Surface roughness of NaCl coating used as release layers in thin film production

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Abstract. Surface roughness scans of NaCl (salt) coatings on silicon wafers were carried out using an Atomic Force Microscope. Metrology was carried out on single layer salt and also salt coated with a thin aluminium layer which is a representative high power laser target. All were characterised 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 in atmospheric conditions for few days. The roughness increased by almost five time for an exposed salt coating. For a salt coating with an aluminium layer the roughness only increased slightly (~1nm). This suggests that it’s important to coat materials for the laser target on to salt release layers almost immediately from vacuum. This work enables production processes and quality control for high power laser targets to be defined and an understanding of possible pinhole production in thin film coatings.

1. Introduction
The use of salt as a release layer for thin film coatings to prepare targets for high power laser experiments has been used for many years [1]. In addition a soap solution, or other salt material can also be used, with each type or release mechanism having its benefits. The use of salt coated by vacuum deposition is desirable as the release layer thickness can be controlled precisely using the thin film crystal monitors in vacuum coating plants. A release layer is essential when fabricating thin film targets, as it is designed to stop the metal coating from adhering to the supporting substrate material. These slides can be lowered in to de-ionized water to lift the metal foil by dissolving the salt. The water tension keeps the metal foil suspended on top, which allows to pick up even a very delicate foil directly with the target mount.

Whilst work has been carried out to understand the release nature of the salt coating, the behaviour over time of this and its effect on the laser target is to be studied. It is important to have a clear idea of the origin of the surface roughness of this layer as any metal coated on the surface follows the same form. To provide consistent thin film targets, that are pinhole free and that survive at ultra-low thickness with a good surface finish, the surface of the release layer must be studied.

2. Experimental set up
In this report, two main conditions were investigated. The first parameter studied was the surface roughness study of a salt release layer straight from the vacuum chamber. The second parameter was the study of salt surface after being left out in the atmosphere for a week. The humidity of the class
10000 clean room is around 40 ± 2 percent. All the coatings were carried out using Physical Vapour Deposition (PVD) using a thermal evaporation source with a limited source size. The test samples used in this experiment were pieces of Silicon (Si) wafers with roughness of <1nm [2]. The coatings were repeated three times. The average values of the three are quoted in this paper. The Si wafers were plasma cleaned using an Oxygen plasma prior to any coating. A salt coating of 100nm was coated on to two sample Si wafer pieces. This method involves heating the material (salt in this case) until it sublimes. The salt vapor is then allowed to deposit on the Si wafer producing a coating [3].

For an effective PVD process and a high quality coating, the Si wafer needs to be in a good vacuum to have little residual water content on the substrate as well as to give a good mean free path to the sublimed salt. The chamber pressure for the salt coating was 3.4×10^{-6} mbar. Two Si wafer pieces were coated with salt. Immediately after coating, one of the Si pieces was removed and placed under an Atomic Force Microscope (AFM) to be characterised. The second sample was pumped back down to a vacuum of 2.5×10^{-6} mbar and coated with 100nm of aluminium (Al). During this process of removing the first sample the chamber was vented to dry nitrogen to avoid contamination and immediately pumped back down. A load lock, although desirable was not available in this case. The second sample was measured after it was coated with aluminium and then both measurements were repeated after being left at atmospheric conditions for a few days.

3. Data Collection
Figure 1 below shows the way data collection took place. Both Si samples were scanned using an AFM 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.

![Figure 1. Image shows the data collection carried out in this project.](image)

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 [4], which gives the morphology of the surface. The cantilever oscillates close to 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 [5] as shown in figure 2.
Surface characteristics can be described on different length scales, roughness for high spatial frequencies and waviness for variations on larger scale. In this report, surface roughness was studied further which is the finest irregularities of a surface that arises from a production process of a material. The AFM tip used for the measurements in this report has a radius of curvature of 20nm. The AFM settings were as below. The range for the scan was 5.1969um with 1024 pixels per line and a scan rate of 0.1Hz.

4. Data analysis software – Gwyddion
The AFM data generated needs to be analyzed to give it clarity. Gwyddion is ideal for flattening and filtering AFM images to look at the surface in more detail. All the images in this report went through the following flattening process [7].

- 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.

