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3D SEM for surface topography quantification - a case study on dental surfaces.

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Abstract. 3D analysis of surface topography is becoming a more used tool for industry and research. New ISO standards are being launched to assist in quantifying engineering surfaces. The traditional optical measuring instrumentation used for 3D surface characterization has been optical interferometers and confocal based instrumentation. However, the resolution here is limited in the lateral dimension to the wavelength of visible light to about 500 nm. The great advantage using the SEM for topography measurements is the high flexibility to zoom from low magnifications and locating interesting areas to high magnification of down to nanometer large surface features within seconds. This paper presents surface characterization of dental implant micro topography. 3D topography data was created from SEM images using commercial photogrammetric software. A coherence scanning interferometer was used for reference measurements to compare with the 3D SEM measurements on relocated areas.
As a result of this study, measurements emphasizes that the correlation between the accepted CSI measurements and the new technology represented by photogrammetry based on SEM images for many areal characterization parameters are around or less than 20%. The importance of selecting sampling and parameter sensitivity to varying sampling is high-lighted. Future work includes a broader study of limitations of the photogrammetry technique on certified micro-geometries and more application surfaces at different scales.

1. Introduction and background

In recent years 3D analysis has become a tool extremely practical and widely used for both industry and research. To assist in measuring of the engineering surfaces new ISO standards were established. Traditionally, the surfaces are characterized by optical interferometers and confocal instrumentation but their resolution are limited in the lateral dimension to the wavelength of the visible light (around 500 nm). Thanks to other instrumentation which uses electromagnetic radiation with smaller scale than the visible light like x-ray-, electron-and neutron irradiation, it is possible to explore smaller features [1].

Today the scanning electron microscope (SEM) is utilized not only in the medical and biological field, but has lot of applications in industry. Unfortunately SEM measurements are just 2D images (in x-y scale) and height details (z-scale) are only represented by color or grey scales. The great advantage in using SEM is that measurements in scale from millimeters to nanometers are possible. Besides investigation of features on surfaces is easy procedure with large magnification options in case of

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surface measurement. The intermediate between 3D images and SEM measurement is photogrammetry, the technology that involves extraction of 3D geometry information from 2D pictures. The measurements received by this technique promise high accuracy compare to another technique, but still a lot of investigation remains. There are a number of algorithms for reconstruction of SEM stereo images to obtain 3D topographical data, one of them are described by P. Podsiadlo and G.W. Stachowiak [2]. The 3D reconstruction trough stereo-photogrammetry has some critical factors: quality of scanning images and instrument set-up [3].

The aim of this work was to compare the measurement results obtained by photogrammetric technique implemented in SEM and measurement performed using coherence scanning interferometer on a dental implant micro screw.

2. Materials and methods

2.1 Measuring equipment
  The coherent scanning interferometer
Surface measurements were effectuated using CSI (MicroXam, ADE Phase shift Technologies)[5]. This is an optical measurement instrument efficient to obtain 3D topographies of surfaces down to nanometer scale. 3D interferometric profiling splits a beam of light, reflecting it off both the test and reference surface, then the light recombines and interference patterns are detected by a high resolution camera. Afterwards this information interprets to provide height information on a pixel basis, building a highly resolved 3D representation of the surface. The resolutions for CSI used in experiment were in the x- and y- axis 0,5 µm and 0,1 nm in z-depth. The accuracy for the instrument is better than 0,1 %.
  The Scanning Electron Microscope
The measurements were done using SEM JEOL JSM-6480[6]. The principle of the SEM is to scan the surface of the sample measured by an electron beam in order to explore it by successive lines. A detector picks up the signal generated by the interactions between the beam and the surface. The image obtained by SEM is in 2D. Spatial resolution at 20 kV is 3 nm for secondary electron image, and 4 nm for backscattered electron image.
  Photogrammetric technique – principle and algorithm
3D measurement data was created from 2D SEM pictures using photogrammetry technique software (MeX 5.1, Alicona[7]). This type of software provides 3D images reconstructed from 2 or 3 2D pictures taken from different points of view. Thus the method used is based on the principle of the stereoscopy [2]. SEM images have a very high resolution but nevertheless, they are still in 2D, which is incomparable with 3D topographies obtained using the interferometer. The accuracy of the SEM stereoscopy measurement depends on the magnification and the pixel size.

