Characterization of Low Temperature Aluminum Oxide (Al_2O_3) Atomic Layer Deposition

by Andrew Chen and Richard X. Fu

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14. ABSTRACT

Al_2O_3 was deposited on the surface of silicon wafers at 200, 150, 100, 80, 50, 25 °C using a Cambridge Nanotech Fiji Atomic Layer Deposition (ALD) tool. The low temperature atomic layer deposition of ultra thin films is very important because many substances cannot withstand high temperature depositions due to low melting and boiling points. ALD is traditionally performed at a high temperature, but low temperature deposition is needed for more fragile and delicate substances. The use of ultra thin films and low temperature atomic layer deposition is directly applicable to semiconductor engineering and many nanotechnology applications. We studied the effectiveness of low temperature atomic layer deposition and compare it to high temperature atomic layer deposition by measuring oxide thickness, resistivity, surface roughness, and pinhole density. We found that as the temperature of deposition was decreased, the oxide thickness increased, and the resistivity of the film decreased. Surface roughness and pinhole density also increases when the temperature of deposition is dropped.

15. SUBJECT TERMS

Atomic Layer Deposition (ALD), Plasma ALD, pinhole density, resistivity, Al_2O_3 film, AFM

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1. Introduction

Atomic Layer Deposition (ALD) is a unique technique for depositing ultra thin and conformal films on the substrates (*1-4*). However, this process is optimized to be run at a higher temperature. ALD has numerous applications in both nanotechnology and semiconductor engineering (*5-8*). Low temperature ALD is critical because many delicate and temperature sensitive substrates such as mercury cadmium telluride (HgCdTe), grapheme, polymer, cannot withstand a high temperature deposition. In this project, we investigated ALD of Al_2O_3 films onto silicon wafers at low temperatures: 200, 150, 100, 80, 50, and 25 °C. After the depositions were completed, various tools including the Leica Optical Microscope, Prometrics 4-Point Probe, Woollam Spectroscopic Ellipsometer, Veeco Nanoman Atomic Force Microscopy (AFM) were used to measure the pinhole density, resistivity, oxide thickness, roughness values of the films in order to determine the quality of the Al_2O_3 films.

2. Experimental

ALD Al_2O_3 films were deposited on silicon wafers at temperatures of 200, 150, 100, 80, 50, and 25 °C with plasma assisted deposition used the Cambridge Nanotech Fiji ALD machine. The precursors that we used were Trimethyl Aluminum (TMA), and Oxygen. Argon is used as the purge gas which removes excess precursors from the machine.

Upon completion of the ALD, the wafer is moved to the Woollam Spectroscopic Ellipsometer for film thickness measurement. The Prometrics 4-Point probe was employed for resistivity measurements. The Veeco Nanoman AFM was used to determine film surface roughness and morphology by two-dimensional (2-D) and three-dimensional (3-D) images.

3. Results and Discussion

3.1 Oxide Thickness

The Woollam spectroscopic ellipsometer was used to measure the oxide thickness of the Al_2O_3 film on the silicon wafer. We found that as the temperature of the atomic layer deposition was lowered, the oxide thickness of the Al_2O_3 was increased (table 1 and figure 1).

Table 1. Plasma ALD Al_2O_3 film thickness at different temperatures.

Temperature (ºC)	**Cycles**	**Oxide Thickness (Å)**	**Min (Å)**	**Max (Å)**	**Std. Dev.**	**Uniformity (%)**
25	200	334.59	332.74	336.85	1.88	0.91
50	200	304.73	302.31	307.05	1.70	0.56
80	200	266.50	264.58	268.31	1.82	0.68
100	200	238.31	236.91	239.55	1.17	0.49
150	200	217.53	216.08	219.04	1.11	0.51
200	200	193.89	193.44	194.40	0.37	0.19

