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Accuracy and reproducibility of ambient topographies at the nanoscale by AFM: several months of metrological monitoring

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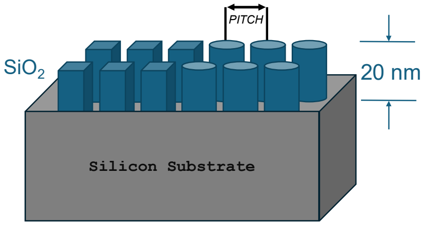
Abstract. With the high integration of microelectronic devices, nanoscale surface characterization is critical to process development. The widely used atomic force microscopy provides 2D characterization of the surface at the nanoscale. In this paper, a statistical study of the topographic mode PeakForce Tapping mode is performed. A silicon calibration sample with structures of 20 nm, is characterized, and from 28 days of measurements at the same location, data processing and analysis is proposed to determine the reproducibility of the height measured by AFM. From the section analysis, an uncertainty of 0.26 nm in air is determined. Statistical analysis is also used, and the mode is applied to characterize a dielectric material on the top of a highly integrated capacitor.

1 Introduction

With the high integration of microelectronic devices, the structural, electrical and mechanical properties of materials and system at the nanoscale is a key in the development. In recent years, Atomic Force Microscopy (AFM) has emerged as a key characterization technique[1], not only within research laboratories but also in industrial production lines, offering high spatial resolution and versatility[2]. Based on the interaction between the apex of a tip with the studied material, AFM allows to determine the topography by scanning the surface, measuring and controlling interaction forces at the nanoscale. The scanning direction has been a significant source of uncertainty from the comparison of three different AFM image processing software used to measure the step height of a monoatomic Silicon (111) crystal lattice[3]. Additionally, a comparison of SiO_2 nanoparticle size measurements using SEM and AFM revealed a higher level of uncertainty in SEM measurements, further demonstrating the accuracy and reliability of AFM for nanoscale metrology[4]. In this article, the intermittent Peak Force Tapping mode is studied in order to determine the reproducibility of the height AFM signal. For this, a microelectronic device consists of periodical step height of 20 nm structures was used. A metrological approach is investigated, Peak Force Tapping is used by two users and 28 days data are analysed. Furthermore, the method is applied on a high-integrated silicon capacitor sample for dielectric step height measurement.

1.1 A periodical Si/SiO_2 microelectronic sample

For the statistical and metrological study of nanoscale height determined by AFM, a commercial calibrated silicon sample is used. This sample ensures a reliable calibration in directions X and Y through the distinct size of the microelectronic structures, also a precise calibration in the Z direction through the fixed step height of 20 nm for all different geometries and shapes. The sample studied consists of a silicon chip, featuring fabricated silicon dioxide structures with different geometries like holes and pillars with circular, lines and square shapes. This silicon sample provides constant periodic arrays. All structures are fabricated on the same chip with a step height of 20 nm. Figure 1 represents a schematic view of the structure of the studied sample and a Scanning Electron Microscopy (SEM) top view.



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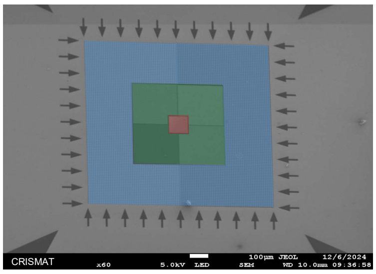
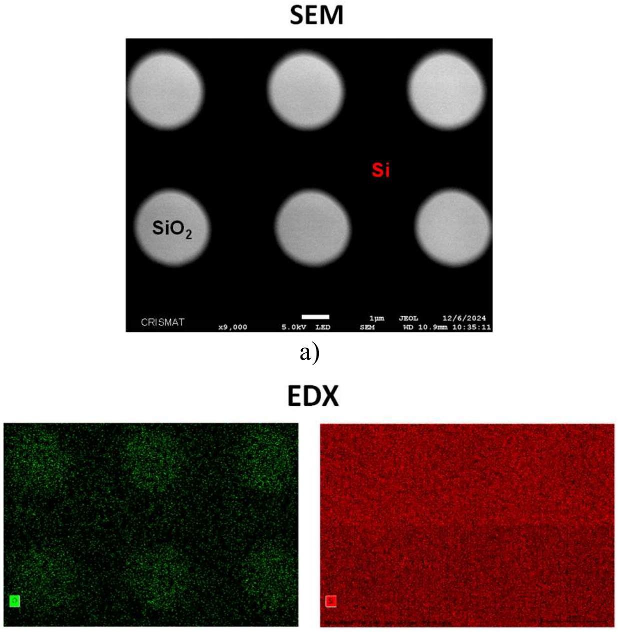