2.2. Sample – Dental implant
In this case study measurements were performed on an anodically oxidized dental implant screw [4]. The roughness for this sample is really important to evaluate because it determines the quality of its anchorage in the jaw bone. Its surface has many pores which are obtained by anodic oxidation. The dental implant has been acquired with the magnifications 30x, 500x, 2000x and 10000x using the SEM and the magnification 100x using the interferometer. The ultrasonic bath with a cleaning solution was used for cleaning of dental implant sample. To compare the images obtained with the SEM and the interferometer, the same area were allocated.

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[5] ADE Phase Shift MicroXAM Optical interferometric profiler characteristics [online].
http://www.tcd.ie/CMA/misc/MicroXam.pdf (page accessed in February 2013).
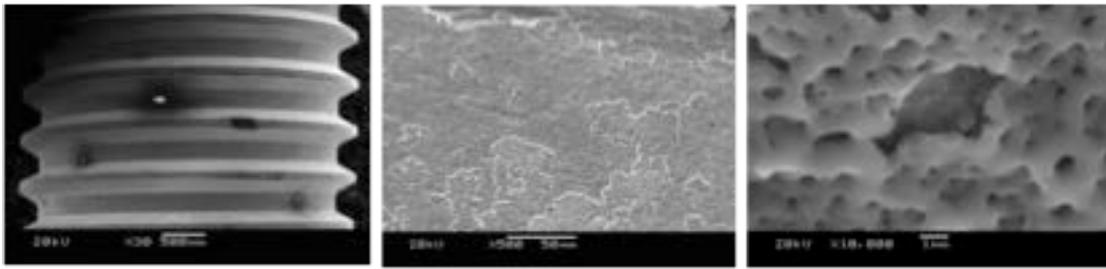[6] www.jeol.com
[7] http://www.alicona.at

Figure 2.1 Dental implant. SEM pictures at 30x, 500x, and 10 000 magnifications.

2.3 Comparison method
3D surfaces based on SEM images were reconstructed with MeX software, and afterwards exported to the analysis software MountainsMap from DigitalSurf[8]. Three images were used for stereoscopic reconstruction. SEM images were obtained by eucentric tilt of the sample, angle of tilt was 5°.The non-measured points of the surfaces obtained using the interferometer were replaced by a value obtained as compared to their neighboring valid points. As a result, filling-in is perfectly invisible and very efficient. The form of the surface has been removed. In this study the polynomial method of third degree was used.
The relocation area for comparison for the SEM and the interferometer is demonstrated in the (figure 2.2).
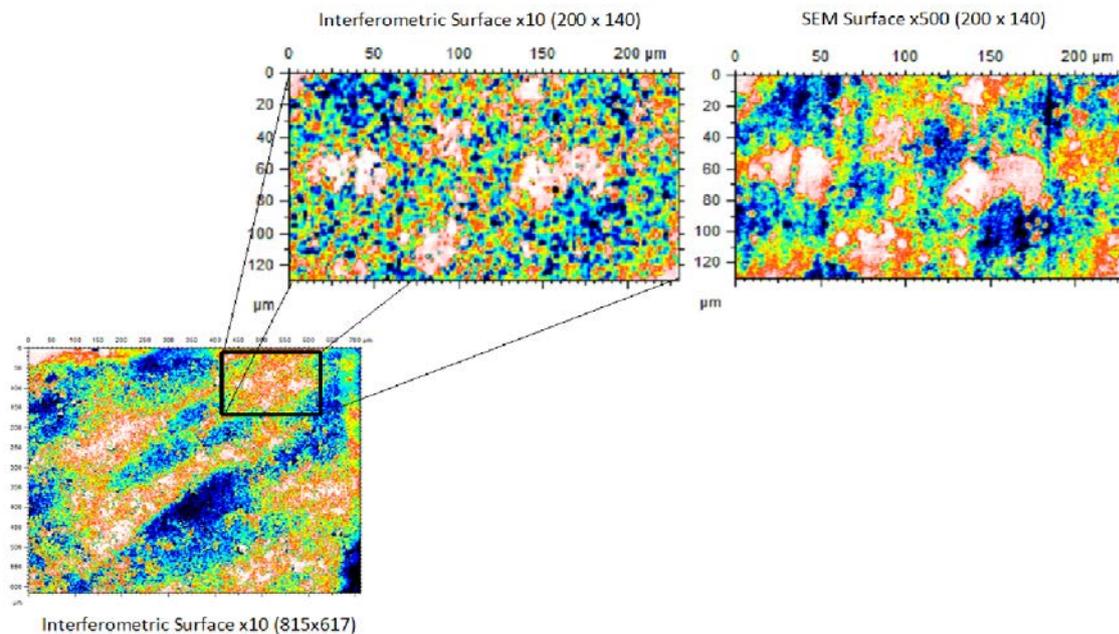

