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SiO_2 hollow spheres prepared by plasma deposition on polystyrene spheres and subsequent calcination

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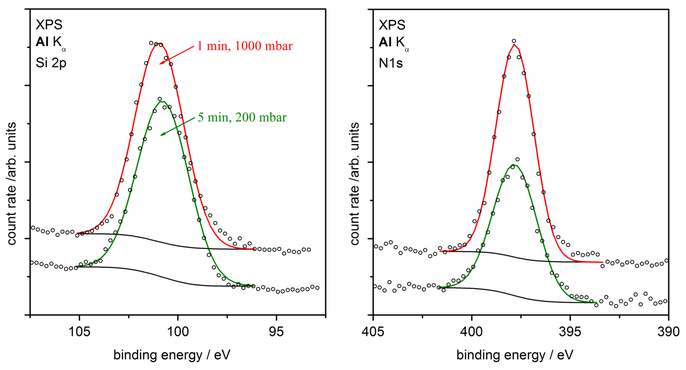
Introduction

Multilayer polystyrene (PS) colloid crystals are exposed to a dielectric barrier discharge (DBD) in a mixture of nitrogen and silane as processing gas. This yields a closed layer of silicon nitride on the PS spheres. In a second step silicon nitride is oxidised to SiO_2 in an oxygen-DBD. Finally PS is pyrolytic decomposed leaving a colloid crystal of hollow SiO_2 spheres. The SiH_4-N_2-plasma treatment is applied at different gas pressures, especially atmospheric pressure and different times, respectively. So the thickness of the silicon nitride layer can be modulated. For characterization and validation of each step mainly X-ray photoelectron spectroscopy (XPS), atomic force microscopy (AFM) and confocal laser scanning microscopy (CLSM) are used.

Silane-Nitrogen-Plasma Treatment



Preparation of PS crystal
fcc/sc PS beads (Ø600 nm)
on copper template.