b)

Fig. 1. a) Schematic of the sample design showing circular and square pillars of SiO_2 having a 20 nm step height and b) SEM view of the calibration sample divided into three zones (colours are added to locate the region), in blue having a 10 µm pitch distance, zone 2 in green having a 5 µm pitch distance and zone 3 in red having only circular pillars with a 500 nm pitch distance.

SEM images, using a JEOL JSM 7200F were performed to get the contrast between the silicon substrate and the silicon dioxide structures as seen in Figure 2, Energy Dispersive X-ray (EDX) was also performed for silicon and oxygen. Elemental compositions maps are reported in the Figure 2, silicon and oxygen maps confirm silicon presence on all the scanned area and high oxygen concentration on the circular pillars, the higher concentration of oxygen on the circular pillars indicates the presence of silicon dioxide structures.



a)

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Fig. 2. a) SEM image of the periodic array of circular pillars with a fixed 5 µm pitch distance, b) corresponding EDX (Energy Dispersive X-ray) silicon and oxygen maps.

1.2 Atomic Force Microscopy and used tip

A commercial Bruker dimension ICON AFM is used for all acquisitions presented in this paper. This AFM allows different topographic modes: in contact, no-contact, intermittent (also called Tapping) and PeakForce Tapping. The AFM is installed on an anti-vibration isolation table inside an acoustic isolation. Measurements were performed in ambient environments and a sensor of temperature and humidity was connected to monitor changes during scanning.

The tip used for all topographic measurements is a scan-asyst air tip. This tip is made of silicon nitride, consisting of a 100 µm V-shaped cantilever of 0.65 µm thickness having a reflective aluminum coating. This cantilever has alow spring constant of 0.4 N/m, a resonance frequency of 70 kHz paired with a very sharp triangular tip having a 2 nm radius. Figure 3 shows the SEM images performed on a new scan-asyst air tip. The apex of the tip can be observed.

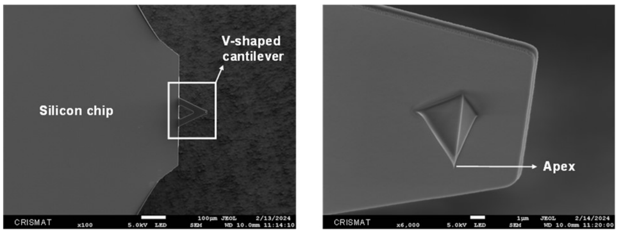


Fig. 3. SEM images of a new Bruker scan-asyst air tip, showing the V-shaped cantilever with its corresponding sharp tip attached to its end.

2 PeakForce tapping mode

First, the topographic mode PeakForce Tapping mode is used. This mode uses a sinusoidal wave having a 2 kHz frequency that induces the oscillation of the cantilever off its resonance frequency. A synchronization algorithm is set at about half of the period to extract the vertical maximum interaction force that is call PeakForce Tapping, this helps the reduction of the setpoint value with respect to the PeakForce value identified at each pixel [5]. The setpoint value is the maximum force applied by the tip onto the sample. Furthermore, another synchronization algorithm separates the tip sample interaction and non-interaction regions effectively so it can overcome the ringing signal fluctuation. The main advantage of this mode is its high scanning speed in parallel with accurate topography measurements with a minimum damage of the tip and the sample due to PeakForce control pixel by pixel. Figure 4 shows the schematic diagram of the PeakForce Tapping mode. Different stages are represented and the evolution of the force signal as a function of the time is reported.