Figure 2.2: Example of comparison using relocated areas for CSI and SEM.

To highlight the differences between the SEM and the interferometer, two comparisons were made. The first compares the parameter S_a of the interferometric surface with that one of the measured surfaces for different wavelengths. To do this, several SL-band pass filters with different wavelengths, -nesting index, were applied to the surfaces. The SEM has a higher resolution than the interferometer and the comparison with the CSI is made with both the SEM surface with the higher resolution and the SEM surface re-sampled to the same resolution as the interferometric surface.

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[8]http://www.mountainsmap.com

The second comparison was a global comparison of all of the parameters of the interferometric surface and SEM surface (re-sampled and not). No filter is applied for this second comparison.

3. Result and discussion

Visual and overall Comparison of CSI and SEM measurements
The following results concern the dental implant based on single relocated measurements applying ISO standard Areal parameters [5], [6].
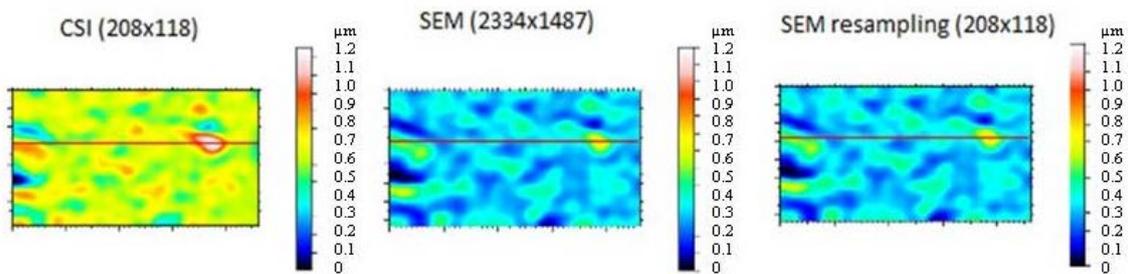

Figure 3.2 Relocated surfaces obtained with the two different measuring instruments. The measurement size is the same for all three measurements (200x140µm) but the sampling numbers differs (208x118, 2334x1487, 208x118). The solid line indicates a profile through the major peak.

The extracted and relocated surfaces show a strong visual correlation in spite the use of two totally different metrology technologies, the one using visible light and the other evaluating the interaction between impacting electrons on the surface.

The amplitude parameters
The second comparison is a comparison of an advanced set of areal roughness parameters obtained from the SEM images, the interferometer and the re-sampled SEM images (fig. 3.3).

The averaging amplitude parameters S_a, S_q, and extreme amplitude parameters Sp (peak height), S_v (valley depth), and Sz (maximum height) give information of the height of the surface profile. The values of the averaging parameters, differs between 18% (S_q) and 21% (S_a) with higher CSI measurement values. Peaks, depths and total height differ all between 5% and 13% and re-sampled SEM measurements seem to underestimate the extreme points (S_q) of the surface with around 5%. However, interferometer and high resolution SEM measurements seems to agree about valley and peak heights on a 5% level indicating a good agreement between the two technologies.

The parameters S_{sk} and S_{ku} (skewness and kurtosis of the amplitude distribution curve) give more information about the distribution of the amplitudes. S_{sk} is the degree of symmetry of the surface heights about the mean plane.

