Surface roughness of NaCl coating used in thin film production

Contact *pawala.ariyathilaka@scitechprecision.com*

P.Ariyathilaka

Scitech Precision Limited, Rutherford Appleton Laboratory Harwell Campus, Chilton, Didcot, Oxon, OX11 0QX

D.Haddock

Central Laser Facility, Rutherford Appleton Laboratory, Harwell Campus, Chilton, Didcot, Oxon, OX11 0QX

Abstract

Surface roughness scans of NaCl (salt) coatings on silicon wafers were carried out using an Atomic Force Microscope. Four main scans were looked at, one with a salt coating and the other with salt and aluminum. Both were looked at straight from vacuum and again after being exposed to the atmosphere for a few days. Results suggest that the surface roughness increased drastically after being left out for few days; almost 5 times more when it was just salt, and approximately 1.5 times when it was salt coated with aluminum.

Introduction

Both Scitech and the Target Fabrication group (TFab) at the Central Laser Facility (CLF) have been doing simple salt (NaCl) coatings as a release layer for a number of years. Release layers are needed when making micro targets with thin film coatings. Without a release layer, the coating would adhere to the glass slide or silicon wafer. Once a material is coated on to the release layer, the float off method is used on the slides. Float off method involves carefully sliding the coated glass slides into a container of deionized water which dissolves the salt leaving the thin film floating on top of the water.

Salt coatings are done numerous times a week at Scitech but a thorough understanding of the coating is yet to be written down. But why is it so important to understand? Understanding the surface roughness of the salt coating as well as how it is affected with time will help the thin film coating production further. It is also important to see whether any imperfections on the salt coatings are carried through to subsequent coatings.

This report will look at the surface of a salt coating straight from vacuum and after a few days in the atmosphere. Also it will address the flatness of a subsequent coating on the salt layer. Figure 1 below shows an array of targets with an Al coating on top of a release layer salt coating.

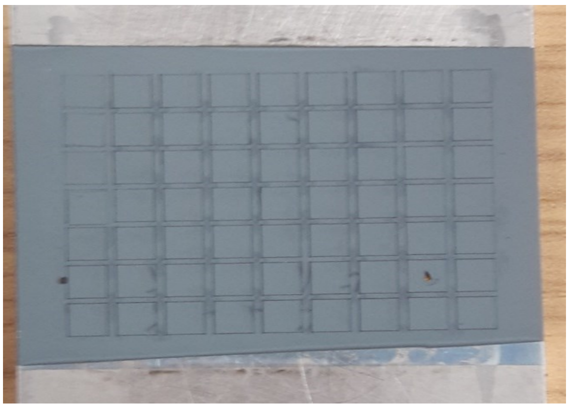


Figure 1 showing a thin film coating of Al on an array of targets.

Methodology

Two silicon wafers were plasma cleaned prior to any coating to remove any dust that might be on the surface. Then an ~100nm thick salt coating was deposited using physical vapour deposition (PVD). This method involves heating the material (salt in this case) until it sublimes. The salt vapor is then allowed to condense on the Si wafer producing a coating[1].

For an effective PVD process, the Si wafer needs to be in a good vacuum. (The better the vacuum then the longer the mean free path of the vapour giving a better coating.) The chamber pressure for the salt coating was 3.4×10^{-6} mbar. With the specific piece of kit used in this experiment a vacuum of 10^{-6} is considered good which will give long mean free paths. After this coating one of the Si test pieces was removed to study the surface whilst the second one was put under a vacuum of 2.5×10^{-6} mbar. On this ~100nm aluminum was coated using PVD.

Both the salt wafer and the salt + aluminum coated wafer went through two sets of scans. First one was straight from the vacuum chamber. A second set of scans were taken after both test wafer were left in the atmosphere for a few days.

Data collection

Both samples were scanned using an Atomic Force Microscope (AFM) in order to collect the surface roughness data. The AFM used in this experiment was a Veeco diCaliber with a resolution of around <0.6nm in the z direction.

AFMs can operate in scanning mode or in tapping mode. In this experiment tapping mode was used. The AFM operates by scanning a fine tip attached to the end of an oscillating cantilever across the sample[2] which gives the morphology of the surface. The cantilever oscillates around its resonance frequency and lightly taps the surface as the sample is scanned. An incoming laser beam reflects off the cantilever on to a position sensitive photodetector which contains a four segmented photodetector[3] as shown in figure 2.