With the AFM tip being 20nm in radius, a Fourier filter was applied to remove the very high frequencies with a cut off wavelength of 1/20nm. Once the image is leveled and filtered, the roughness is measured using the roughness tool. An image of an example 1D roughness scan is shown in figure 3. To measure the roughness, a cut off value needs to be set. The cut off in Gwydion is in terms of Nyquist frequency, this was set to 0.003 in this report, with a pixel size of ~5nm this equals to a cut off wavelength of ~3um. It should be mentioned that the roughness measurements here are not done exactly to ISO standards. The same cut off is chosen for all the data for a direct comparison.
Figure 3. Shows the roughness measurements in Gwyddion. For this particular 1D roughness scan, the roughness was 29.5nm. This figure is for a scan of a NaCl sample exposed to atmosphere.

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 [8]’. This evaluation line is drawn through the filtered image in five different places as shown in figure 4.
Figure 4. Image above shows where different 1D roughness lines were taken. The roughness lines are shown in red. The average roughness values quoted in the paper are the averages of the above lines, averaged over the three repeated samples. Also note that the 1D roughness are drawn separately when measuring the roughness. The above image is only drawn to show the 1D roughness lines.

5. Discussion
The roughness values are shown in figure 5. The average roughness for a salt coated Si piece from vacuum was 7.7nm which increased to an average of 38.1nm when left out in the atmosphere which was an increase by almost 500%. This can be attributed to the salt grains being crystalized once in contact with the moisture in air. The salt and Al coated Si wafer sample from vacuum gave a roughness value of 3.5nm, which increased to 4.7nm once left in atmosphere for a few days, a change of approximately 1nm. It is noticeable that a salt coating left out in has a much higher roughness variation over time than that of the coating of salt and aluminium. A possible explanation could be that the aluminium coating has sealed the salt completely from the atmosphere, or limits the moisture penetration, limiting the size of the crystals. The difference in the two example salt coatings are shown in figure 6.
Figure 5. Shows the roughness of the samples

Figure 6. Image showing a comparison of a coating straight from the vacuum (left) vs being out in the atmosphere (right).

After the surface roughness scans were complete, the test Si wafers samples which had salt and aluminium coatings were floated off using the standard method of dipping in de-ionised water. The purpose of this was to see whether there’s any big discrepancies between the measurement on a wafer and a measurement that was carried out after processing the foil into a target geometry. The average roughness for a floated off foil (now single layer aluminium as the salt is dissolved) was 4.1nm, suggesting that the slightly larger roughness noticed on the salt and aluminium from atmosphere samples were only present on the salt later and hasn’t influenced the floated off foil. An AFM scan was done on
both sides of the floated off film. The roughness on both sides show no significant difference. It is possible to infer that the morphology of the salt hasn’t transferred completely to the coating when it is removed from the salt substrate.

![Photograph showing optical micrograph images of Al coating on Si wafer (left) and an assembled Al target on a Cu mount. (right)](image)

**Figure 7.** Photograph showing optical micrograph images of Al coating on Si wafer (left) and an assembled Al target on a Cu mount. (right)

6. **Conclusion**

This report looked at the surface roughness of salt (NaCl) coatings that are used as release layers in laser target manufacture. Two samples were looked at in detail with each coating being repeated three times. One sample had a coating of 100nm salt only and the second sample had 100nm salt followed by 100nm of aluminium. The roughness was measured with an AFM for all the samples. For both samples, one measurement was done straight from the vacuum chamber and the second was once the samples were left out in the atmosphere for a week.

The results found show that the roughness of both the samples increased after they were left out in the atmosphere with the single layer salt coating showing the largest and most significant roughness increase. It is concluded that the reason for this is the crystallization of the salt grains due to the moisture in the air. The salt coating with an aluminium top coating showed reduced increase in roughness, and this is suggested to be due to the barrier layer that is introduced between the salt and the atmosphere preventing some crystallization.

It is also noted that when a target foil (the aluminium layer) is removed from the substrate (salt) and suspended over a target support, a similar flatness is observed as for the unexposed target foil on a substrate, even after it has been exposed to a few days out in the atmosphere, and whilst that is encouraging for target foils, for filters and applications that require pinhole free foils, the stress on the thin layer in being on a rough surface and then flattened out again could cause small tears and pinholes in the foil. In further studies the same experiment could be carried out with varying salt thicknesses to determine whether this has any effect on the roughness of the target foil. To achieve a flat coating for a laser target, the time between the salt release layer coating and target material coating must be minimum. However exposing the release layer to atmosphere could be a way to develop some surface structure in a controlled manner.
References

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