Nominally for the dental surface, S_{sk} is negative (around -1 for all the measurements) indicating the slight predominance of valleys before peaks which describe surfaces with good volume retention.
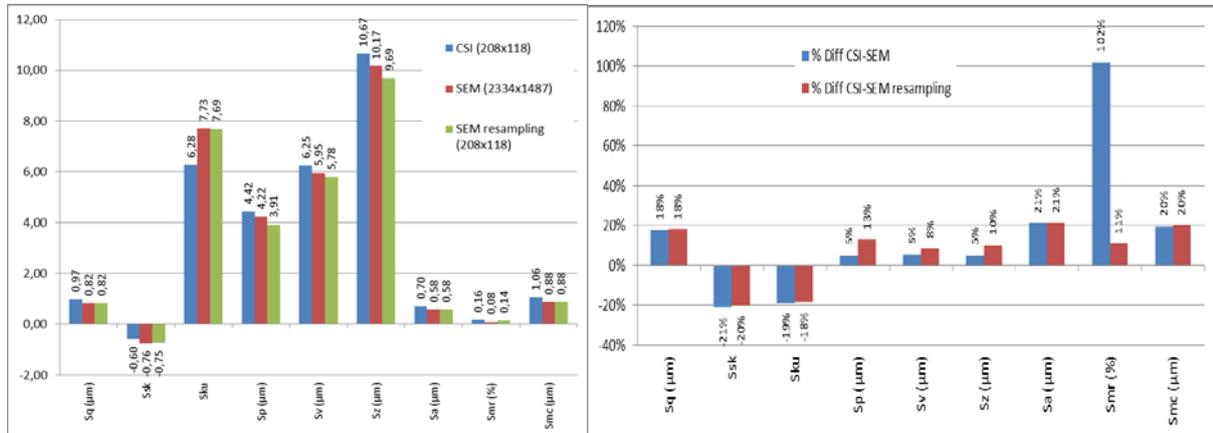

Figure 3.3 Absolute value of differences of the parameters obtained using the SEM, the SEM re-sampling and the interferometer.

The kurtosis, S_{ku} values between 6-8 indicate a more "central" distribution of the amplitudes in comparison the ideal normal distribution of heights. Both S_{sk} and S_{ku} parameters indicate about 20% larger values for CSI measurements (fig. 3.3) hence, stronger negative skewness and higher kurtosis for SEM measurements.

The spatial- and hybride parameters
The spatial parameters (S_{al}, S_{tr}, S_{td}), indicate the wavelength where repeating structures start dominate before random anisotropic structures (S_{al}), degree of anisotropy S_{tr} and S_{td} indicating the texture direction of the global surface profile.
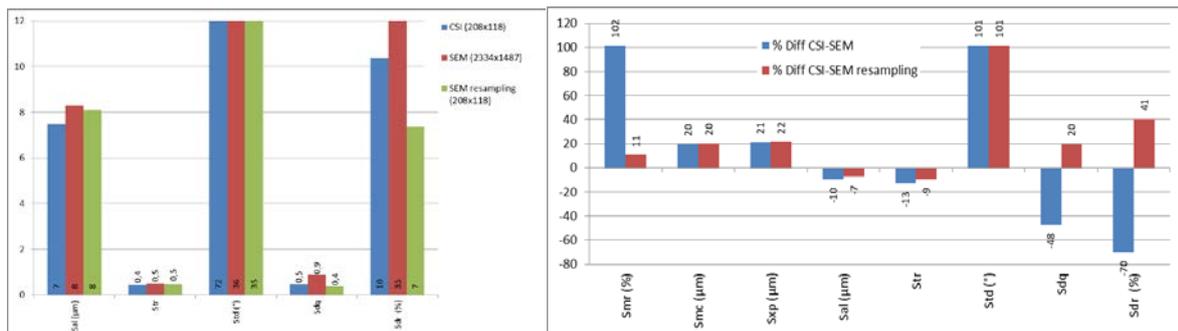

Figure 3.4 Absolute- and relative differences of the parameters obtained using the interferometer (CSI), the SEM, and the SEM re-sampled to the same lateral resolution as the CSI measurements.

