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CeO_2 Deposition using E-beam#2

Objective: To develop an e-beam-grown CeO_2 film with a smooth surface profile.

Experimental:

1) Loading a Si sample onto the hot chuck, loading CeO_2 e-beam material into a graphite crucible, putting the crucible into one of pockets of crucible holder, changing a new, Ag-coated quartz crystal monitor (QCM), and putting a heat shielding if doing a high temperature deposition.

2) Pumping the chamber pressure down, and, after the cross-over from mechanical pump to cryogenic pump, setting the chuck temperature (e.g., inputting 475 for 200^{o}C) and letting it heats up.

3) Setting the program (prog#6) parameters as: 1) density=7.13; 2) z-ratio=1.00; 3) tooling factor=100.

4) After the chamber pressure pumping down to 3~4Xe-6 (at this time, the target chuck temperature should already be reached), turning the BOC and Beam Sweep switchers on, turning the power supply on, pressing the buttons of high voltage and filament current, slowly tweaking the current up until one can see the beam spot on the CeO_2 material through the window.

5) Lowering the beam sweep frequencies along both the longitudinal and lateral directions to 2, then, adjusting the amplitudes and positions of the beam spot along the both directions so that the spot covers the material within the crucible, then, increasing the sweep frequencies along both the longitudinal and lateral directions to 32 and 15, respectively.

6) If the oxygen gas flow is needed, turning the gas knob to a vertical position to let gas in, then, turning the gas flow switch on, slowly increasing the gas flow to the target one (the maximum O_2 gas flow is 45 sccm, beyond which the high-voltage and current will be shut down by itself).

7) Adjusting the filament current so that the CeO_2 deposition rate reaches the target and waiting for 5 minutes for degassing, then, zeroing the deposition thickness and opening the shutter to perform the film deposition.

8) Shutter will be closed when the target film thickness is reached, then, setting the chuck temperature to 15 and letting it cool down (let the O_2 flow for another 20 minutes before turning the gas flow off).

9) Venting the chamber when the chuck temperature drops to below 100^{o}C and getting the sample.

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Results:

1) Sample#1:

Deposition Condition: O_2 flow-rate=40sccm, at room temperature, tooling factor=80, deposition rate=1 Å/s, nominal thickness= 100 nm, using non-heating sample holder (base pressure=3.25e-6 Torr; the pressure during the deposition~4.48e-4 Torr).

Result: film thickness=315 nm, n=1.737 at 632.8 nm (MSE=36.1, fit range=458-1684 nm). The calibrated Tooling Factor=252.

2) Sample#2:

Deposition Condition: O_2 flow-rate=40sccm, at 250^{o}C (580), tooling factor=220, deposition rate=1 Å/s, nominal thickness= 100 nm, using heating sample holder (base pressure=3.75e-6 Torr; the pressure during the deposition~4.10e-4 Torr).

Result: film thickness=45.3 nm, n=1.896 at 632.8 nm (MSE=34.3, fit range=375-1685 nm). The calibrated Tooling Factor=99.7.

3) Sample#3:

Deposition Condition: No O_2 flow, at 200^{o}C (475), tooling factor=100, deposition rate=0.5 Å/s, nominal thickness= 100 nm, using heating sample holder (base pressure=4.45e-6 Torr; the pressure during the deposition~3.45e-5 Torr).

Result: film thickness=117.1 nm, n=2.123 at 632.8 nm (MSE=26.4, fit range=400-1688.4 nm). The calibrated Tooling Factor=117.

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Figure 1 Refractive index, n, and extinguish coefficient, k, of sample#3 (Bill Mitch took the measurement).

