

Versatile Synthesis of Nanometer Sized Hollow Silica Spheres

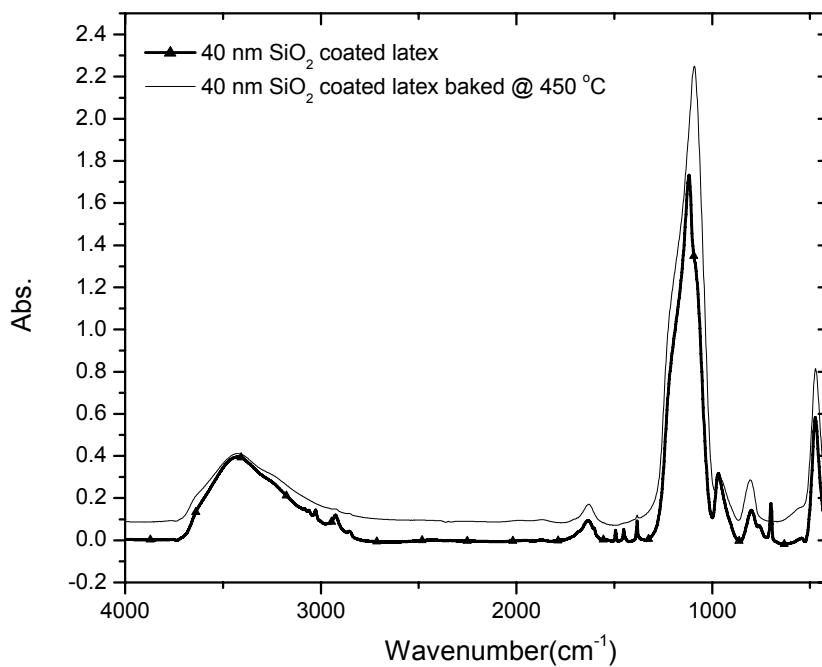
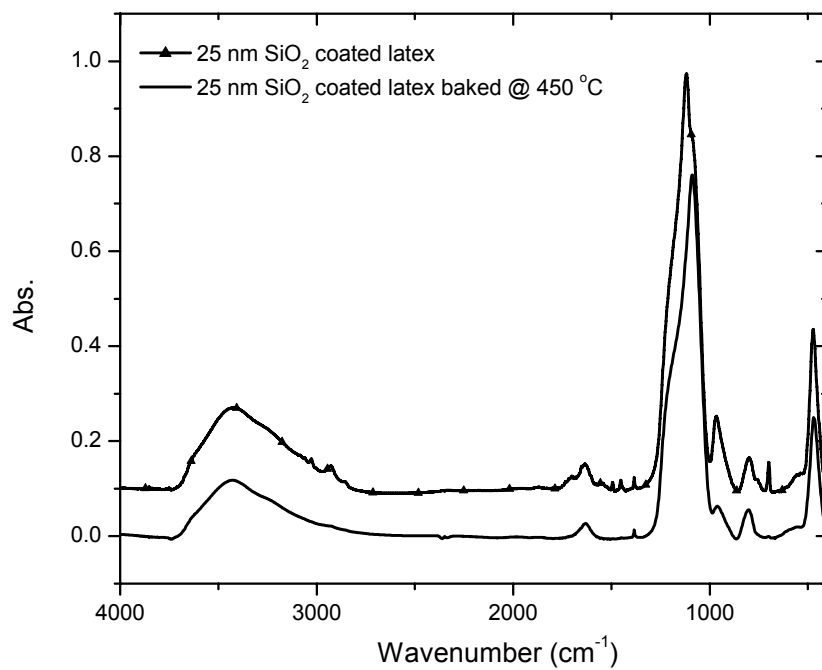
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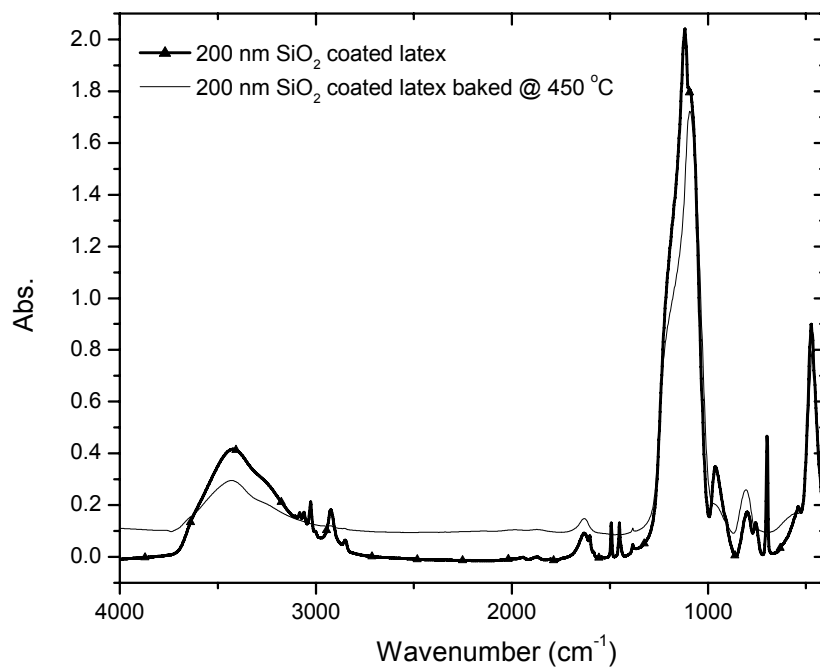
IBM Almaden Research Center, 650 Harry Road, San Jose, CA 95120, United States. Fax: 1 408 927 3310; Tel: 1 408 927 1646; E-mail: rdmiller@almaden.ibm.com