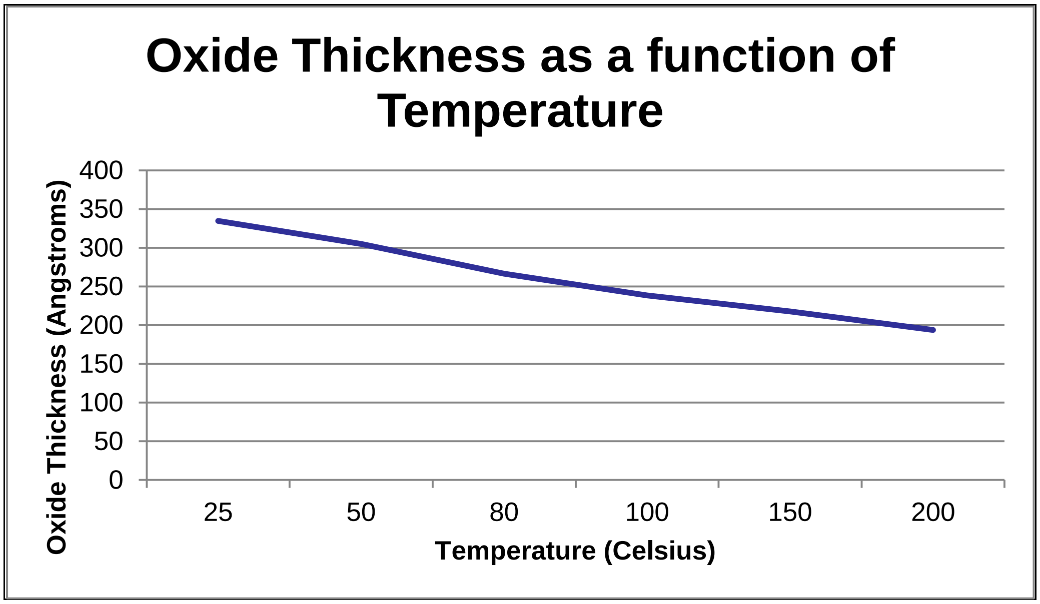


Figure 1. Plasma ALD Al2O3 film thickness at different temperatures.

3.2 Al_2O_3 Film Resistivity

We took resistivity measurements of the Al_2O_3 film using the Prometrics 4-Point probe. Resistivity is a material property that gauges how strongly a material opposes the flow of electrical current. A standard 5-point test was used when measuring resistivity (figure 2).

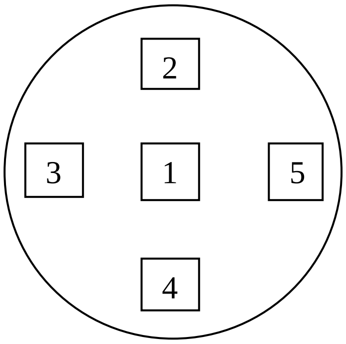


Figure 2. Resistivity test locations on the wafer.

From table 2 and figure 3, we can see that the resistivity of the Al_2O_3 film decreased with the deposition temperature decreasing.

Table 2. Plasma ALD Al2O3 film resistivity at different temperatures.

Temperature of Deposition (°C)	Number of Cycles	1 (Ω/Sq)	2 (Ω/Sq)	3 (Ω/Sq)	4 (Ω/Sq)	5 (Ω/Sq)	Average (Ω/Sq)
25	200	65.01	65.16	64.62	66.68	66.61	65.61
50	200	74.43	72.29	74.12	74.19	77.03	74.54
80	200	68.05	67.42	67.49	67.81	70.56	68.27
100	200	70.95	68.87	69.87	67.91	70.51	69.62
150	200	73.38	69.57	69.47	81.14	71.62	73.04
200	200	105.7	113.6	122.2	127.0	114.8	116.66