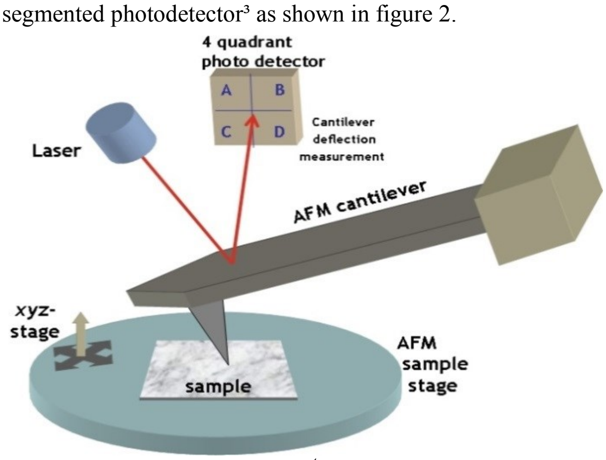


Figure 2 showing an AFM in action[4].

When studying a substrate there are surface imperfections that can be measured such as roughness, texture, waviness etc. The parameter being studied in detail in this report is the surface roughness which is the finest irregularities of a surface that arises from production processes.
The AFM settings used in this research were as below. The range for the scan was 5.1969um with 1024 pixels per line and a scan rate of 0.1Hz.

Data analysis software – Gwyddion
The AFM data generated was to be analyzed using Gwyddion. The program is ideal for flattening images and filtering noise to look at surface roughness. All the images in this report went through the following filtering process[5].

- Plane level = Which does a mean plane subtraction for the full set of data. The plane is computed from all the image points.
- Align using rows = This shifts the lines in the x axis such that the median of difference between vertical neighboring pixels is zero. This keeps rather large features.
- Remove scars = These are parts of the scan that is affected by common scanning errors. This function corrects for this.
- Three point levelling = Three points of similar height are selected then a plane is computed from the average of these points and subtracted from the dataset.
- Smooth = smooths the image by taking the mean of two neighboring pixels. This is done for all the remaining pixels.

Once the image is levelled, the roughness is measured using the roughness tool as shown in figure 3. To find the roughness a cut off value needs to be set. As a rule of thumb the cut off can be set to five times that of the spacing between the profile features of the sample[6]. With this in mind the cut off was set to 0.01 in this report. The value is specified in terms of Nyquist frequency.

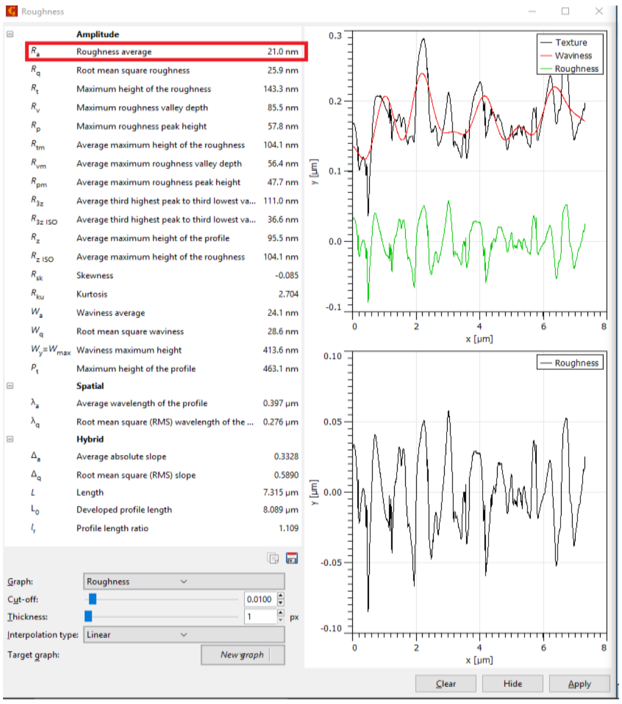


Figure3: shows the roughness tool in Gwyddion. In the red box is the average one-dimensional roughness.

Within Gwyddion, 1D roughness is defined as "Arithmetic mean deviation. The average deviation of all points roughness profile from a mean line over the evaluation length[7]".
This line is drawn through the filtered image in four different places as shown in figure 4 with the light grey line. The waviness is adjusted as previously stated. For consistency the four roughness values noted were approximately kept in the same area for all the scans. These were taken from different parts of each image.

