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Comparison of areal measurements of the same zone of etched Si and hydroxyapatite layers on etched Si using different profiling techniques

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Keywords: Surface roughness, optical profilometry, Coherence Scanning Interferometry (CSI), AFM, sample marking, silicon, hydroxyapatite, photolithography

ABSTRACT

In the field of areal surface roughness measurement, characterization using several techniques can be helpful to better understand the performance of each technique and to improve the overall precision. Measuring exactly the same area with different techniques in practice is not easy. Such studies are of great interest in order to characterize and to understand important new materials today such as semiconductor alloys and graphene for silicon technologies, or biomaterials such as hydroxyapatite for use in human implants.

In this work, two types of samples based on a silicon wafer were made by marking with a fractal, multi-scale photolithographic mask and etching. The first sample consisted of a bare silicon wafer with a pattern consisting of 2.4 µm deep numbered square features. The second sample was a rough layer of hydroxyapatite deposited from a solution of simulated body fluid on a similarly etched silicon wafer. The same zone of several squares on the two samples were measured by interference microscopy, AFM and ESEM.

The 2D cross sectional profiles and 3D views from the different results were then compared using different analytical measurement software tools. While the general shapes of the measured microstructures were similar, several differences also appeared. Variations were found of up to 7 % in the depths of the etched features measured with the different techniques. This is ascribed to instrumentation calibration errors, probe/surface interactions and to differences in measurement procedures between the software used. Artifacts were also visible at square edges due to probe/source interactions.

1. INTRODUCTION

In the areas of micro and nano surface roughness and shape measurement and the development of new optical measurement techniques, characterization using several techniques can be helpful in benchmarking, to better understand the performance of each technique and to improve the overall measurement precision[1]. This is all the more true when the surface structure deviates from a homogeneous and flat surface to one that is heterogeneous and rough[2]. Coherence scanning interferometry (CSI) or white light scanning inerferomety (WLSI) as it is also known, is an important 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[3]. Being an optical technique, CSI also has its limits, making it necessary to compare measurements with those from other types of techniques to help in the understanding of the different sources of errors. For example, it is well known that CSI can produce artifacts in the measurement of rough layers due to the steep surfaces involved[4].

In the results of materials analysis presented in the literature, in which several techniques are used on the same sample, comparative measurements are more often made in different places[2, 5]. Measuring exactly the same area with different techniques in practice is not easy. Such studies are of great interest in order to characterize and to understand

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important new materials today such as semiconductor alloys and graphene for silicon technologies, or biomaterials such as hydroxyapatite for use in human implants[5].

One way of identifying exactly the same place of a sample under different microscopes is to mark the sample. Many different types of techniques exist for marking, such as by using ink[6], mechanical etching, pulsed laser ablation[7], laser marking with a photonic jet[6], thin film deposition, photolithographic etching and position indexing[8]. Several factors influence the choice of marking technique, such as the material properties (physical, chemical, roughness…), the marking resolution required and attainable on a given sample and the field of view required in order to be analysed by the different characterization methods used.

In this work it was decided to begin with roughness measurements on two types of samples. The first consisted of simple etched structures in a smooth silicon wafer and the second, a rougher surface consisting of a layer of hydroxyapatite (biomaterial) deposited on the same type of etched silicon wafer. A photolithographic mask was developed to produce the etched structures so that it was possible to identify and measure exactly the same area. The mask was designed with a fractal form of squares, with different geometries varying in size for rapid and efficient localization of sample features varying from mm^2 to μm^2. The first sample consisted of the bare silicon wafer with an etched pattern consisting of different sized numbered square features with a depth of about 2.4 µm. The second sample was a rough layer of hydroxyapatite deposited from a solution of simulated body fluid (SBF) on a similarly etched silicon wafer. The same zone of several squares on the two samples were analyzed by interference microscopy (a Leitz-Linnik research microscope and a Zygo NewView 7200 commercial microscope), AFM (Park XE70) and ESEM (environmental SEM).

The 2D cross sectional profiles and 3D views from the different measurements were then compared using different analytical measurement tools (MountainsMap, a LabView based laboratory-developed program and Zygo and Park proprietary software). The results of the comparisons made between the different measurements 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 artifacts. 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.