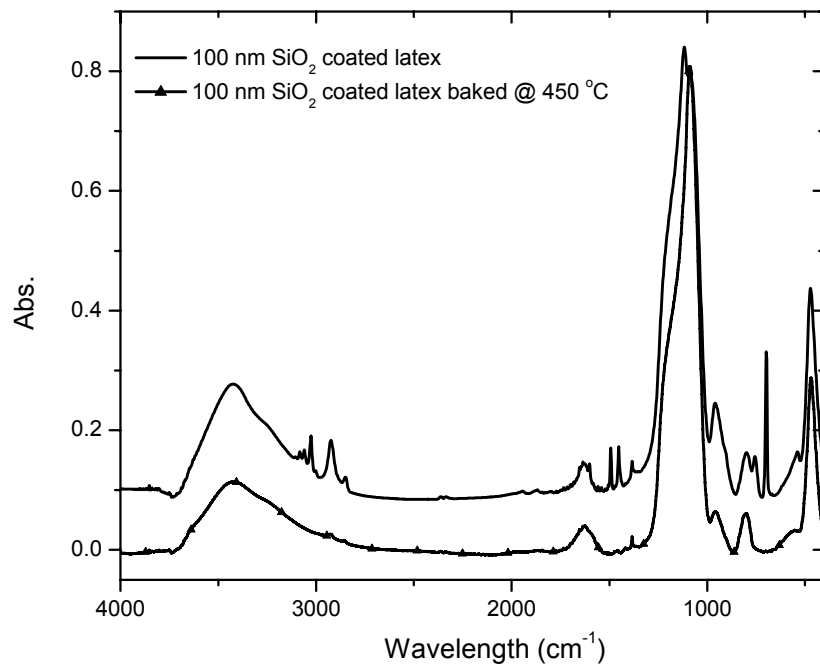
Supplementary Information

To synthesize the silica coated particles, the latex dispersions (2.4. to 2.6 w%) were injected into a 0.58 w% sodium silicate solution at pH = 9.7. The final concentrations ranged from 0.1 to 0.5 w% and the reaction mixtures were stirred for 24 hrs. To prevent precipitation due to the drop in pH, the dispersions were diluted to twice the volume of the reaction mixture after which they were dialysed against water to remove the excess salts. In a typical experiment, the silica coated particles were transferred to CH₃CN by diluting 25 mL of a 0.1 w% aqueous dispersion with CH₃CN (250 mL, after which all solvents were removed on a rotary evaporator until 15 – 25 mL remained. To this dispersion CH₃CN (100 mL) was added and the volume was again reduced to 15 mL, this procedure was repeated twice. To the resulting water free dispersion, 1 mL 1,1,1,3,3,3-hexamethyldisilazane was added and the reaction mixture was stirred for 16 hrs. To this 35 mL MeOH was added and the precipitate was collected by centrifugation, washed 3 times by repetitive dispersion/centrifugation cycles in MeOH and air-dried, resulting in 22 mg of a white product. For TEM carbon coated formvar grids (Ted Pella Inc.) were used and for SEM silicon wafers.

IR spectra before and after calcination of coated polystyrene lattices with different diameters, measured in KBr pellets.