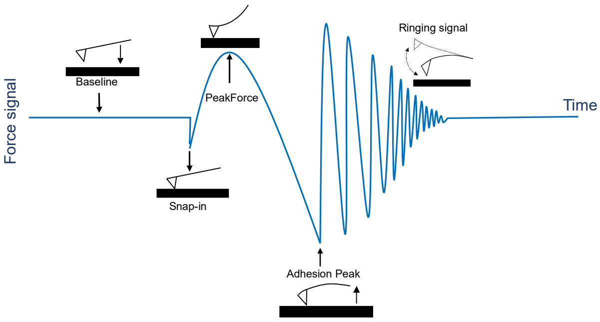


Fig. 4. Schematic diagram of the PeakForce Tapping mode.

The same scanning parameters were used for each repetitive scan performed in this paper. They are present in Table 1.

Table 1. The fixed scanning parameters with their corresponding values for each scan-asyst topographic measurement.

Scanning Parameter	Value
Scan Size	30 µm x 30 µm
Number Of Pixels	512 pixels x 512 pixels
Scan Rate	0.4 Hz
AC frequency	2 kHz

In order to characterize the used AFM tips, a sample called TGT1 consisting of a periodic array of peaks made of silicon is used. Topographic images are represented in Figure 5 (a). The PeakForce Tapping topographic measurement is performed on a single peak using a very slow scan rate of 0.1 Hz, a resolution of 512 × 512 pixels and a scan size of 1 µm x 1 µm for better accuracy. From this measurement, six section analyses are taken at the highest point of the acquisition while ensuring that each curve has the same maximum. This allows for the determination of the tip radius, which corresponds to the highest point across all six curves. The AFM 3D view of the used scan-asyst air tip, along with its corresponding section analyses, is shown in Figure 5 (c, b, and d) respectively. Based on the obtained graphs, the tip radius was found to be 2.7 nm, which is very close to that of a new tip, theoretically expected to have a radius of 2 nm. This result confirms that the PeakForce Tapping mode is a mode that minimizes the damage on the tip even after 6 scans.

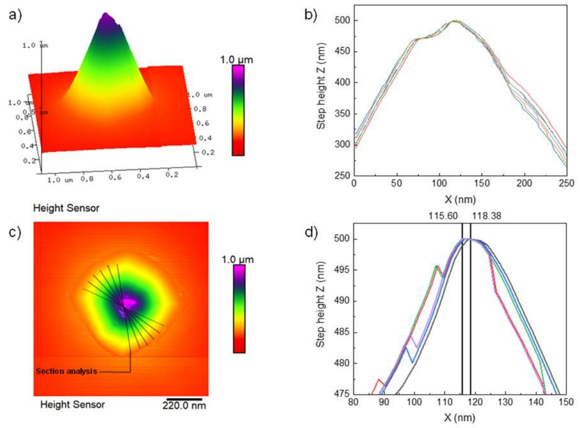


Fig. 5. a) 3D view of the AFM height sensor on the scan-asyst air tip, b) six section analysis on the highest point of the AFM acquisition on a single peak imaged, c) AFM 3D view of the height sensor acquisition of the used Bruker scan-asyst tip, and d) zoomed in view at the peak of graph b to extract the exact tip radius value.