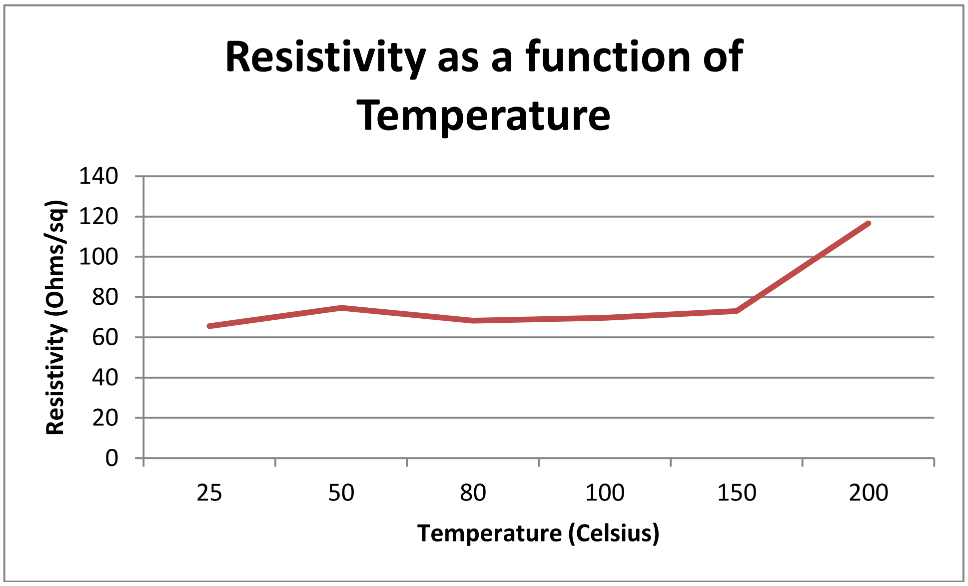


Figure 3. Plasma ALD resistivity at different temperatures.

3.3 Al_2O_3 Film Surface Roughness and Morphology

Using the Veeco Nanoman AFM, we were able to study the Al_2O_3 film's surface roughness and morphology. All of the films had relatively smooth surfaces regardless of the deposition temperature. The AFM allowed us to find two different roughness values, Ra and Rq. The Ra value is the arithmetic average of the absolute values of the surface height deviations measured from the mean plane. The Rq roughness value is the root mean square average of height deviations taken from the mean image data plane (table 3 and figure 4).

Table 3. Plasma ALD Al_2O_3 film roughness.

Temperature (ºC)	Number of Cycles	Rq (nm)	Ra (nm)
25	200	0.222	0.161
50	200	0.226	0.160
80	200	0.204	0.157
100	200	0.187	0.145
150	200	0.161	0.124
200	200	0.139	0.108

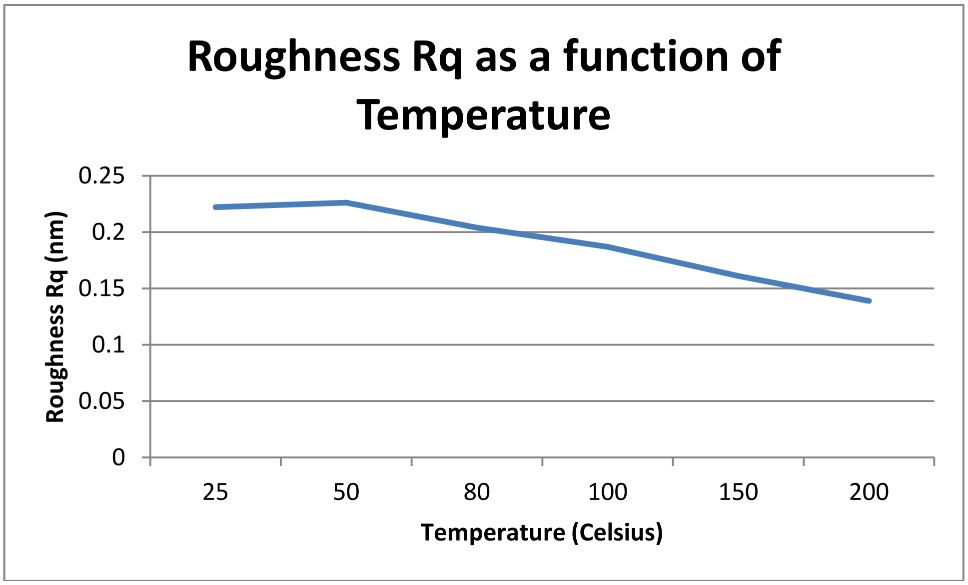
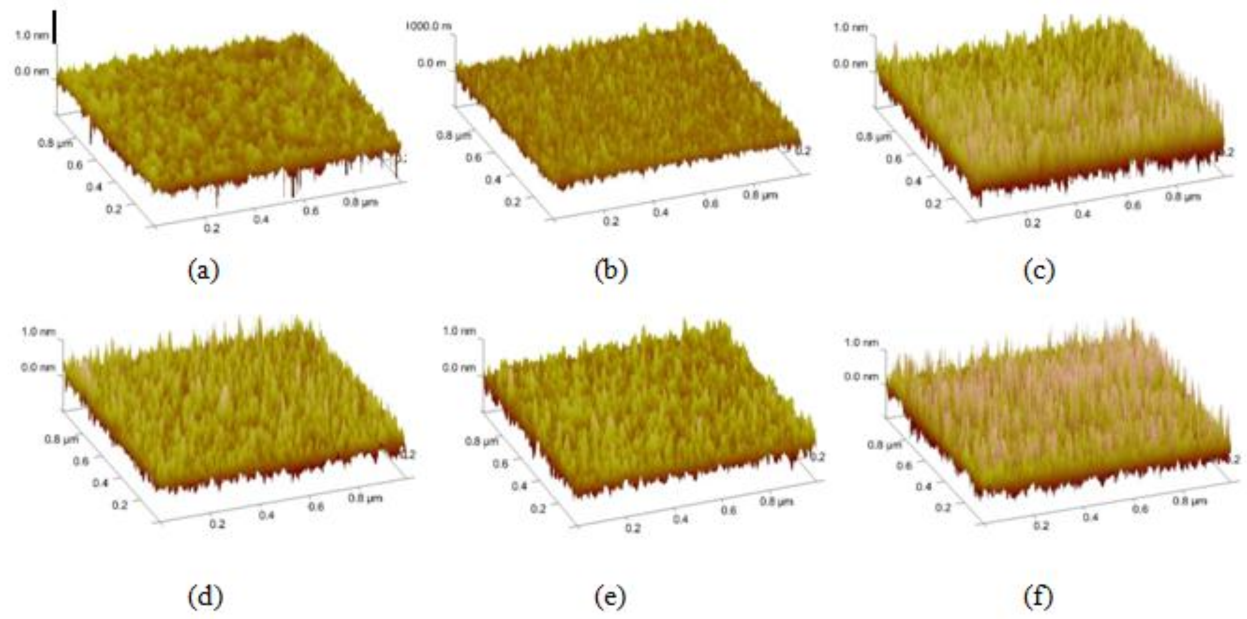