S_{al} is around 8um for all measurements indicating that also the interferometer with the lower resolution should be able to pick up dominant repeating features while the sampling distance is well below 8um (fig. 3.4). The S_{tr} is overall close to 0.5 indicating neither clear isotropic nor anisotropic main structures and S_{td} directions are therefore less meaningful for the evaluation. SEM measurements are 7-13% higher for S_{al} and S_{tr} measurements but the 100% difference for S_{td} measurements of texture directions is caused by the ambiguous anisotropy as indicated by the S_{tr} at around 0.5 as discussed above.

S_{dq} and S_{dr} are the hybrids parameters characterizing the rms slope and the developed area ratios where the S_{dr} is expressed as the percentage of additional surface area contributed by the texture as compared to an ideal plane the size of the measurement region. S_{dr} is affected both by texture amplitude and spacing. The SEM resolution (2334x1487 measurement ponts) is much higher than the SEM re-sampled and the CSI interferometer (both 208x118 measurement points), and the increased parameter values for SEM measurements indicate that the dental surface has a structure below the resolution if the CSI.

The volume parameters and named feature parameters
The volume parameters V_m, V_v, V_{mp}, V_{mc}, V_{vp}, and V_{vc} (see fig. 3.5 below) show a minor difference when re-sampling the SEM measurements. The interferometer measurements are between 7% to 28% higher. This increase for interferometer measured parameters could be a result and correlates well to the increased CSI amplitude values observed in fig. 3.3 above.
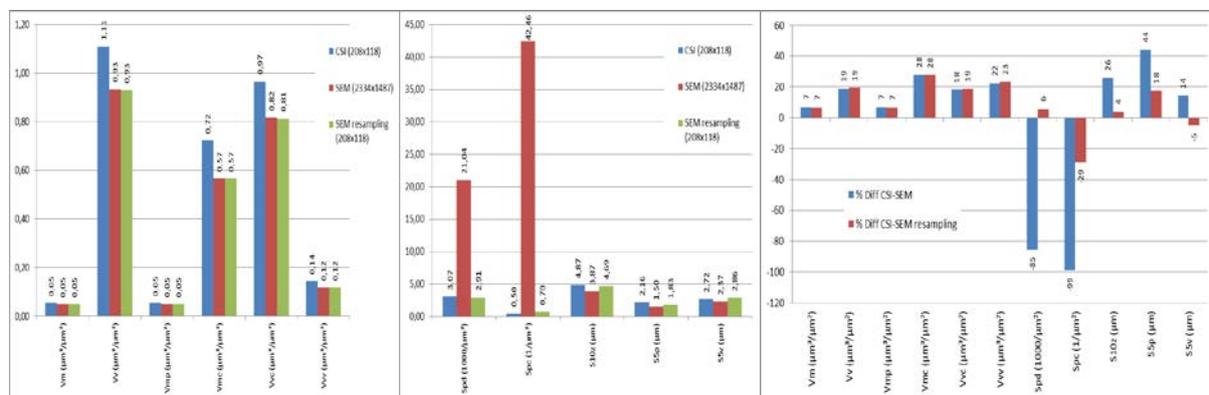

Figure 3.5 Absolute- and relative differences of the volume parameters and named feature parameters obtained using the interferometer (CSI), the SEM, and the SEM re-sampled to the same lateral resolution as the CSI measurements.

The named feature parameters denoting the average 10point height difference, S_{10z}, between the five highest peaks, S_{5p}, and the five deepest valleys, S_{5v}, show both a 20% difference between original measured- and re-sampled SEM measurements due to the sampling sensitivity of the parameters (fig. 3.5). Again, absolute interferometer measurements of the peak- and valley height parameters follow the results from the amplitude height parameters above with higher peak- and valley heights here. 14%-44% smaller heights are measured with the high resolution SEM and as a result of the sampling distance sensitivity of the parameters peaks- and valleys are being redefined resulting in re-sampled SEM measurements are more similar to interferometer measurements (-5%-17% higher CSI parameter values).

The peak density, S_{pd}, and peak curvature, S_{pc}, indicate high differences (85%-98%) between low resolution interferometer and high resolution SEM measurements while a re-sampling of the SEM measurements results in a decreased difference (-7% to 28%) between the CSI- and SEM measurements, again highlighting the sensitivity of lateral sampling distance (fig. 3.5).