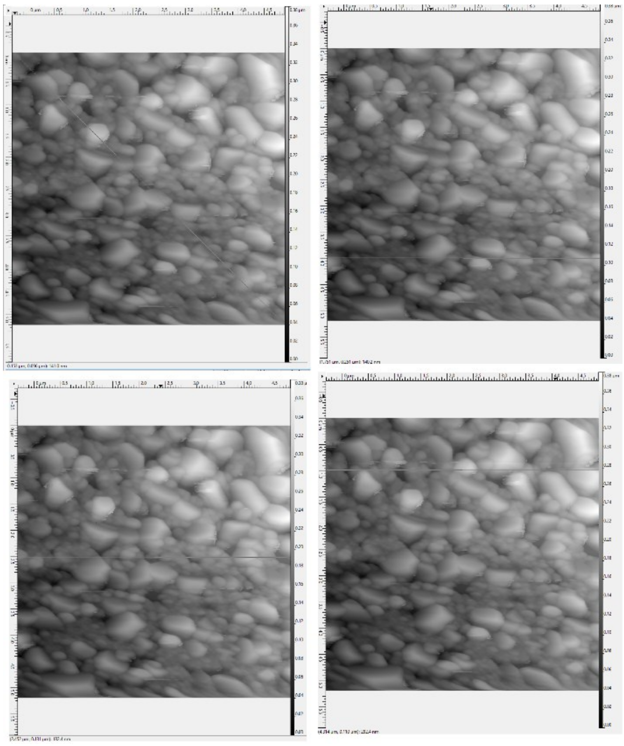
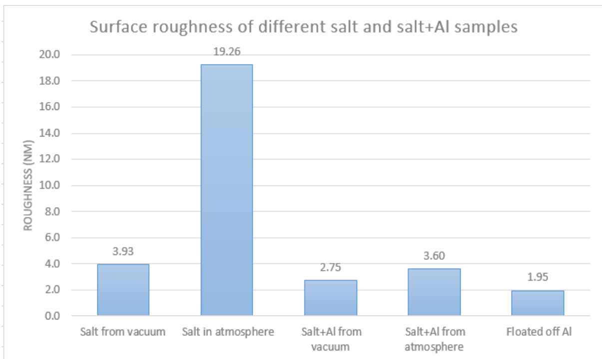


Figure 4: image showing where different 1D roughness lines were taken.

Data analysis

All the roughness values are shown on graph 1. The average roughness for a salt wafer from vacuum was 3.93nm which increased to an average of 19.26nm when left out in the atmosphere. The salt+Al coated wafer from vacuum gave a roughness value of 2.75nm, which increased to 3.60nm once left outside. The salt samples had increased by almost five times in roughness after they were left out in the atmosphere. This can be attributed to the salt grains being crystalized once in contact with the moisture in air. When the salt+Al sample was left out in atmosphere, the roughness increased on average by about 31%. This is less than a nm of roughness.



Graph 1: shows the roughness of the samples

It is also clear that salt left in air has a much higher roughness change that salt that is left with a coating of Al. A possible explanation could be that the Al coating has sealed in the salt completely, limiting the size of the crystals. By referring to figure 5 a visible difference between the two sets of data is clear.

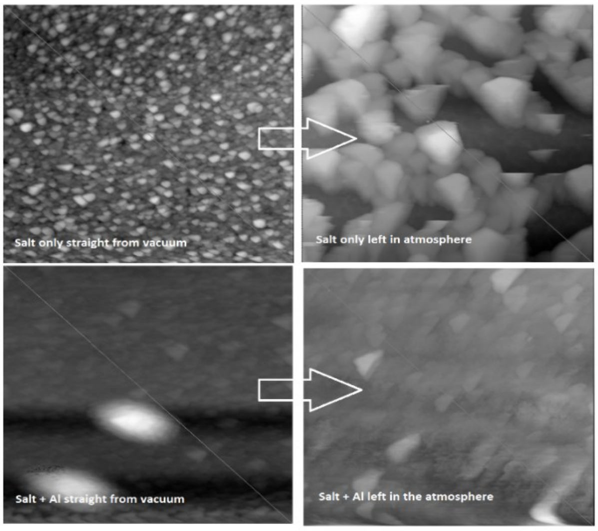


Figure 5: Image showing a comparison of a coating straight from the vacuum vs being left in the atmosphere. (Note that the image 'from vacuum' and 'left in atmosphere' are not the same area).