Figure 4. Plasma ALD Al_2O_3 film roughness Rq at different temperatures.

At the lower deposition temperatures, the roughness Rq and Ra values were higher than the ones at high temperature depositions. However the Al_2O_3 films are thicker at lower temperature depositions even though all wafers were deposited on for 200 cycles.

In figure 5, AFM film images were measured on a 1 nm depth scale and 1 µm by 1 µm area. The darker colors indicate a thinner film while brighter colors up to pink show a thicker one. At 20 ºC (figure 5a), there is a very noticeable variation in colors, and the heights of the peaks, indicating a rougher film compared to the 50 ºC deposition (figure 5e). The 3-D imaging of the AFM was very practical in providing us quality image of the surface and its morphology. We could easily see the fluctuations in height and tell how rough the film surface was. Figures 5a–f show how the surface morphology change as the ALD deposition temperature increases. The peaks become smaller as the ALD temperature is increased, and the samples are generally darker in color because the samples are smoother.



(a) (b) (c)

(d) (e) (f)

Figure 5. AFM 3-D imaging of the Al_2O_3 film deposited 200 cycles at different temperatures: (a) 200 °C; (b) 150°C; (c)100 ºC; (d) 80 ºC; (e) 50 °C; (f) 25ºC.

The AFM also provided 2-D images of the Al_2O_3 films; this showed the differences in the thickness of the Al_2O_3 film adjusted solely to a colored scale. Large fluctuations between dark and light, such as brown and pink, indicate a great change in the thickness, meaning a rougher film (figure 6).