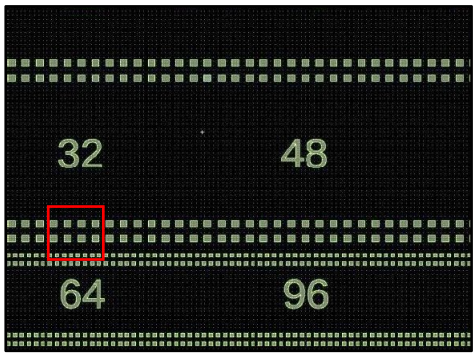
2. EXPERIMENTAL

In this section the photolithographic marking technique used to etch the silicon wafers are described, together with the method used for growing the hydroxyapatite layers. The different topographic measurement techniques used are also explained, as well as the analytical procedures employed to compare the different surface shape measurements.

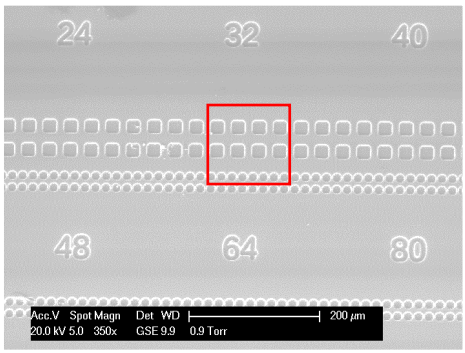
2.1 **Fractal test pattern developed**

The mask technique that seemed the most appropriate for the silicon wafers was that using a UV photolithography and RIE etching. Because the etching depth is not completely uniform over the whole of a wafer diameter using RIE etching, tending to be greater near the center, the geometrical characteristics of a test pattern should 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 under different microscopes. For rapid and efficient localization, different geometries with varying sizes have been developed, one of which is based on a fractal geometry using numbered square patterns of different sizes. The design of the photomask was carried out using scripts available with microelectronic CAO software (LayoutEditor, Juspertor), the portion of interest being shown in Figure 1(a).

The test pattern was etched in the silicon wafer using standard photolithographic procedures, using a positive photoresist, direct contact in a mask aligner and an RIE etching time of t = 2 min 15 s. The resulting etched mask pattern on a silicon wafer with the zone of interest is shown in Figure 1(b) using ESEM.



(a) CAO image of the part of the mask used



(b) Result of marked silicon wafer using SEM

Figure 1. Details of the photolithographic test pattern developed to mark the silicon wafer and area of interest identified for the measurements (red square)

2.2 Growth technique used for hydroxyapatite layers

Hydroxyapatite (HA) is an inorganic material ($Ca_{10}(PO_4)_6(OH)_2$) that is chemically similar to the mineral component of bones, teeth and hard tissues in mammals. The study of synthetically grown layers of HA and composite materials with nanodiamonds, polymers, etc. is important today in the fields of human implants and new biological scaffolds[9]. The measurement of the topology of these rough layers is a key part of their characterization[5]. So as to be able to measure the same zone with the different techniques, the same type of silicon wafer etched with the fractal pattern used previously was used as a substrate and a partial layer of HA was grown, with enough HA to be measured while leaving spaces to be able to recognize the pattern.

(a) The silicon wafer samples immersed in the SBF

(b) The beaker, samples and SBF during heating and stirring

Figure 2. The HA layer growth technique on the silicon samples in SBF

The HA layers were 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[10]. 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 (Figure 2(b)).

2.3 Measurement techniques used

The different microscope techniques used to characterize the samples are now described.

i) Leitz-Linnik microscope

The Leitz-Linnik microscope is an interference microscopy system developed at ICube, based on white light

scanning interferometry and described in [5]. The Leitz-Linnik microscope is 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[2] using rapid image acquisition (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. The visibility measurement technique is derived from Teager-Kaiser energy operators[11, 12].

ii) Zygo NewView 7200 commercial microscope

The Zygo NewView 7200 interference microscope used was a commercial 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 maximum field size is 50x50 µm for 256x256 pixels, with a lateral resolution of R_{lat} = 0.012 µm to 0.195 µm depending on the field size.