3 AFM acquisition and image processing

After AFM measurements. NanoScope Analysis software is utilized for all image processing in this study. The calibration sample addresses sample tilt in the Z-direction, which can affect raw AFM data. To obtain accurate surface measurements, a flattening step is necessary to remove the sample tilt introduced when fixing the sample to the AFM holder. The flattening process can be complex and sensitive, potentially leading to inaccurate imaging results. In our case, 1st degree flattening is applied, which removes a linear tilt from the raw data. However, performing standard flattening on the whole AFM acquisition can produce misleading results for our samples that have structures with a 20 nm step height. A standard 1st degree flattening will take each line recorded on the acquisition and remove the tilt on each pixel, some lines will have the different shapes in our sample affecting the baseline level of our flattening leading to wrong results. To address this issue, a 1st degree flattening is implemented while excluding the silicon dioxide shapes from the calculation, in this case only the substrate is taken into consideration making the baseline at around 0 nm and leaving the real shapes of our SiO_2 structures. This approach ensures that only the baseline at around 0 nm is considered, preserving the true step height of our structures as seen in Figure 6 (b). The graph in Figure 6 (c) represents two cross section analysis, the red one corresponds to the raw data represented by the red line in (a) and the black line corresponds to the section analysis in (b) presented by the black line. This graph shows the importance of performing the right flattening step to obtain accurate AFM data without affecting the real step height of the SiO_2 structures.

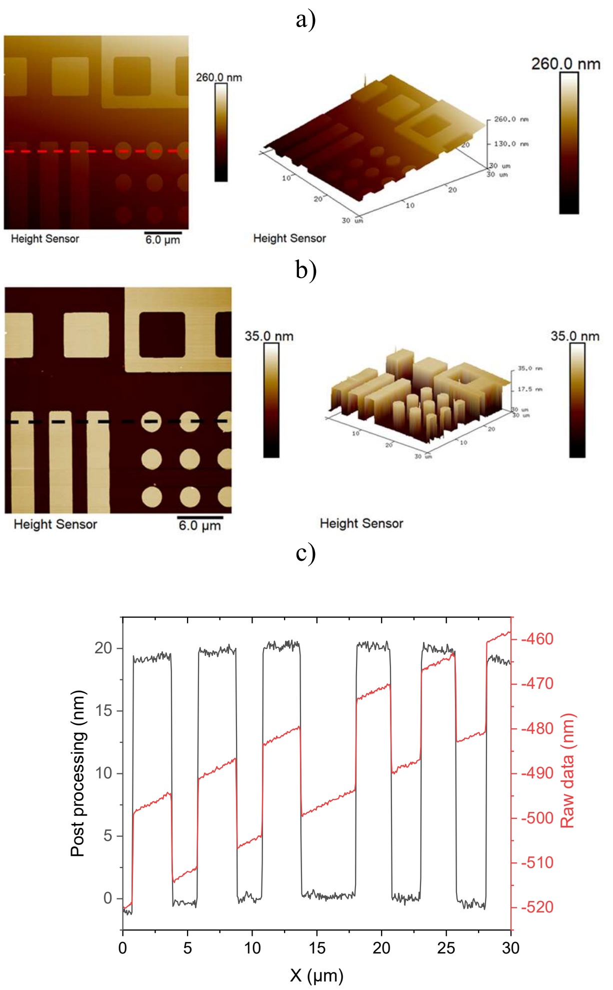


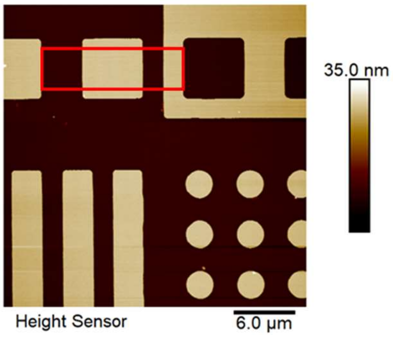
Fig. 6. a) The 2D map raw data AFM height sensor acquisition on the chosen zone of the sample with its corresponding 3D view b) flatten step with the excluded shapes, c) post flattening results on the height sensor acquisition with the 3D view and d) section analysis before and after the flattening process.