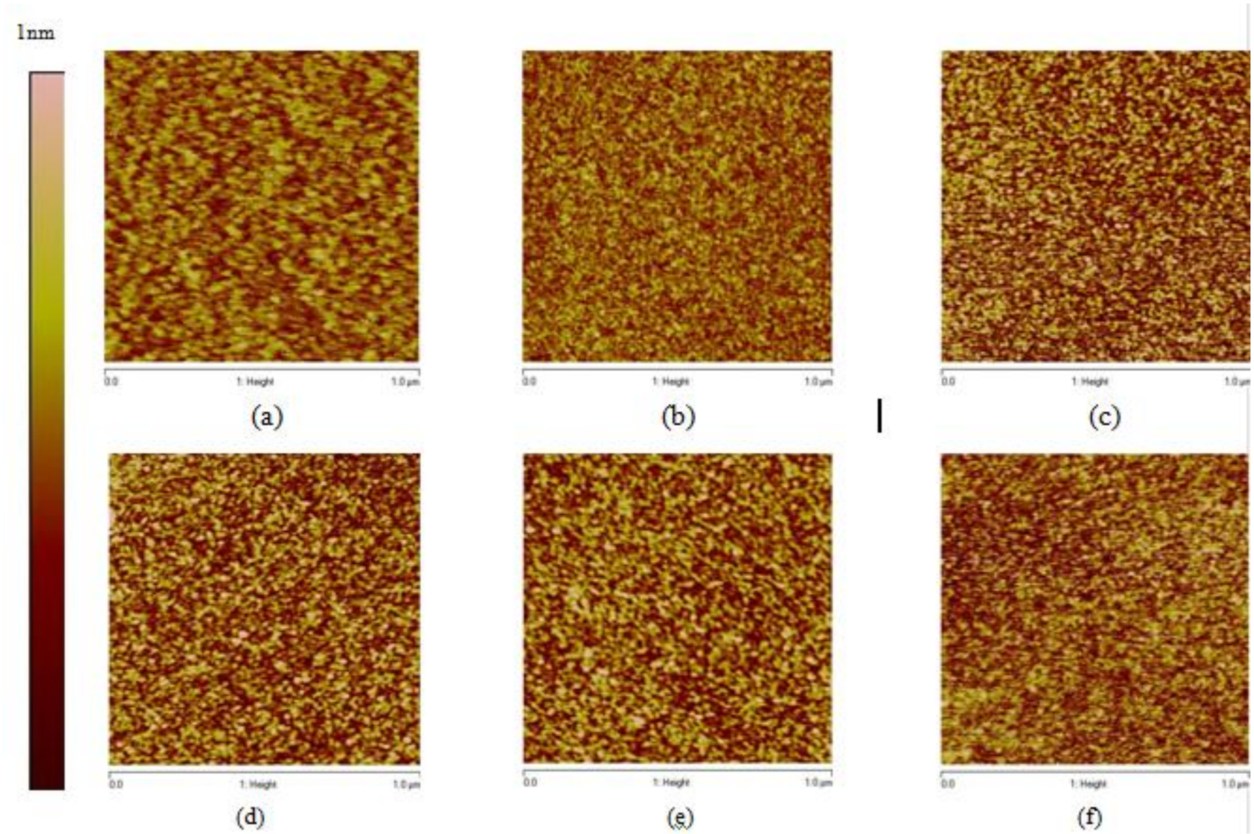


Figure 6. AFM 2-D imaging of the Al2O3 film deposited at different temperature for 50 cycles with plasma-assisted ALD: (a) 200 ºC, (b) 150 ºC, (c) 100 ºC, (d) 80 ºC, (e) 50 ºC, (f) 25 ºC

Progress from figure 6a–f, we can notice that there is a greater variation in the color of the 2-D images. This means that there are larger height variations as the ALD temperature is decreased and that the films are becoming rougher. The color scale allows variations in the thickness to be easily noticed on the 2-D imaging of the AFM.

3.4 Pinhole Density

ALD is well known for its extremely uniform thin film deposition; however, even ALD films still have pinholes in the film. A higher pinhole count or pinhole density indicates poorer quality of film. A Leica Optical microscope was used to examine and take images of the surface of the Al_2O_3 film. A representative field of view was selected on each silicon wafer on the 10x magnification. The number of pinholes would be divided by the total area of the field of view ($4.43*10^{-2}$ cm^2) to find out the pinhole density of the film. As we lowered the temperature of deposition we notice a gradual trend upwards, though there was an anomaly at 80 ºC where it had the highest pinhole density of all the films (table 4, figures 7 and 8). If we exclude this film then there is a very obvious correlation between temperature of deposition and pinhole density; the higher the temperature, the lower the pinhole density.

Table 4. Pinhole density of Al2O3 films at different temperatures.