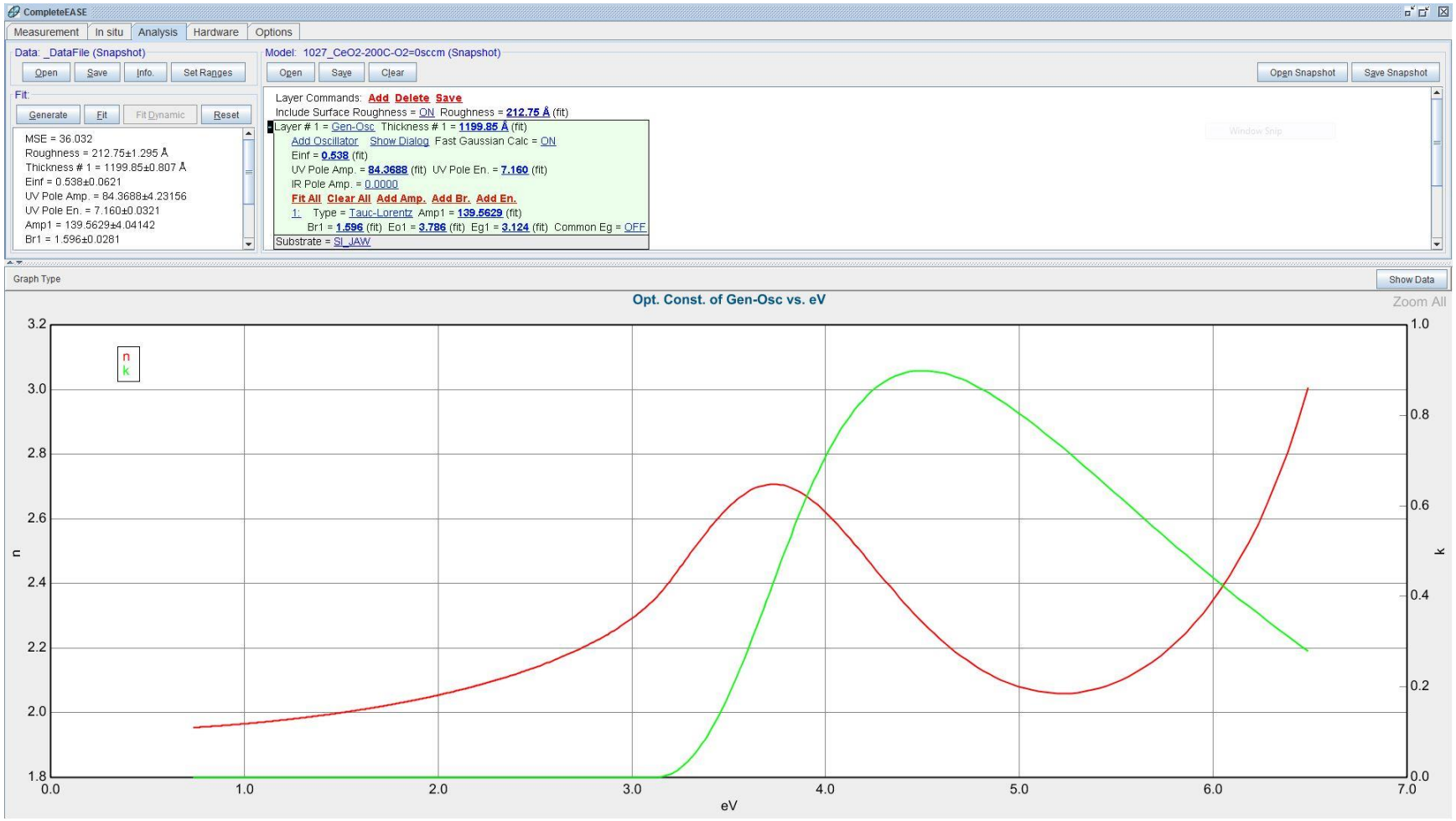
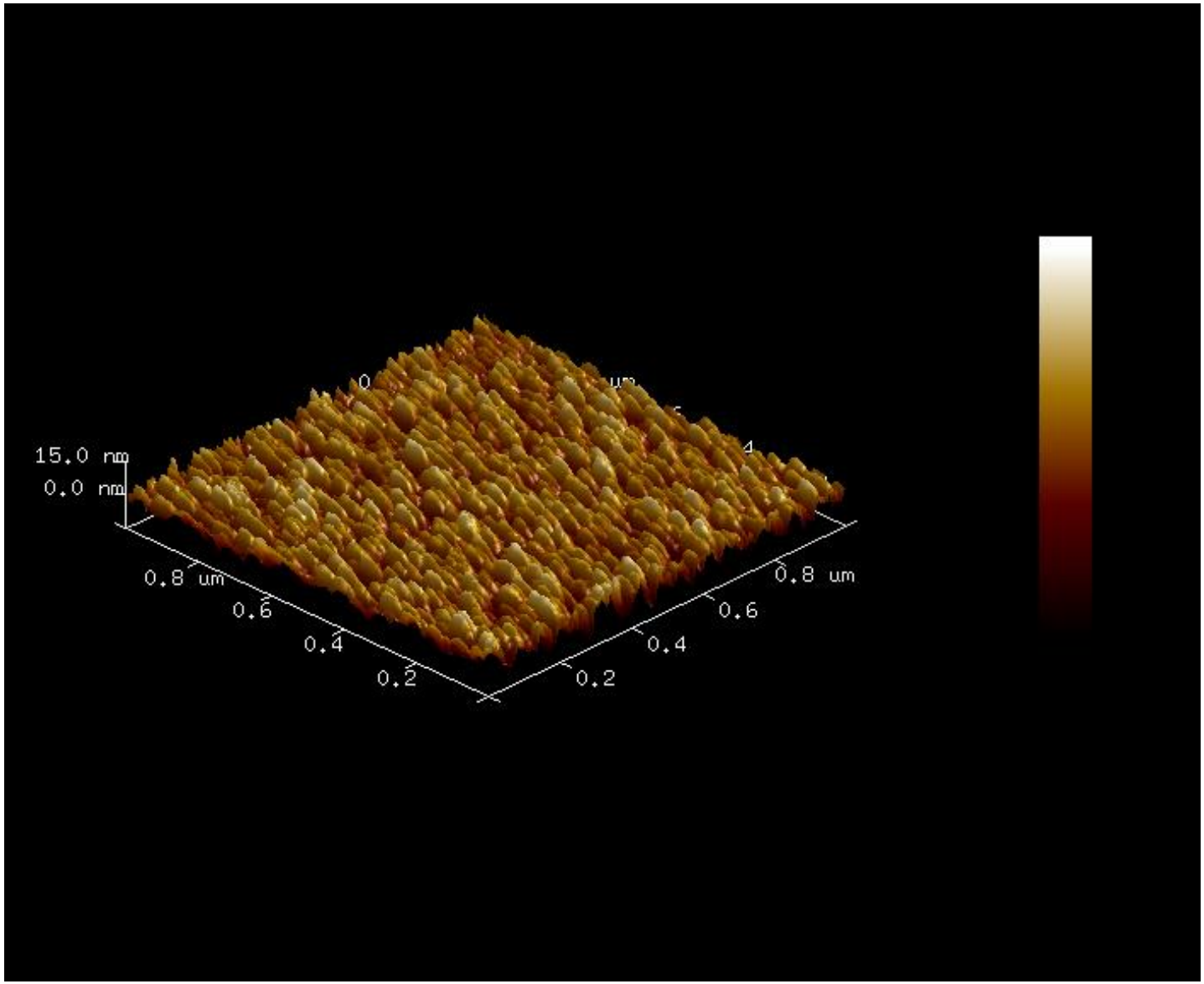