The designated area shown in Figure 6 is carefully selected for conducting all repetitive AFM measurements on the sample, motivated by two key factors. First, the area's distinct features allowed for easy optical detection, ensuring consistent positioning and enabling repeated measurements on the same structures across multiple sessions. Second, this region exhibited a variety of geometries, including square holes, square pillars, vertical lines, and circular pillars. This diversity within a single area provided a comprehensive representation of the sample's topographical features, facilitating a more thorough and efficient analysis of surface properties.

4 Section analysis on a single square pillar

For the metrological analysis, 28 measurements were recorded by two users. Figure 7 (a) represents the AFM acquisition of the calibrated sample with the use of six different tips of the same type. The total size of the acquisition is 30 µm x 30 µm. The height scale bar is 35 µm. From the AFM acquisitions, an area was selected for the metrological analysis. This area is the height of a square structure. An average section analysis having a size of 14 µm x 4 µm was taken in the red box represented in Figure 7 (a) for 28 different daily measurements. To be able to study the real step height variation of the square pillar present on this section analysis, the baseline should be at exactly 0 nm from its sides. To overcome this point, the mean value RMS from the baseline is subtracted from the image data scale to get a 0 nm baseline at the sides of the square pillar. After doing the same procedure for each of the 28 measurements, the plotted section analysis on the same graph is presented on Figure 7 (b), the focus will be on the variation of our step height on the pillar thus for x between 4 µm and 10 µm.

a)



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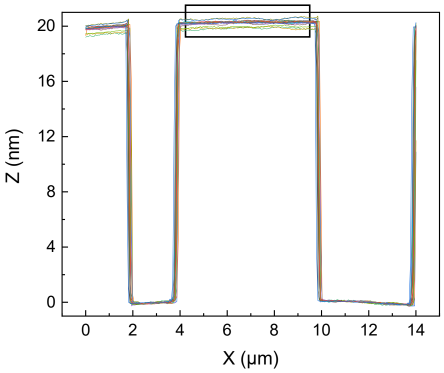

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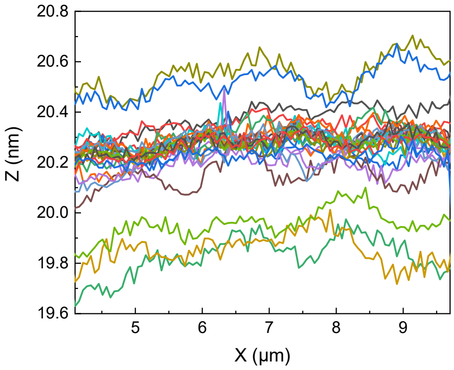


Fig. 7. a) AFM height sensor acquisition on the designated zone of the sample with a red box showing the place of the section analysis box, b) 28 curves for the same section analysis each one representing a day of measurement, c) zoomed in view on the square pillar for x between 4 µm and 10 µm.

For each curve recorded, the average value of the step height Z with its corresponding standard deviation was calculated and plotted with six different tips of the same type, each tip represented in a colour in Figure 8. The average step height value was found to be 20.260 nm with a standard deviation of 0.169 nm, thus the type A uncertainty (standard uncertainty of the mean) was of found to be 0.031 nm.

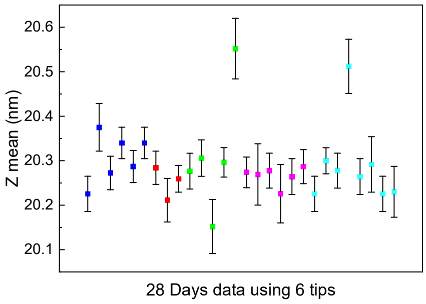


Fig. 8. Average value of the step height Z for each curve recorded on the square pillar, for 6 different tips used.