Temperature (°C)	**Number of Pinholes**	**Pinhole density (cm^2)**
25	5	112.87
50	4	90.29
80	7	158.01
100	4	90.29
150	3	67.72
200	2	45.15

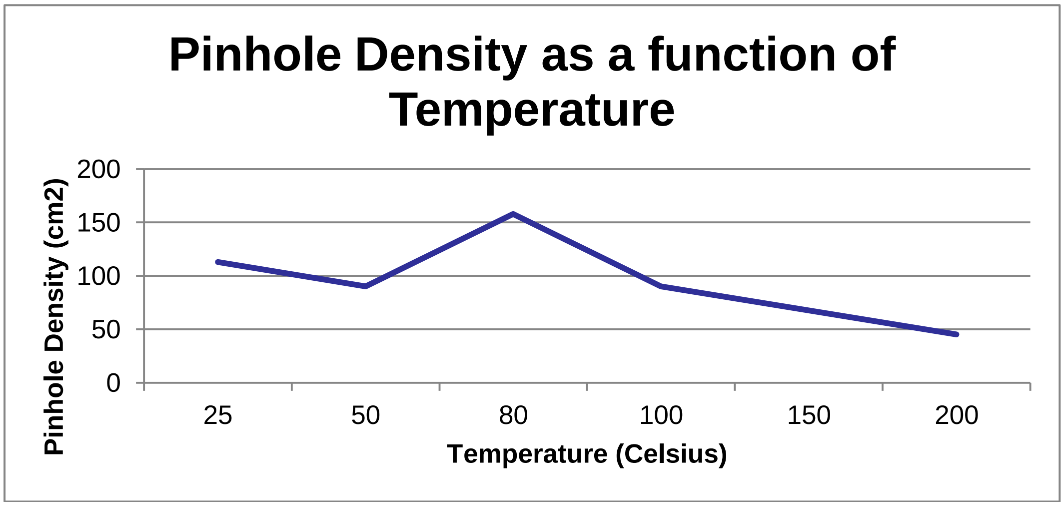
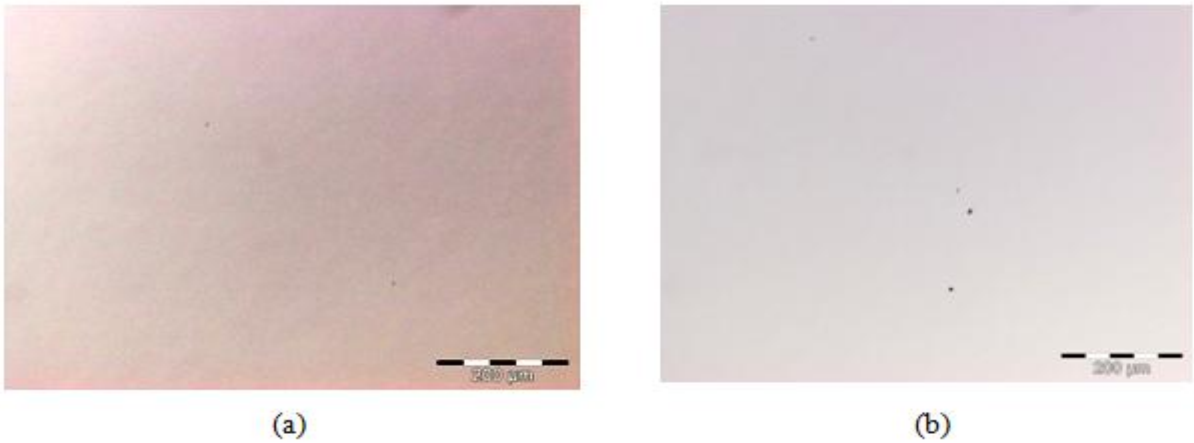


Figure 7. Graph of pinhole densities of Al_2O_3 at different temperatures.



(a) (b)

Figure 8. Optical microscope field of view of pinhole densities of Al_2O_3 at different temperatures: (a) 200 °C and (b) 50 °C.

4. Conclusions

From our experiment, we can conclude that as the ALD temperature is decreased and the resistivity of the film is decreased. As temperature of deposition is lowered, the oxide thickness of the Al_2O_3 film is thicker. The pinhole density of the film appears to increase as the ALD temperature is decreased. According to AFM measurements, roughness also increases as the temperature of deposition is decreased. However, even at 25 ºC, the good quality of film was produced.

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List of Symbols, Abbreviations, and Acronyms

2-D	two-dimensional
3-D	three-dimensional
AFM	Atomic Force Microscopy
Al2O3	aluminum oxide
ALD	Atomic Layer Deposition
HgCdTe	mercury cadmium telluride
O2	oxygen gas
Ra	roughness, Arithmetic average of the absolute values of the surface height deviations
Rq	roughness, root mean square average of height deviations
Si	silicon
SiO2	silicon oxide
TMA	Trimethyl Aluminum

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