Figure 2 AFM of sample#3: it shows there is a column structure (Bill Mitch took this sample surface scan).



4) Sample#4:

Deposition Condition: O_2 flow-rate=45sccm, at 200^{o}C (475), tooling factor=100, deposition rate=0.5 Å/s, nominal thickness= 100 nm, using heating sample holder (base pressure=3.80e-6 Torr; the pressure during the deposition~4.5e-4 Torr).

Result: film thickness=130 nm, n=1.797 at 632.8 nm (MSE=42.8, fit range=400-1688.4 nm). The calibrated Tooling Factor=130.

Figure 3 Refractive index, n, and extinguish coefficient, k, of sample#4 (Bill Mitch took the measurement).

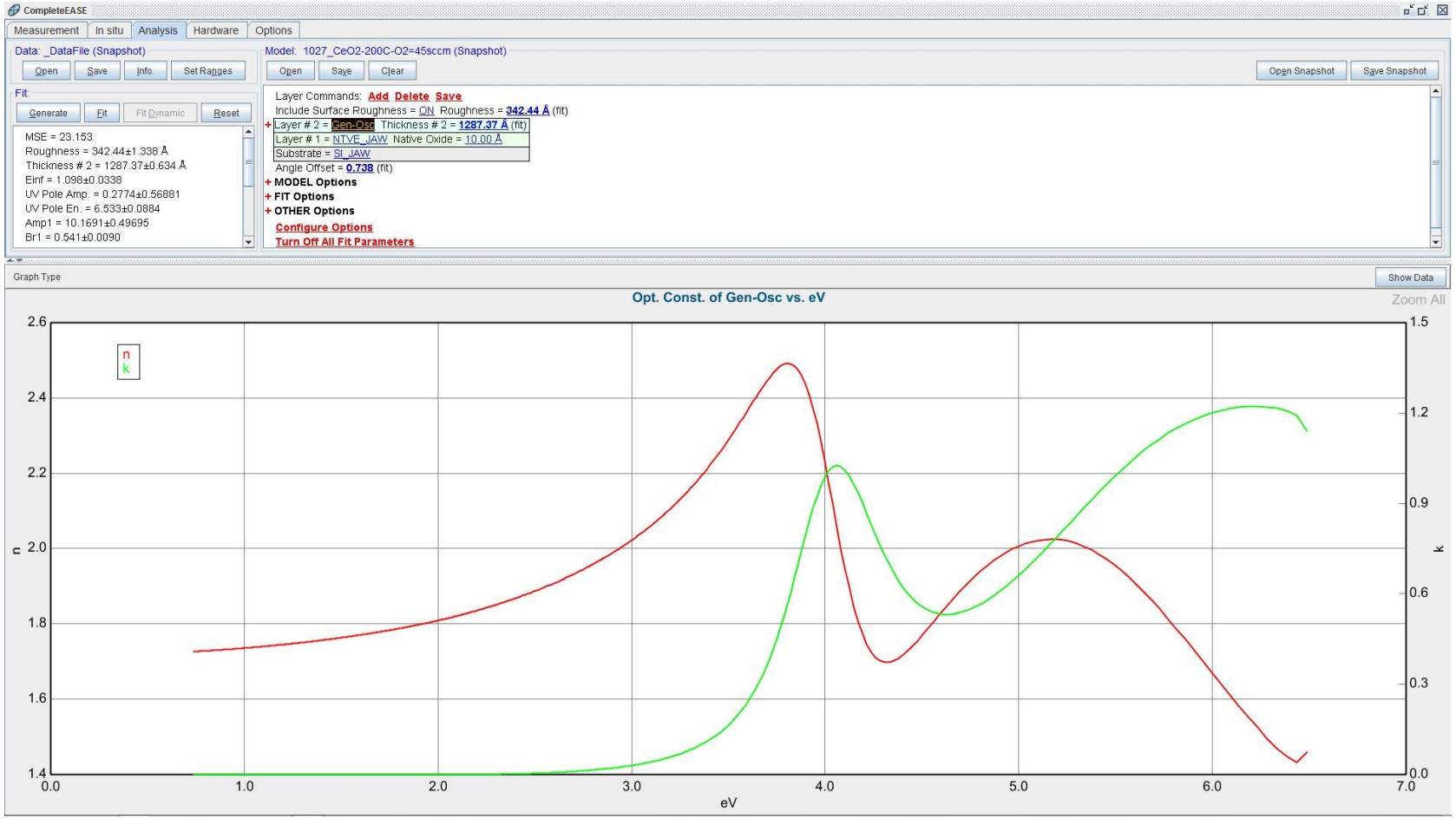
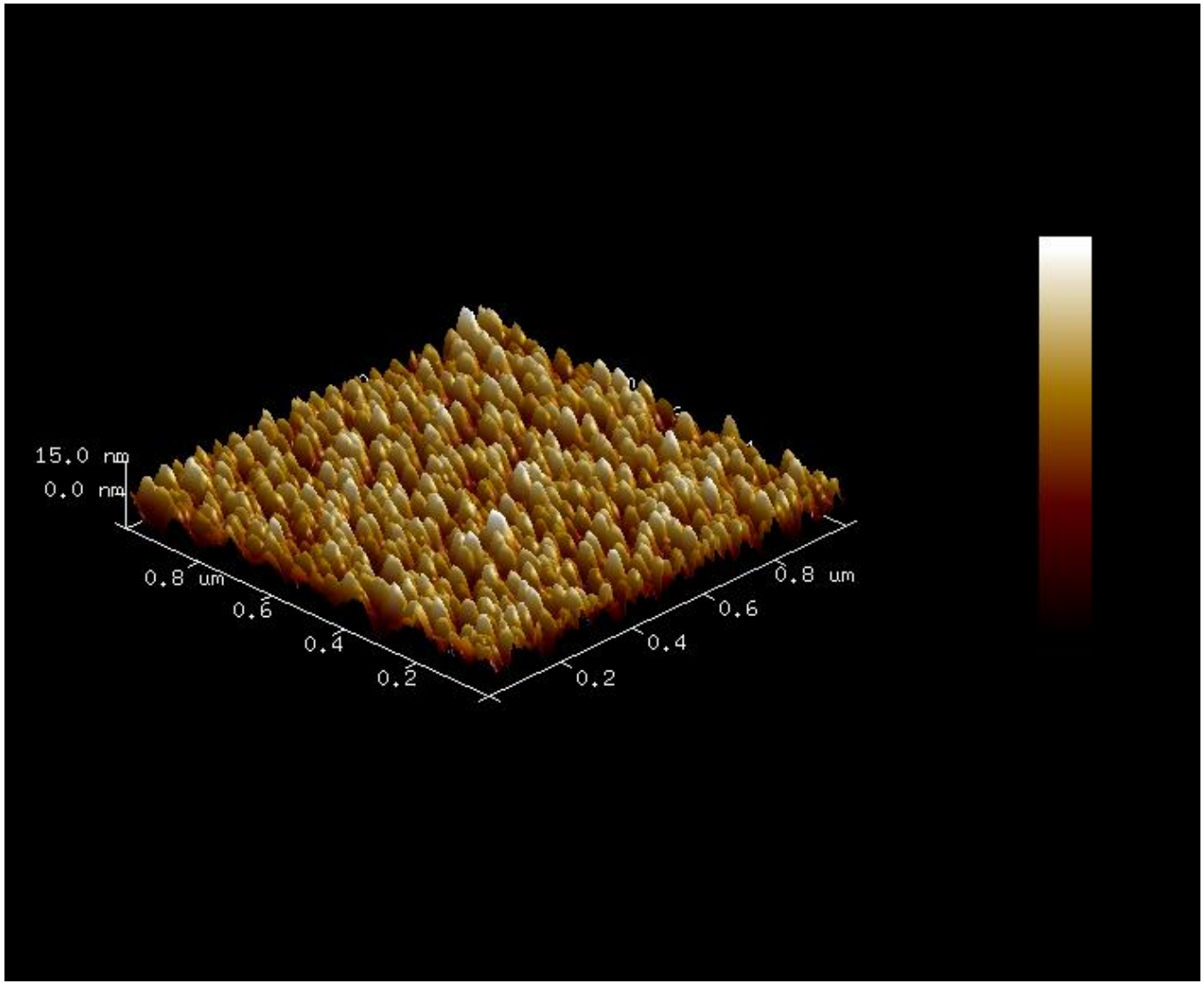