Further named feature parameters
Intresting parameters are the named feature parameters dale area, S_{da}, hill area, S_{ha}, dale volume, S_{dv}, and hill volume, $S_{hv,}$ see fig. 3.6 below.
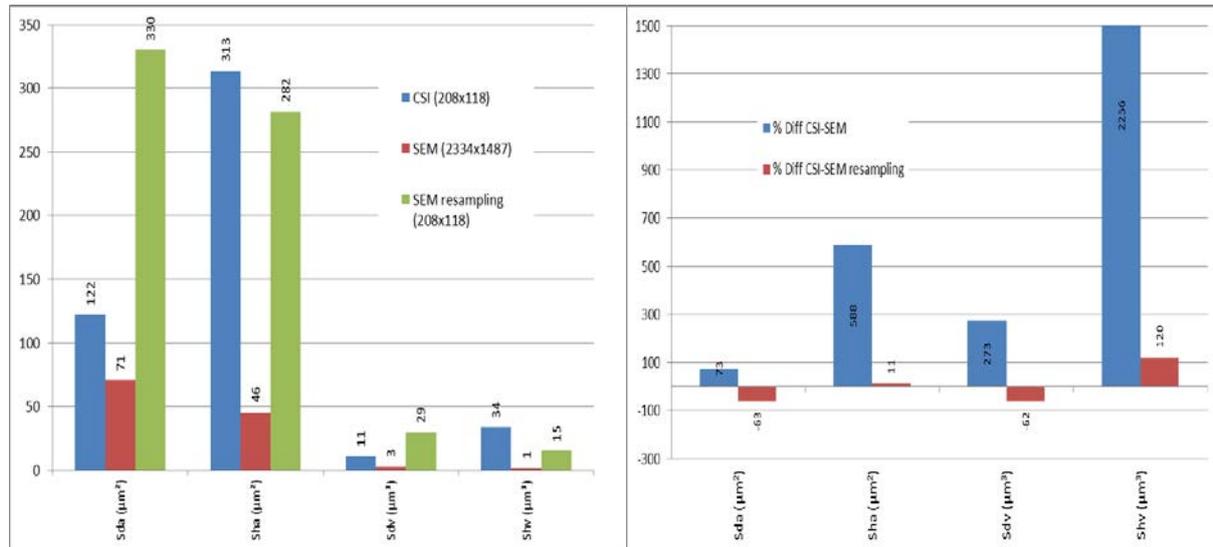

Figure 3.6 Absolute- and relative differences of the feature parameters obtained using the interferometer (CSI), the SEM, and the SEM re-sampled to the same lateral resolution as the CSI measurements.

In absolute values the difference between the instrument types are large. Dale- and hill areas and volumes are following the same trends in absolute differences. The hill areas, dale volumes and hill volumes show a significant sampling distance dependence shown by the 273% to more than 2000% smaller SEM measured parameter values for high resolution measurements. This is also clear through the high difference between parameter values calculated with high resolution SEM compared to the re-sampled values.

4. Conclusions
Several conclusions can be drawn from the dental implant measurement using coherence scanning interferometry (CSI) and 3D SEM technology.

- For measurements using the same sampling distance SEM and Interferometer measurements normally for a majority of characterization parameters not exceed 20% difference.
- SEM average amplitudes parameters S_a, S_q are around 20% smaller than CSI ones.
- Several parameters, S_{mr}, S_{dr}, S_{dq}, S_{pd}, S_{pc} are highly sampling dependent and can vary 56%-100% between the high resolution- and re-sampled SEM measurements.
- Dale- and hill parameters are heavily sampling dependent and for the dental implant huge variations (up to 900%) are found indicating the sensitivity of this set of 3D characterization parameters.
- Dale- and hill parameters show significant differences between SEM- and CSI measurements for both high- and low resolution measurements.

5. **Future works**

Further comparisons using known certified geometrical features will be made to verify and further explore the results from this study.

More studies at different scales and for other applications will further help the introduction of the new 3D SEM metrology technique and enable limitations and possibilities to be determined.

6. References

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