iv) ESEM environmental microscope

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

2.4 **Analytical procedures for roughness analysis**

To analyze and compare the measurements from the different microscopes, two types of software were used, Mountains Maps v6 (from Digital Surf) and our own in-house software CPM 2.2. The Mountains Map software was used to produce the false color images of the height data and the 3D views of the results from the Leitz-Linnik, Zygo and AFM microscopes. It was also used to produce the line profiles of the results from the Zygo and AFM microscopes. Due to problems of importing the full depth 16 bit images from the CPM 2.2 LabView program into Mountains Map, CPM 2.2 was used directly to produce the line profiles from the Leitz-Linnik measurements.

In order to be able to compare 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 the each microscope (Figures 3 and 4). The square was identified using the numbering system.

3. SURFACE ROUGHNESS MEASUREMENT RESULTS

The results of the surface topography measurements made with the different microscopes are now given.

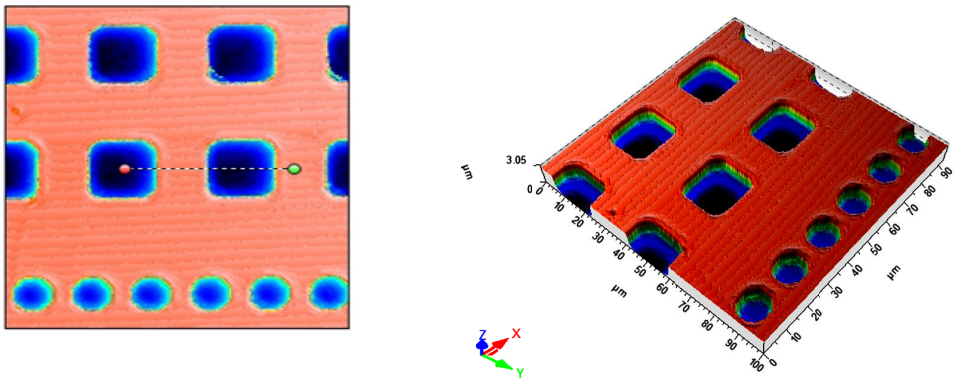
3.1 **Results on patterned silicon wafer**

A first comparison of the different measurements using the false color height images and 3D views (Figure 3) shows certain broad similarities in the results found of the same region of the sample surface. This is confirmed by an initial observation of the 2D profiles (Figure 4). But on closer study of these 2D profiles, differences between the measurements begin to appear. For example, a comparison of the average depth of the etched squares found by each technique shows significant variations (Table 1).

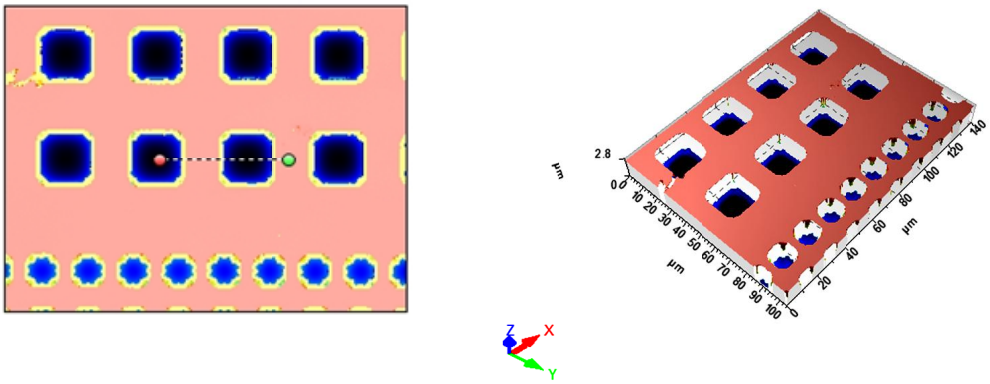
Table 1. Comparison of depth measurements from Figure 4 using each technique

Technique	Average measured depth	Estimated depth error	Estimated percentage error
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 %