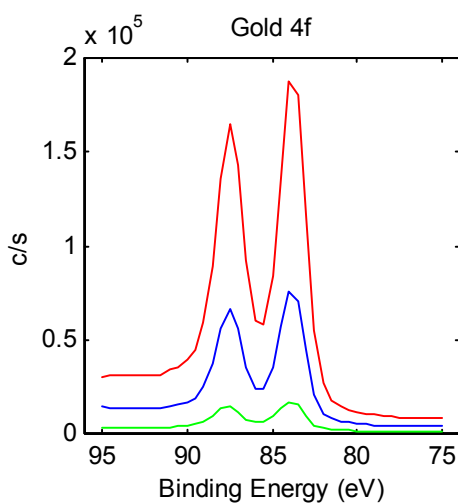
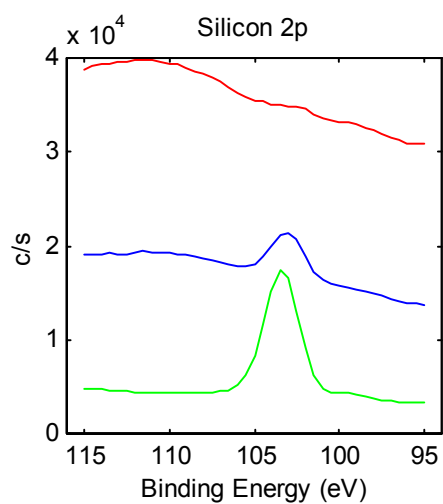
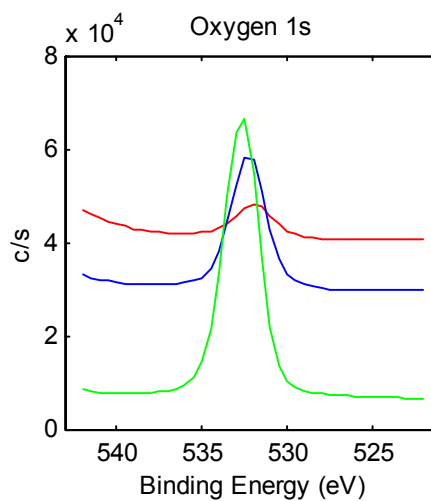
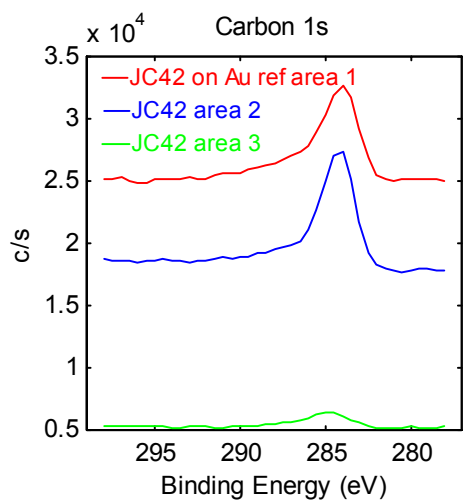
XPS measurements were carried out on drop-cast samples placed on gold-sputtered silicon wafers, the thickness of the gold layer was 350 nm.

Atom concentration table:

	C1s	O1s	Si2p	Au4f	O/Si	C/Si
Au ref	41.3	13.9	0.0	44.8	na	na
25 nm	34.7	41.0	10.6	13.7	3.8	3.3
25 nm calcined	4.0	67.0	26.7	2.4	2.5	0.1
AU ref	41.6	13.1	0.0	45.3	na	na
40 nm	36.3	47.3	14.2	2.1	3.3	2.5
40 nm calcined	9.5	66.0	24.5	0.0	2.7	0.4
Au ref	43.1	15.1	0.0	41.7	na	na
100 nm	12.9	62.6	23.2	1.3	2.7	0.6
100 nm calcined	4.7	67.7	27.4	0.2	2.5	0.2
Au ref	38.2	14.1	0.0	47.8	na	na
200 nm	7.2	65.7	26.5	0.6	2.5	0.3
200 nm calcined	26.7	50.8	17.6	4.8	2.9	1.5

High resolution XPS of the 25 nm sample; red = reference, blue = coated; green = calcined

Jeroen45.spe: C, Si content on Au IBM Almaden
2002 Jul 11 Al mono 25.2 W 200.0 μ 45.0° 3.9831e+004 max 1.78 min
Si2p/Area3: JC42 area 3/1 (SG5)



High resolution XPS of the 40 nm sample; red = reference, green = coated; blue= calcined

