DEPOSITION OF Ti CLUSTERS ON Si SUBSTRATE USING DC MAGNETRON SPUTTERING

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Introduction

Clusters are aggregates of atoms or molecules, generally intermediate in size between individual atoms and aggregates large enough to be called bulk matter. Cluster study draws interest for several reasons: Fundamental physical interest due to their reduced size. Technically this is an interesting material because of their unique electronic and optical properties. Also, they have potential as catalyst for chemical reactions. Our study evolves in three parts:

- Study of cluster formation in a tubular vacuum chamber and cluster growth.
- Deposition of clusters on a substrate varying deposition time, at different vacuum chamber temperatures and applied substrate voltages.
- AFM-, XPS and X-ray reflectivity study of the deposited clusters.

Experiment

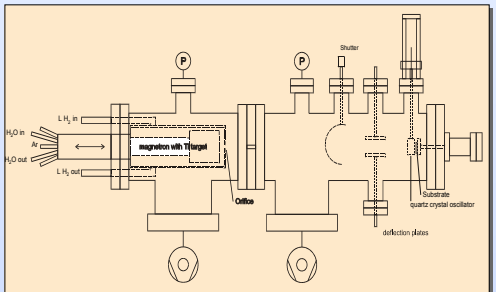


Figure 1 : Schematic view of the experimental setup

Materials and Methods

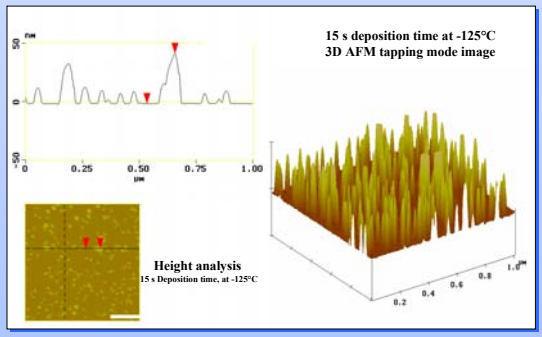
Sputtering and Deposition equipment: We use a **NC200** nanocluster source with **Ti** as target. **DC Magnetron** discharge is used to generate the clusters inside a liquid nitrogen cooled aggregation tube with Ar as working gas. After cluster formation it is deposited on a Si substrate. The schematic view of the experimental set-up is shown in **figure 1.**

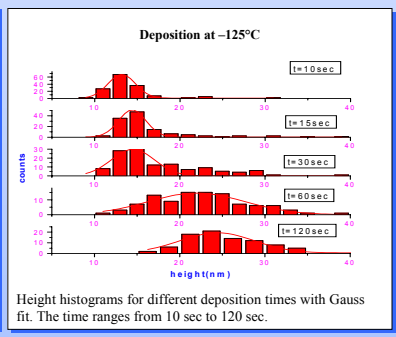
X – ray Photoelectron Spectroscopy (XPS): The chemical composition of the film is analysed by XPS using a VG Microtech MT 500 system, with magnesium anticathode in the x-ray tube producing characteristic Mg Kα rays of 1253.6 eV operating at 12 kV and 20 mA.

Atomic Force Microscopy (AFM): We use tapping mode AFM (Digital, nanoscope IIIa) to image the substrates with the clusters. The scanning AFM tip is oscillating at a resonant frequency; due to interaction with the surface, at close approach the amplitude shifts.

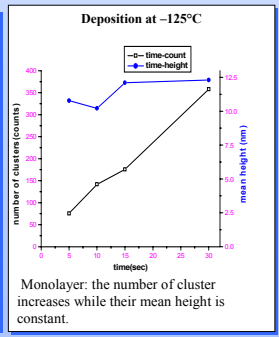
X - ray Reflection method: Average particle size, average surface coverage and particle cross-section can be determined by using X-ray reflection method.

Results and Discussion

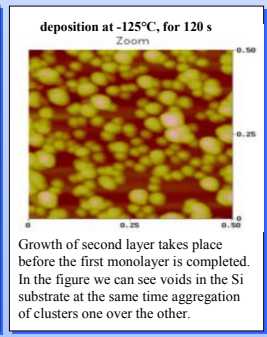




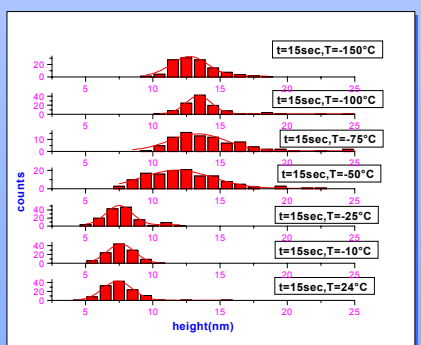
Height histograms for different deposition times with Gauss fit. The time ranges from 10 sec to 120 sec.