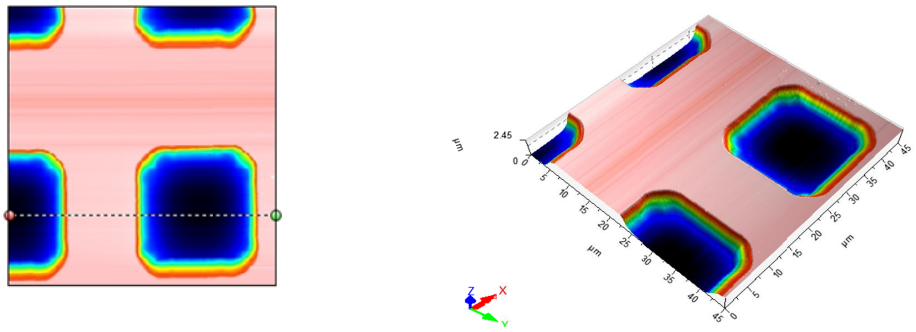
The error estimations for each technique were made in the following way. The error of ± 0.04 µm for the Leitz-Linnik measurement is mainly due to a combination of errors in the piezo step positioning, the envelope determination algorithm and the reference mirror flatness, resulting in an overall error of ± 1.6 %. For the Zygo NewView 7200, the error of ± 0.02 µm is based on the quoted precision of ± 0.75 % of the depth measurement. In the case of the AFM measurement error of ± 0.16 µm (± 7 %), the main source of error is the non-linearity at high depths (> 1 µm). The precision of the AFM measurements could also be improve by placing the instrument in a sound proof enclosure, as well as by optimizing the choice of the control parameters for the measurement.



(a) Leitz-Linnik microscope



(b) Zygo NewView 7200 microscope



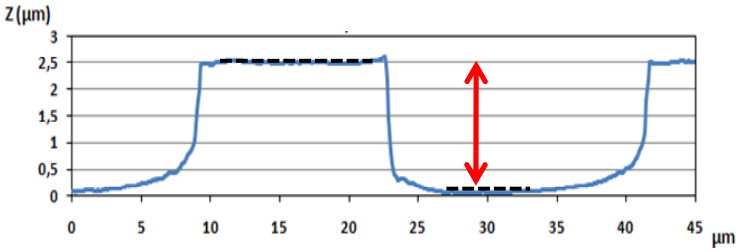
(c) AFM Park XE70 microscope

Figure 3. Comparison of surface shape measurements of etched squares in Si measured at the same place by different microscopes (left: false color height image; right: 3D view)

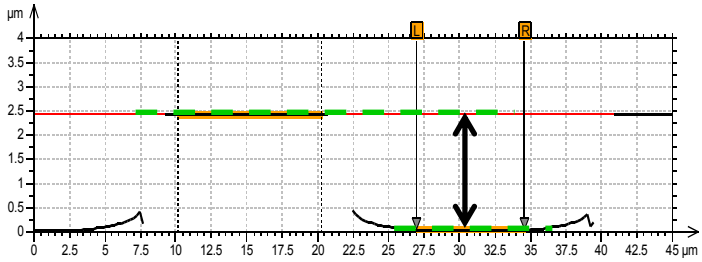
Differences between the 2D profiles can also be observed near to the measurements of the edges in all three cases. For both interferometric measurements there are edge effects that are visible, either in the form of an over estimation of

the top of the edge (in the case of the Leitz-Linnik, Figure 4(a)) or an under estimation at the bottom of the edge (in the case of the Zygo, Figure 4(b)). These are similar to the well known "batwing" artifacts when a step height is near to the coherence length of the light used due to mixing of signals coming from the top and bottom of the edge[13]. In the case of the Zygo (Figure 4(b)), measurement points are missing between the top and bottom of the edges due to the lack of fringe signals on the steep slopes due to the limited numerical aperture of the objective[4].