Jeroen48.spe: C, Si content on Au

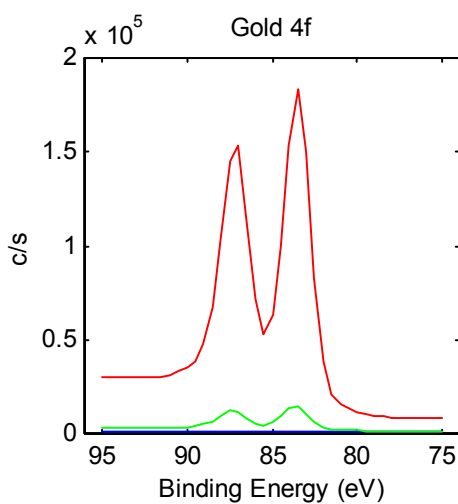
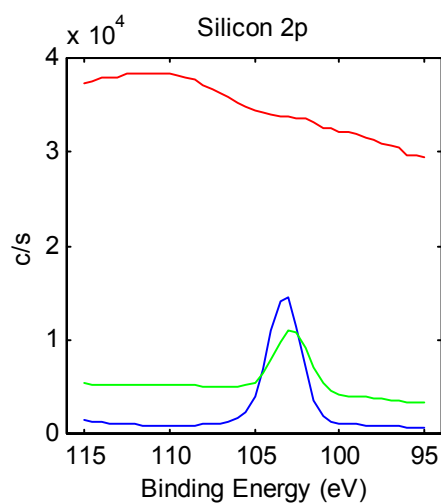
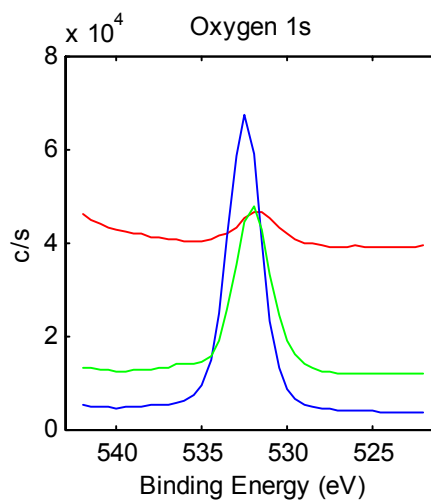
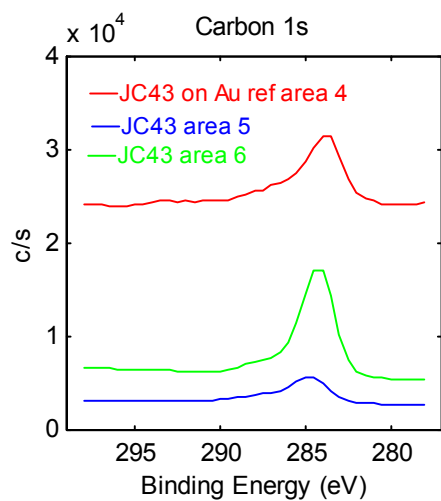
IBM Almaden

2002 Jul 11 Al mono 25.2 W 200.0 μ 45.0°

3.8416e+004 max

1.78 min

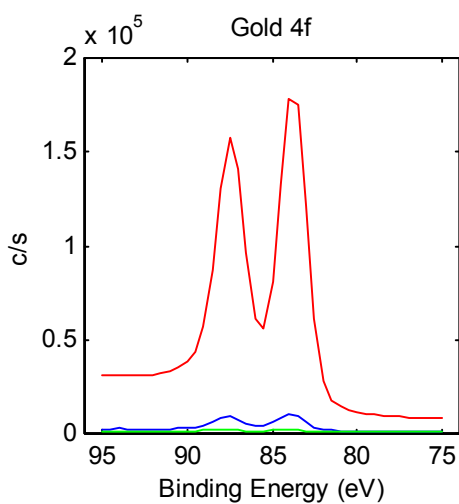
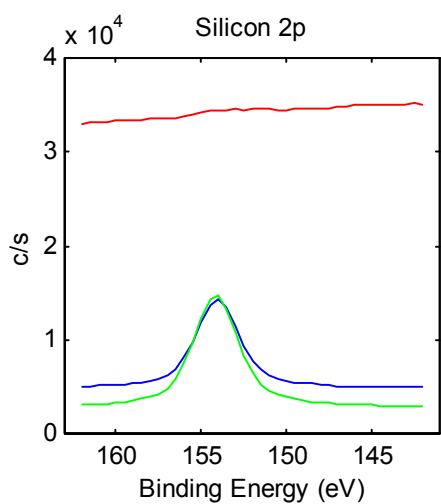
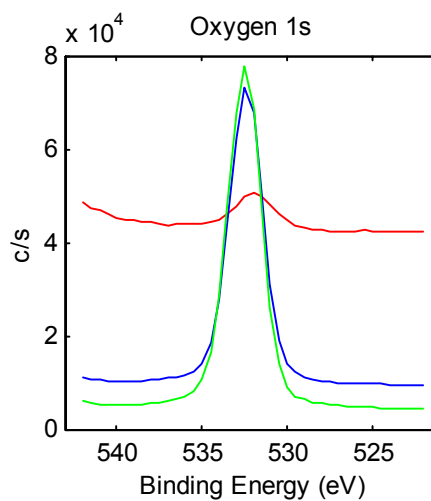
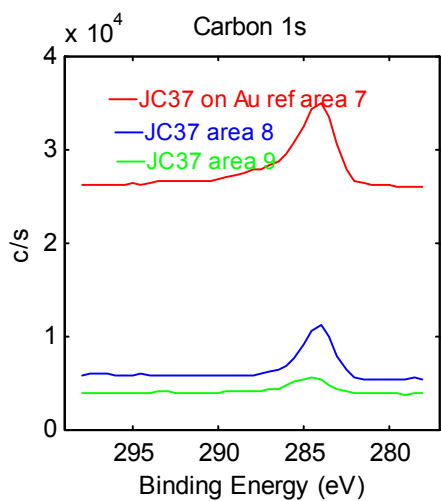
Si2p/Area6: JC43 area 6/1