Monolayer: the number of cluster increases while their mean height is constant.



Growth of second layer takes place before the first monolayer is completed. In the figure we can see voids in the Si substrate at the same time aggregation of clusters one over the other.



Histograms of the cluster diameters with Gauss fit, as determined from the AFM by varying the temperature in the vacuum tube.

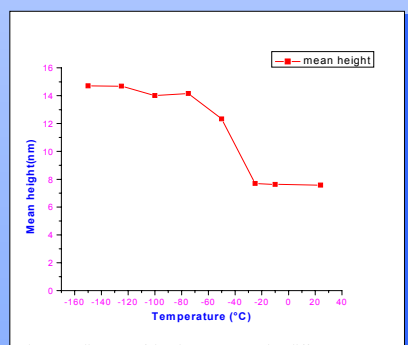
Conclusion

With a NC200 nanocluster source using DC magnetron discharge Ti clusters are formed. The clusters were deposited on a substrate, and characterised with AFM, XPS and X-ray reflectivity.

AFM results show that first a monolayer with clusters of constant height forms within the first 30 seconds, the number of clusters increases linearly with deposition time. Then, growth into the third dimension starts. Eventually, after 30 min deposition time, XPS results show that the Si substrate is completely covered by Ti_xO_y.

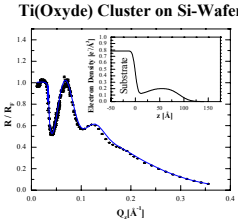
On cooling the cluster forming chamber, it is found from AFM that the height of the clusters increases from 8 – 13 nm, especially in the temperature range of 20°C to –80°C. On cooling, the velocity and thus the mobility of atoms decreases, causing larger clusters. Applying a voltage to the substrate is another, less efficient way to control the cluster size.

XPS measurements were done after exposing the sample it to ambient air. Then, it was found that the Titanium to Oxygen ratio is 1 to 2, suggesting oxidation in air forming TiO_2. Since Titanium metal is coated with an oxide layer that usually renders it inactive. The same is observed with a substrate covered with clusters.

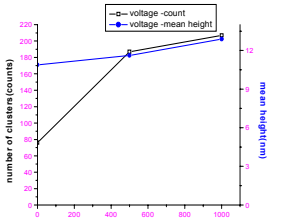


The mean diameter of the clusters prepared at different temperatures in the cluster forming chamber, as deduced from the histograms.

Ti(Oxyde) Cluster on Si-Wafer

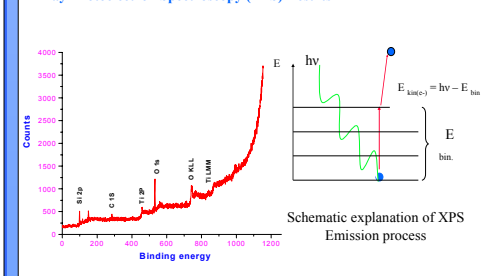


Normalised X-ray reflectivity of a sample containing Ti_xO_y clusters on Si substrate (t = 0.5 min), with the electron density profile used to simulate the measurements (solid line). The average cluster diameter is calcultaed to be 10 nm, ($d=2\pi/\Delta Q_z$), which confirms the AFM results.

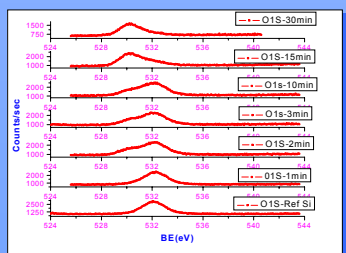


On increase of the substrate voltage, a higher surface coverage is found. This feature is attributed to an increased attraction between clusters and the substrate.

X- ray Photoelectron Spectroscopy (XPS) Results



Schematic explanation of XPS Emission process



The XPS spectra of Oxygen peak show a regular transition from SiO2(binding energy 532 eV) to TiO2 (binding energy 530 eV) with increase of deposition time, After 30 min the SiO_2 substrate is completely covered.

References

(1). Hellmut Haberland, *Clusters of atoms and molecules,* Springer – Verlag, New York (1994)
(2). Karl-Heinz Meiwes- Broer, *Metal Clusters at Surfaces,* Springer – Verlag, New York (1997)