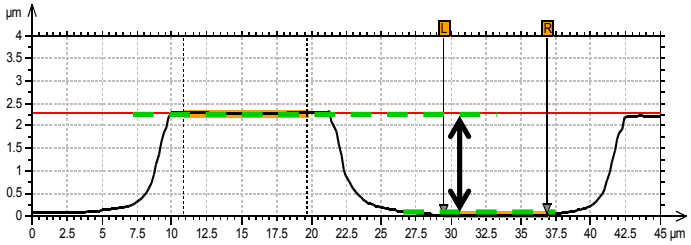
In the AFM measurements, a rounding off of the edges is apparent, which is a result of the convolution between the tip shape and the edge of the etched square. The rounding off was also found to vary for different tip scanning speeds and sample orientations.



(a) Leitz-Linnik microscope: Z = 2.48 ± 0.04 µm



(b) Zygo NewView 7200 microscope: Z = 2.42 ± 0.02 µm



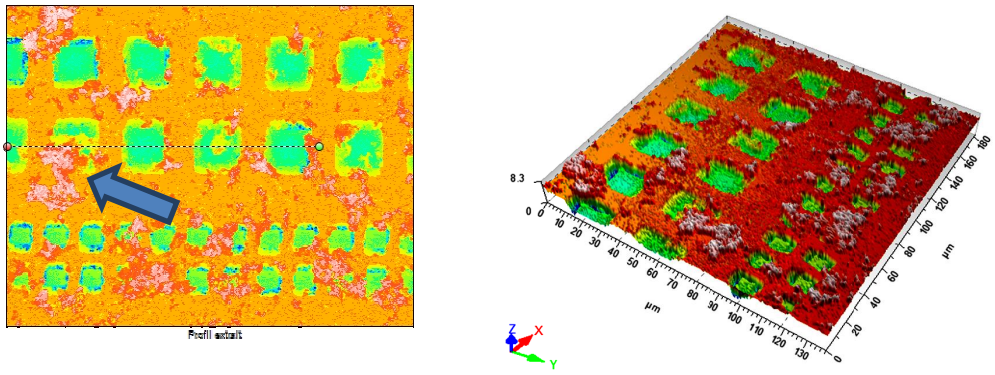
(c) AFM Park XE70 microscope: Z = 2.28 ± 0.16 µm

Figure 4. Comparison of 2D sectional profiles of etched squares in Si measured at the same place by different microscopes

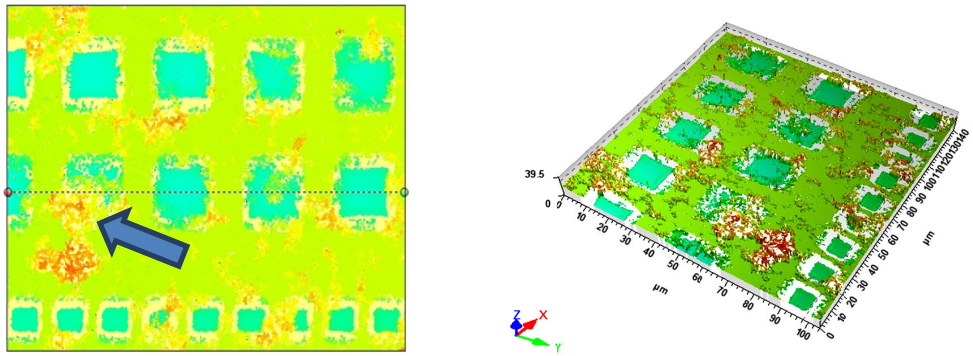
3.2 **Results on hydroxyapatite layer on patterned silicon wafer**

Measurements of the HA layers on etched silicon patterns were not possible with AFM because of the high layer thickness which was greater than the dynamic range of 7 µm of the system, and fragility of the material. It is well known that HA is very difficult to characterize with AFM[5].

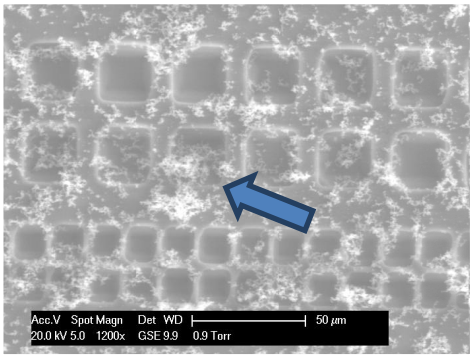
A first comparison of the different measurements using the false color height images and 3D views (Figure 5) again shows certain broad similarities in the results found of the same region of the sample surface. The nature of the clumps of HA, such as the shape of the large clump indicated by the arrow in Figure 5, shows that exactly the same area had indeed been measured in each case.