High resolution XPS of the 100 nm sample; red = reference, blue = coated; green = calcined

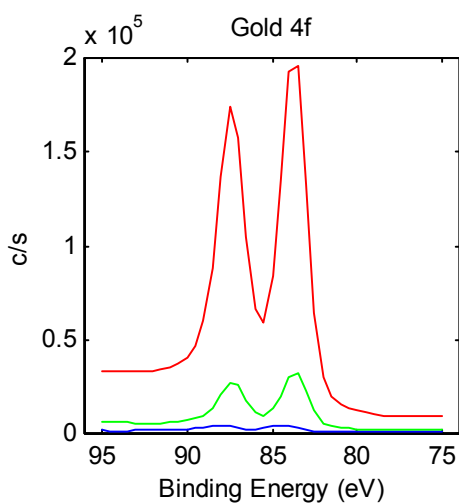
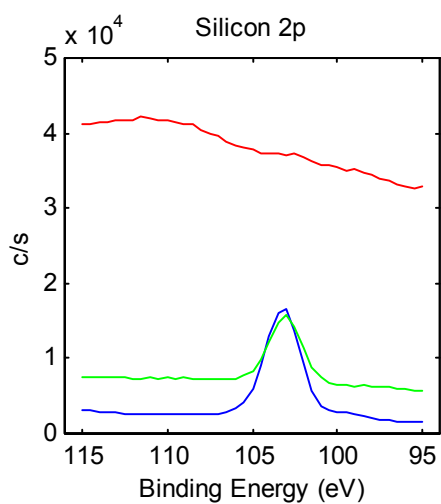
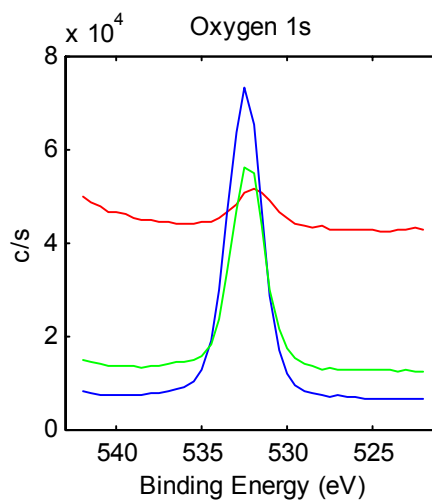
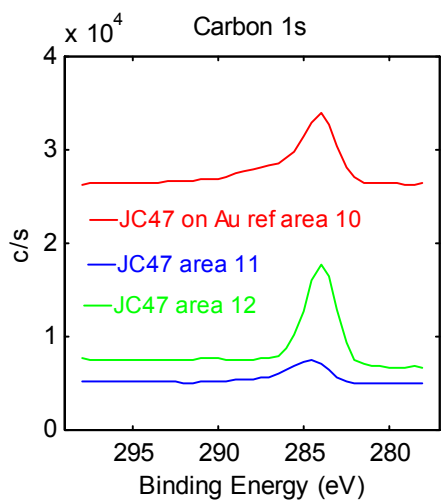
Jeroen51.spe: C, Si content on Au
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C1s/Area9: JC37 area 9/1 (SG5)