Figure 4 AFM of sample#4: it shows there is a column structure (Bill Mitch took this sample surface scan).



5) Sample#5:

Deposition Condition: No O_2 flow, at 200^{o}C (475), tooling factor=100, deposition rate=0.5 Å/s, nominal thickness= 100 nm, using heating sample holder (base pressure=6.50e-6 Torr; the pressure during the deposition: starting at 5.07e-5 Torr., 2.90e-5 Torr. at 50nm, 3.00e-5 Torr. at 100nm).

Result: film thickness=111.9 nm, n=2.14183 at 632.8 nm (MSE=26.2, fit range=400-1688.4 nm). The calibrated Tooling Factor=112.

6) Sample#6:

Deposition Condition: No O_2 flow, at room temperature, tooling factor=220 (using lower room-temperature sample holder, deposition rate=0.5 Å/s, nominal thickness= 100 nm (base pressure=9.50Xe-6 Torr; the pressure during the deposition: starting at 3.64e-5 Torr., 2.04e-5 Torr. at 50nm, 1.42e-5 Torr. at 100nm).

Result: film thickness=97.37nm, n=1.90236 at 632.8 nm (MSE=11.8, fit range=400-1688.4 nm). The calibrated Tooling Factor=214.

7) Sample#7:

Deposition Condition: O_2 flow-rate=45sccm, at room temperature, tooling factor=220 (using lower room-temperature sample holder), deposition rate=0.5 Å/s, nominal thickness= 100 nm (base pressure=4.53e-6 Torr; the pressure during the deposition: starting at 4.38e-4 Torr., 4.37e-4 Torr. at 50nm, 4.38e-5 Torr. at 100nm).

Result: film thickness=107.77nm, n=1.70775 at 632.8 nm (MSE=16.9, fit range=400-1688.4 nm). The calibrated Tooling Factor=237.