specific measuring parameters and calibration of the z measurements. Moreover, this work is a starting point in the very broad research topic, requiring further work to reveal the different parameters that must be mastered and improved at the level of characterization, and to find the most appropriate choice of marking technique for a given material to lead to accurate areal roughness measurements.

Acknowledgements
Thanks are given to C. Heitz for the growth of the hydroxyapatite layers and to C. Munzer for providing the cement paste sample.

References

[1] Leach R K 2010 *Fundamental principles of engineering nanometrology* (Elsevier, Amsterdam)
[2] Leach R K 2011 *Optical measurement of surface topography* (Springer, Berlin)
[3] ISO/CD 25178-600 2013 Geometrical product specifications (GPS) – surface texture: areal – part 600. Nominal characteristics of areal surface topography measuring instruments (Geneva: International Organization for Standardization)
[4] Leach R K Giuscaa C L and Coupland J M 2012 *Proc. SPIE* **8430** 84300H 1
[5] Ottevaere H, Schwider J, Kacperski J, Steinbock L, Weible K, Kujawinska M and Thienpont H 2008 *Proc. SPIE* **6995** 69950J.1-69950J.9
[6] Montgomery P C, Salzenstein F, Montaner D, Serio B and Pfeiffer P 2013 *Proc. SPIE* **8788** id. 87883G 11
[7] Benatmane A Montgomery P C Fogarassy E and Zahorsky D 2003 *Applied Surface Science* **208-209** 189
[8] Schmit J Reed J Novak E and Gimzewski J K J 2008 *Opt. A: Pure Appl. Opt.* **10** 064001 1
[9] Montgomery P C and Gall-Borrut P 1996 *Institute of Physics Conference Series* **149** 147
[10] Coupland J M and Lobera J 2010 *Strain* **46** 69
[11] Pecheva E Montgomery P Montaner D Pramatarova L 2007 *Langmuir* **23** 3912
[12] Deng J W Dua K Wua B Sui M L 2013 *Intermetallics* **34** 75
[13] Galeano J A Sandoz P Gaiffe E Launay S Robert L Jacquot M Hirchaud F Pretet J L and Mougin C 2011 *Biomedical Optics Express* **2** 1307
[14] Abdurrochman A Lecler S Fontaine J Mermet F Meyrueis P Tumbelaka, B Y and Montgomery P C 2014 *Proc. SPIE* **9135** 913523 1
[15] Tsukamoto M Kayahara T Nakano H Hashida M Katto M Fujita M Tanaka M and Abe N 2007 *J. Phys.: Conf. Ser.* **59** 666
[16] Guellil M Montgomery P C Pfeiffer P & Serio B 2014 *Proc. SPIE* **9132** 913204 1
[17] Pecheva E Pramatarova L Maitz M F Pham M T and Kondyuirin A 2004 *Appl. Surf. Sci.* **235** 176
[18] Kokubo T Kushitani H Sakka S Kitsugi T and Yamamuro T 1990 *J. Biomed. Mater. Res.* **24** 721
[19] Salzenstein F Montgomery P C Montaner D and Boudraa A O 2005 *Eurasip J. Appl. Signal Process.* **17** 2804
[20] Harasaki A and Wyant J C 2000 *Appl. Opt.* **39** 2101