(a) Leitz-Linnik microscope



(b) Zygo NewView 7200 microscope



(c) ESEM environmental microscope

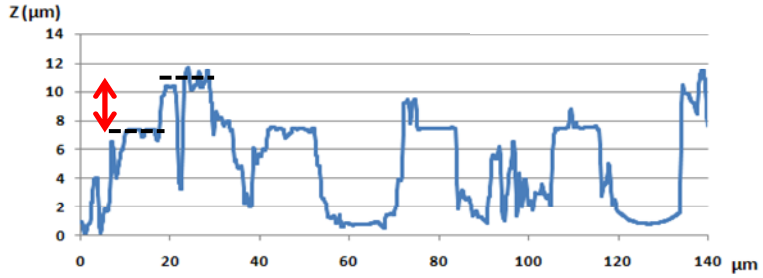
Figure 5. (a),(b) Comparison of surface shape measurements of hydroxyapatite layer deposited on etched squares in Si measured at the same place by different microscopes (left: false color image; right: 3D view) , (c) ESEM image

A comparison of the 2D line profiles (Figure 6) between the measurements on the Leitz-Linnik and Zygo NewView 7200 also shows broad similarities as well as the presence of differences and artifacts. An exact comparison of height measurements 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 caused by the high roughness of HA. Nonetheless, by careful positioning of the cursor lines in the two measurements, the two profiles obtained (Figure 6) were fairly similar. A comparison of the heights at the places indicated is shown in Table 2.

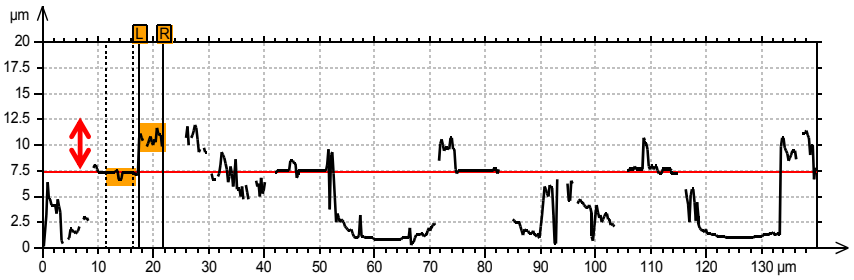
The higher errors compared with the results on the bare silicon are due to the larger contribution in the uncertainty of the measurement with the placing of the measurement cursor in the image due to the high roughness of the HA.

Table 2. Comparison of depth measurements from Figure 6 using each technique

Technique	Average measured depth	Estimated depth error	Estimated percentage error
Leitz-Linnik	3.27 µm	± 0.13 µm	± 4 %
Zygo NewView 7200	3.31 µm	± 0.12 µm	± 3.6 %



(a) Leitz-Linnik microscope: Z = 3.27 ± 0.13 µm



(b) Zygo NewView 7200 microscope: Z = 3.31 ± 0.12 µm

Figure 6. Comparison of 2D sectional profiles of hydroxyapatite layer deposited on etched squares in Si measured at the same place by different microscopes

4. DISCUSSION AND CONCLUSIONS

The aim of this work was to develop a technique that enabled the topographic surface measurement of the same reference point on a given sample using different characterization methods. Such a technique would help to better understand the measurement artifacts and limits of each microscopy method so as to be able to improve the measurement precision of each.

The method chosen was the marking of a silicon sample with an etched fractal pattern of numbered squares using a photolithographic mask. This allowed the successful identification of the same zone of analysis under different microscopes and subsequent measurement. The microscopes used were interference microscopy (research and commercial systems), AFM and ESEM.