After the surface scans were done the test wafers which had salt+Al coatings were floated off using the float off method. The purpose of this was to see whether there are any discrepancies between a wafer with salt+Al and floated off Al. Figure 6 below shows the AFM scan of this. The average roughness was 3.06nm, suggesting that the higher roughness noticed on the Salt + Al samples were only present on the salt and hasn't influenced the floated off Al. An AFM scan was done on both sides of the floated off Al film to confirm that the roughness on both sides is similar. The values obtained were on average the same. So the morphology of the salt hasn't transferred completely to the coating. However, there were places of rough peaks which had a diameter less than a micron with a height between 10 and 20nms, as seen on figure 6. There were only few of these peaks per scan which had a total area of 36um. Also an EDX scan on the sample proved that no salt was left on the floated off Al.

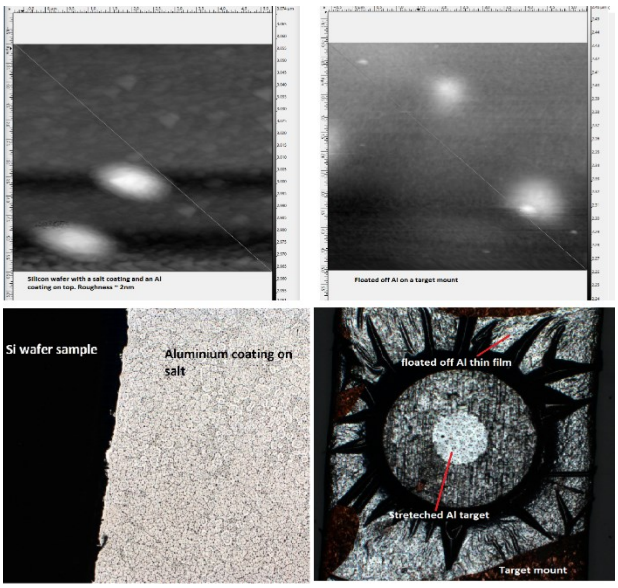


Figure 6: Image showing an AMF scan of the floated off Al

Further work

The work carried out here is only scratching the surface of a much broader subject. The next stage would be to repeat the same experiment a few more times to see if the results agree. All the coatings done here were using a thermal evaporation coating plant.

- In further studies, an electron beam evaporation method should be used and a comparison performed to see if similar results are found.
- Changing the thickness of the salt coating to see if that has an effect on the roughness. It might be interesting to see if a thinner coating gives a smother finish, and also to assess the effect of floating off.
- The rate of coating might also have an influence on the roughness. An interesting study would be to examine more samples produced with different rates.

Conclusions

This report looks at the surface roughness of salt coatings. Two samples were studied with repeated measurements; one with just salt and the other with an Al coating on top. AFM scans were performed both on samples straight from vacuum and the second scan on samples after leaving them in air for a few days. The results showed that the roughness of both the samples increased after they were left out in the atmosphere. Subsequent salt coating roughness change was very significant. The reason for this is the crystallization of the salt grains. The average roughness of the salt coating was 3.93nm straight after coating and 19.26nm after a few days. The roughness of the Al coating increased from 2.75nm to 3.6nm after being left out. So it is evident that the surface roughness changes after a few days. A similar flatness is observed in the resulting coating because the Al coating has a roughness of 3.06nm after being floated off. (This was after a few days exposure to air.)

There is a significant amount of future work that could be carried out to better understand salt thin film deposition processes for example comparing the current work with PVD coatings produced using e-beam equipment.

References

1. P.Mckenna and D.Neely Eds.. "Laser-Plasma interactions and Applications", Chap. 17 "Microtargetry for high power lasers".
2. Veeco Dimension 3100 AFM user manual. Accessed via: https://www.nist.gov/sites/default/files/documents/cnst/nanofab/Veeco_Dimension3100_AFM_USERMANUAL_v1.pdf
3. Wenjie Mai. Fundamental theory of AFM. Accessed via: http://www.nanoscience.gatech.edu/zlwang/research/afm.html
4. Atomic force microscope image. Accessed via: https://simple.wikipedia.org/wiki/Atomic_force_microscope
5. P.Klapetek, D.Necas, C.Anderson. Gwyddion user guide.
6. Ultra precision technologies presentation. Cut offs and measurements of surface roughness. Accessed via: http://www.taylorhobsonserviceusa.com/uploads/2/5/7/5/25756172/cut-offs_and_the_measurement_of_surface_roughness.pdf
7. P.Klapetek, D.Necas, C.Anderson. Gwyddion user guide. One dimensional roughness parameters, chapter 4.