IBM Almaden
3.4940e+004 max
53.29 s

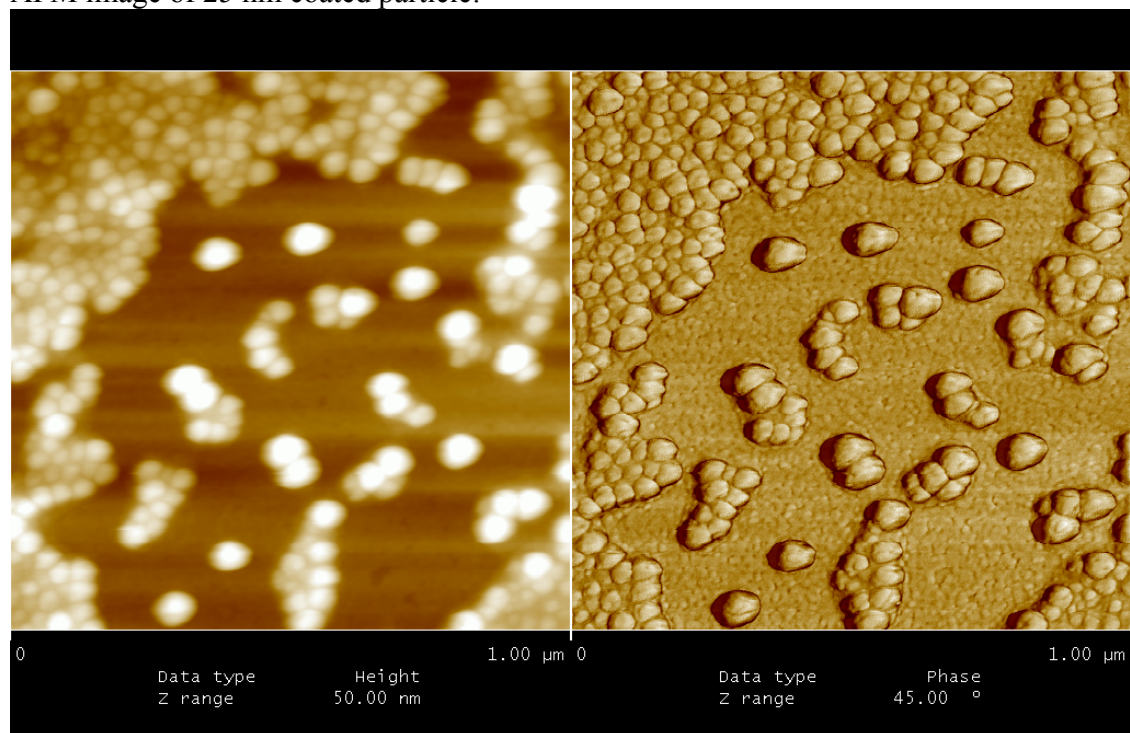


High resolution XPS of the 200 nm sample; red = reference, green = coated; blue = calcined

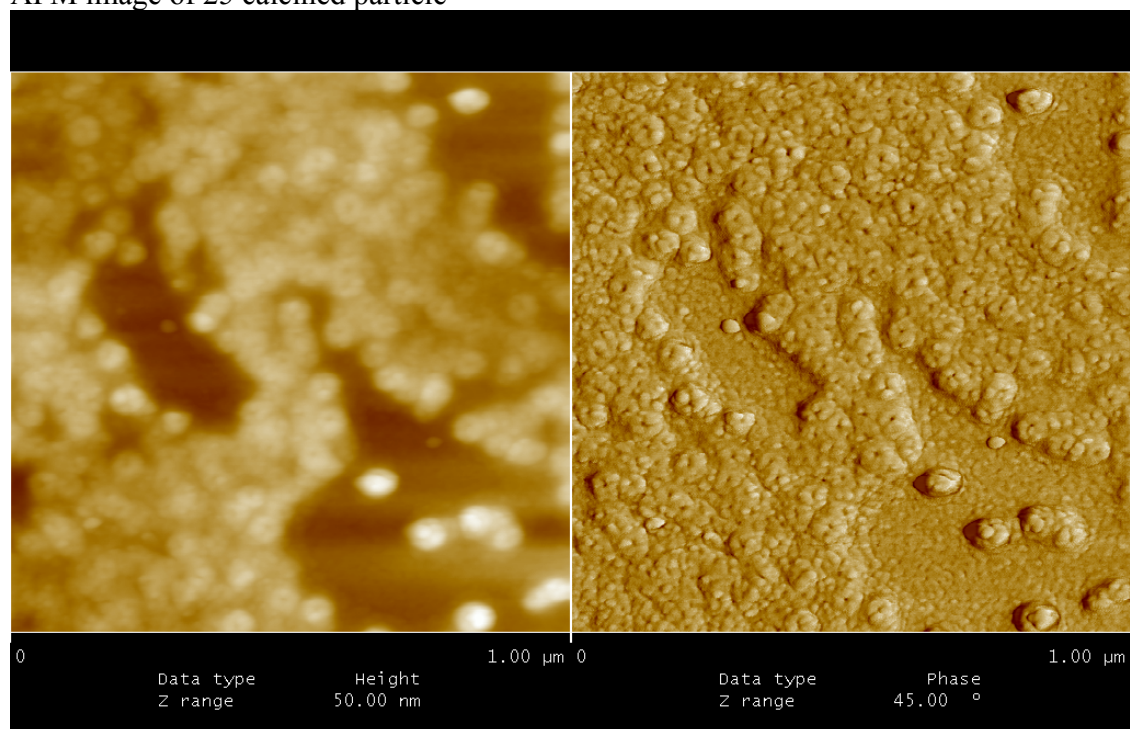
Jeroen54.spe: C, Si content on Au	IBM Almaden
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C1s/Area12: JC47 area 12/1 (SG5)	