Two silicon samples were prepared in this way and one was covered with a partial layer of hydroxyapatite. Measurement of the same zone and comparison of the false color height images, the 3D views and 2D line profiles showed a broad similarity in the results and proof that the same zone had been identified under each microscope. A study in further detail 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, due to uncertainties in the piezo positioning, the envelope algorithm and the reference mirror flatness. This increased to 7 % for the AFM measurements, mainly because of non-linearity at high depth measurement (> 1 µm). The difference between the two interference techniques increased to 4 % for results on the rough HA layer, mainly due to the additional uncertainty 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 conclusion of this work is that there can be significant variations in topographic measurements between different areal techniques both in terms of absolute depth measurement values and details on step edges and rough surfaces. 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 calibration of the z measurements. Using different techniques on the same zone in areal measurement is one solution to cope with the limits of each technique and to help identify the performance, limits and artifacts produced by each method. Moreover, this work is a starting point in this 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 satisfactory areal roughness measurements.

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REFERENCES

[1] Ottevaere, H., Schwider, J., Kacperski, J., Steinbock, L., Weible, K., Kujawinska, M. and Thienpont, H., "Benchmarking instrumentation tools for the characterization of microlenses within the EC Network of Excellence on Micro-Optics (NEMO)," Proc. SPIE 6995, pp. 69950J.1-69950J.9 (2008).

[2] Montgomery, P.C., Salzenstein, F., Montaner, D., Serio, B. and Pfeiffer, P., "Implementation of a fringe visibility based algorithm in coherence scanning interferometry for surface roughness measurement," Proc. SPIE 8788, id. 87883G 11 (2013).

[3] Schmit, J., Reed, J., Novak, E. and Gimzewski, J.K., "Performance advances in interferometric optical profilers for imaging and testing," J. Opt. A: Pure Appl. Opt., 10, 064001, 1-7 (2008).

[4] Coupland, J.M. and Lobera, J., "Measurement of steep surfaces using white light interferometry," Strain 46(1), 69-78 (2010).

[5] Pecheva, E., Montgomery, P., Montaner, D., Pramatarova, L., "White Light Scanning Interferometry Adapted for Large Area Optical Analysis of Thick and Rough Hydroxyapatite Layers," Langmuir, 23, 3912-3918 (2007).

[6] Abdurrochman, A., Lecler, S., Fontaine, J., Mermet, F., Meyrueis, P., Tumbelaka, B.Y. and Montgomery, P.C., "The photonic jet to improve the lateral resolution of laser etching," Proc. SPIE 9135, in press (2014).

[7] Tsukamoto, M., Kayahara, T., Nakano, H., Hashida, M., Katto, M., Fujita, M., Tanaka, M. and Abe, N., "Microstructures formation on titanium plate by femtosecond laser ablation," J. Phys.: Conf. Ser., 59, 666 (2007).

[8] Galeano, J.A., Sandoz, P., Gaiffe, E., Launay, S., Robert, L., Jacquot, M., Hirchaud, F., Pretet, J.L. and Mougin, C., "Position-referenced microscopy for live cell culture monitoring," Biomedical Optics Express, 2 (5), 1307 (2011).

[9] Pecheva, E., Pramatarova, L., Maitz, M.F., Pham, M.T. and Kondyuirin, A., "Kinetics of hydroxyapatite deposition on solid substrates modified by sequential dual implantation of Ca and P ions. Part I. FTIR and Raman spectroscopy study," Appl. Surf. Sci., 235, 176-181 (2004).

[10] Kokubo, T., Kushitani, H. Sakka, S., Kitsugi, T. and Yamamuro, T., "Solutions able to reproduce in vivo surface-structure changes in bioactive glass-ceramic A-W," J. Biomed. Mater. Res., 24, 721-734 (1990).

[11] Salzenstein, F., Montgomery, P.C., Montaner, D. and Boudraa, A.O., "Teager Kaiser energy and higher order operators in white light interference microscopy for surface shape measurement," Eurasip J. Appl. Signal Process., 17, 2804-2815 (2005).

[12] Salzenstein, F. and Boudraa, A.O., "Multi-dimensional higher order differential operators derived from the Teager-Kaiser energy tracking function," Signal Processing, 89, 623-640 (2009).

[13] Harasaki, A. and Wyant, J.C., "Fringe modulation skewing effect in white-light vertical scanning interferometry," Appl. Opt., 39(13), 2101-2106 (2000).