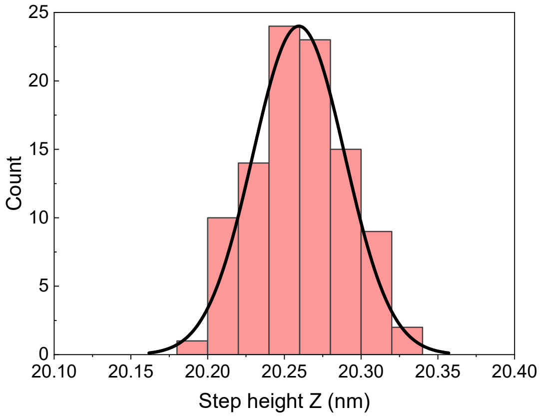
Moreover, the type B uncertainty is calculated by using the AFM uncertainties along the Z-axis given by the manufacturer Bruker. The uncertainty terms for the Z-axis taken into consideration are the Z noise, Z sensor performance, step height repeatability for a step of 20 nm and the roughness Rq. Table 2 shows the contribution of each term to finally add the type A uncertainty value and get the total uncertainty U with 95 % confidence. Finally, the step height is found to be 20.26 nm ± 0.26 nm.

Table 2. Uncertainty budget of the Bruker AFM dimension ICON for a step height of 20 nm.

TOTAL UNCERTAINTY U		
TERMS	**ESTIMATION**	**CONTRIBUTION**
Z Noise	0.035 nm	0.035 nm
Z Sensor performance	0.035 nm	0.035 nm
Step height repeatability (Z)	0.11 nm	0.11 nm
Roughness (Rq)	0.05 nm	0.05 nm
Type A uncertainty		0.031 nm
	Total U (95%)	**0.26 nm**

In addition, Figure 9 (a) shows a typical Gaussian distribution of the most found Gaussians distributions with its corresponding bins for the counts of the Z step height for 98 data points on each curve. Figure 9 (b) shows the histogram distribution of the step height Z for each curve analyzed on the chosen square pillar, each color on the histogram corresponds to a curve analyzed. We could clearly see on the histogram that the highest concentration of counts is for Z between 20.2 nm and 20.3 nm having a maximum of 1153 counts out of 2744 total data counts. For the curve distribution presented in Figure 9 (c), only 6 out of 28 curves that are not centered at the center of the Gaussian distribution at 20.26 nm.

a)



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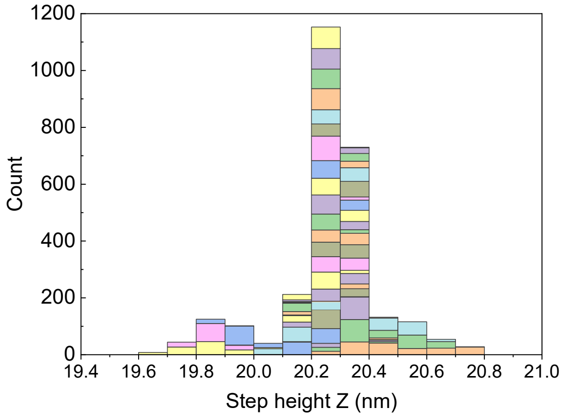

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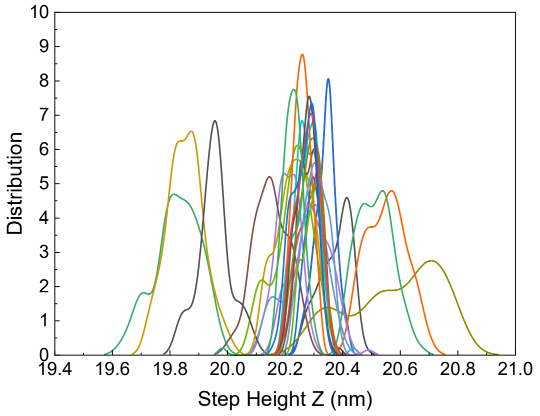

Fig. 9. a) Single curve distribution with its counts bins distribution, b) stacked histograms for all recorded curves and c) all recorded curves step height distribution.