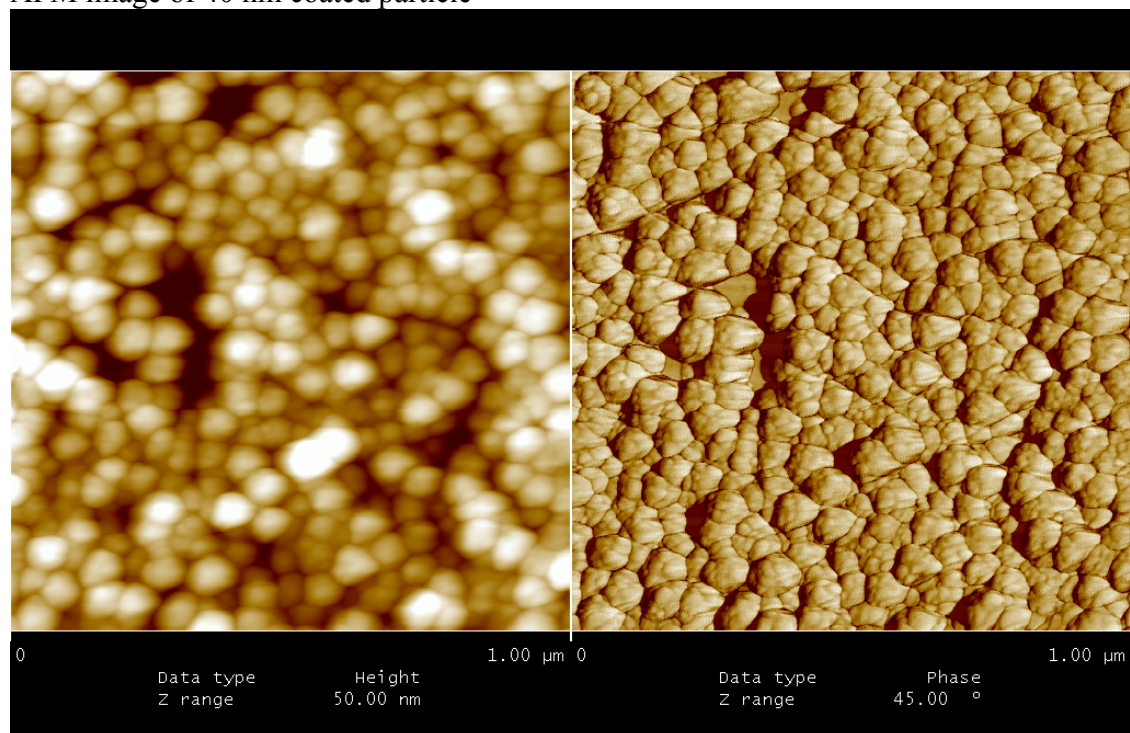
AFM image of 25 nm coated particle:



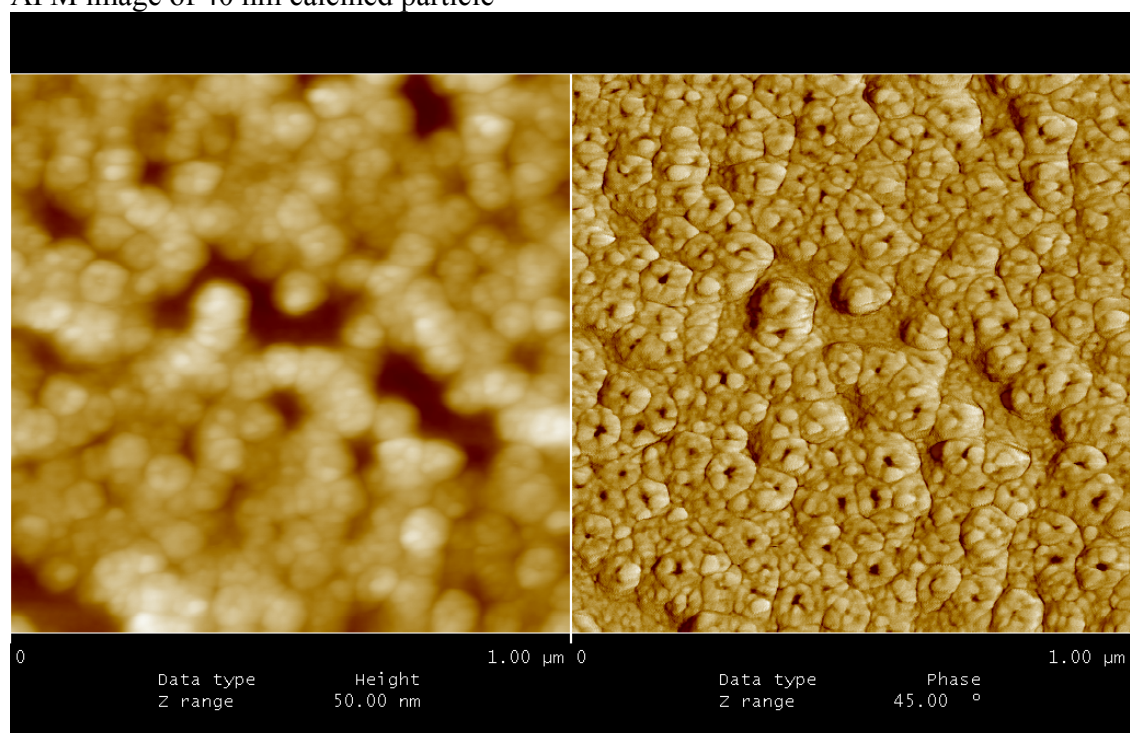
AFM image of 25 calcined particle



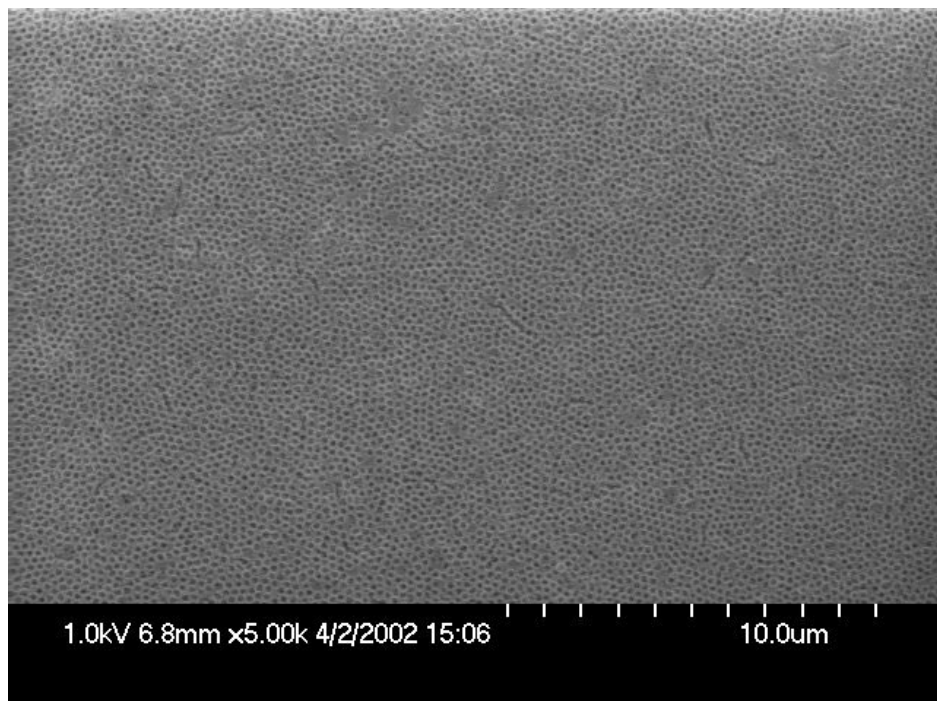
AFM image of 40 nm coated particle



AFM image of 40 nm calcined particle



SEM image displaying the long range order in the slowly formed film of 200 nm coated spheres on a silicon wafer, after calcination.



TEM image of SiO₂ coated polynorbornene-polyethyleneglycol star-shaped polymer. The detailed synthesis of the particles themselves will be published elsewhere (E.F. Connor *et al.* in preparation). For this particular sample a molecular weight of $M_n = 37$ kD was found, with a PDI = 1.4.