5 Application

To illustrate the application of AFM for high integrated microelectronic device. Figure 10 shows two AFM topography measurements on the wafer surface using the PeakForce Tapping mode on a 3D Silicon capacitor manufactured by Murata Integrated Passive Solutions in Caen. Those capacitors are the second generation of its technology and consists of a metal-insulator-metal (MIM) configuration implemented within Si trenches to benefit from the third dimension of the silicon wafer to gain specific surface. Figure 10 (a) represents the top view of the 3D capacitor after the deposition of the top metal electrode. This electrode is made by depositing a highly doped polysilicon layer by Low Pressure Chemical Vapor Deposition (LPCVD). The size of this acquisition is 5 µm x 5 µm with a resolution of 512 x 512 pixels, the height scale bar is 650 nm. In order to reveal and measure the dielectric layer, a semi-automatic polishing step using polishing disks of 1 µm and 0.25 µm grain size is required. Figure 10 (b) shows the post polishing top of this capacitor. The scan size is 4 µm x 4 µm, the structure observed is the remaining Si 3D pillar after Bosch process, the dielectric layer and the trench filling with poly-Si. Most importantly, how the dielectric layer is polished differently from the top and bottom electrodes and have a step height Z of approximately 13 nm that is relatively close to the step height measured in this paper.

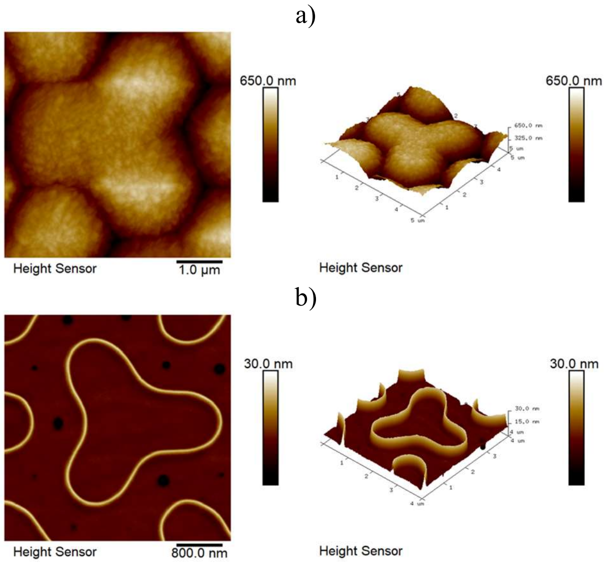

Fig. 10. a) Top view AFM topography acquisition (5 µm x 5 µm) on the 3D capacitor before polishing with its corresponding 3D view, b) top view AFM topography acquisition (4 µm x 4 µm) on the 3D capacitor after polishing with its corresponding 3D view.

This nano-metrological study on the step height measurement of the calibration sample and the studying of the reproducibility of our AFM topographic measurements using the PeakForce tapping mode will help to have accurate step height measurements on dielectric layer, characterized with the same mode and same conditions. Furthermore, this will help us perform very accurate topography mappings with a reproducible outcome with a low uncertainty budget, in parallel with very low damage on the tip and on the sample due to the PeakForce value adjustments pixel by pixel.

6 Conclusion

In this study, the reproducibility of AFM topographic measurements in PeakForce Tapping mode was evaluated using a calibration sample with 20 nm step height structures. The analysis revealed a step height measurement uncertainty of 0.26 nm, demonstrating the method’s precision. This metrological approach contributes to accurate and reproducible step height measurements, particularly for dielectric thickness measurement on highly integrated capacitors having a MIM configurations. Additionally, the results confirm that repeated scans cause minimal tip damage due to the controlled PeakForce values, ensuring measurement reliability over time.

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7 References

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