Plasma treatment
5 min, 200 mbar
1 min 1000mbar
process gas 1.5% SiH_4 in N_2

Stoichiometry @ 200 mbar

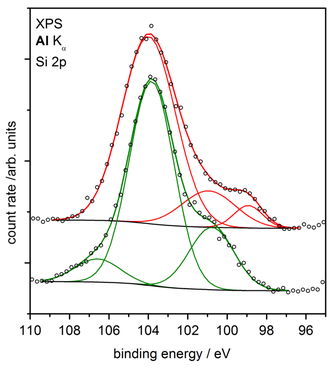
Si 2p (SiN_x)	77 %
N 1s (SiN_x)	23%

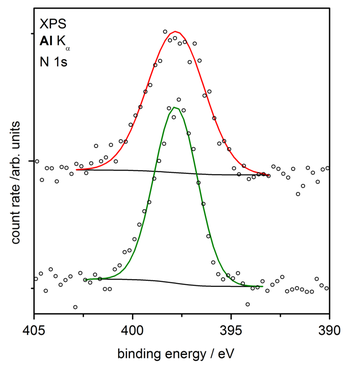
Stoichiometry @ 1000 mbar

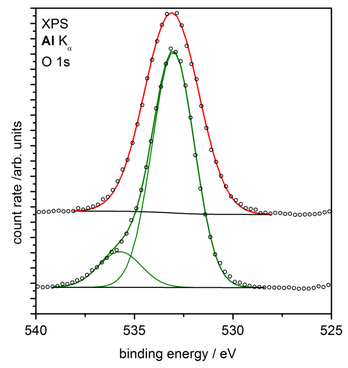
Si 2p (SiN_x)	73 %
N 1s (SiN_x)	27%

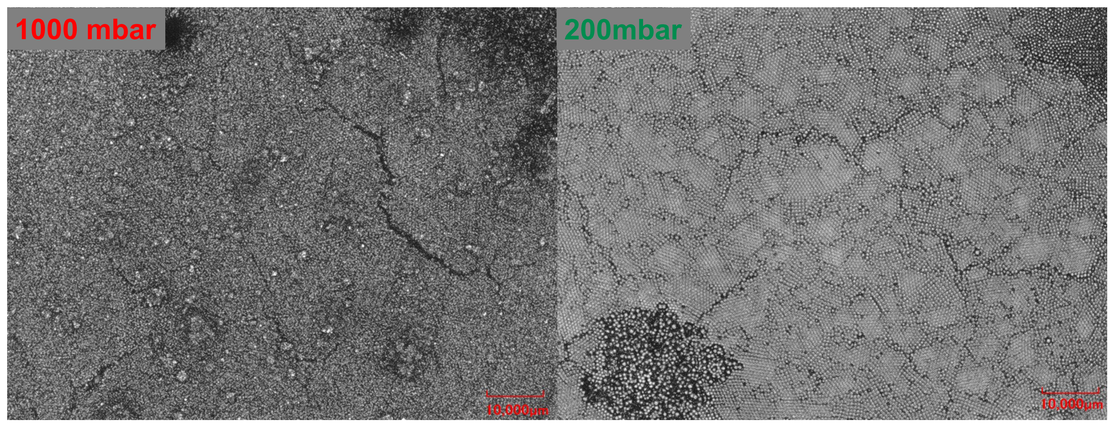
DBD treatment at atmospheric pressure forms silicon nitride particles on the surface. Furthermore DBD induced disruptive breakdowns crack the surface .

Oxygen-Plasma Treatment









Plasma treatment
5 min, 200 mbar
1 min 1000mbar
process gas O_2

Stoichiometry @ 200 mbar

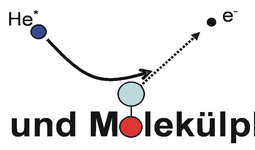
O 1s (SiO_2)	52%
O 1s (SiO_2)	8%
Si 2p (SiO_2)	26%
Si 2p (SiO_2)	3%
Si 2p (SiN_x)	8%
N 1s (SiN_x)	4%

Roughness
S_q = 2.0 nm
(PS-beads S_q = 0.24 nm)

Stoichiometry @ 1000 mbar

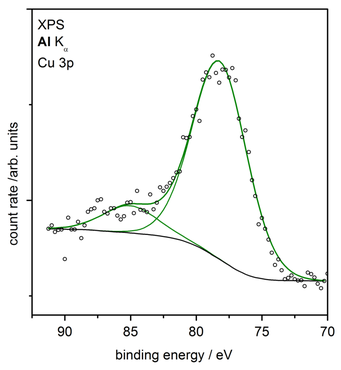
O 1s (SiO_2)	57%
Si 2p (SiO_2)	29%
Si 2p (SiN_x)	5 %
N 1s (SiN_x)	4%
Si 2p (Si)	2%
C 1s (adv. C)	3%

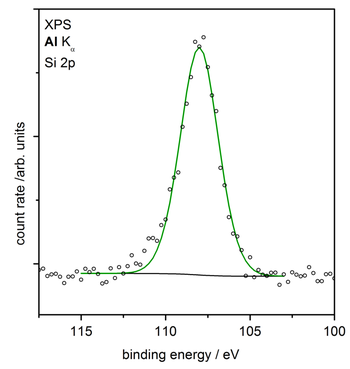
Silicon nitride oxidises to silicon dioxide. XPS measurement suggests also atomic silicon in the case of atmospheric pressure treatment.

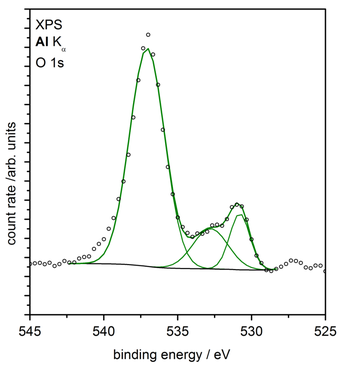


Atom- und Molekülphysik an Oberflächen

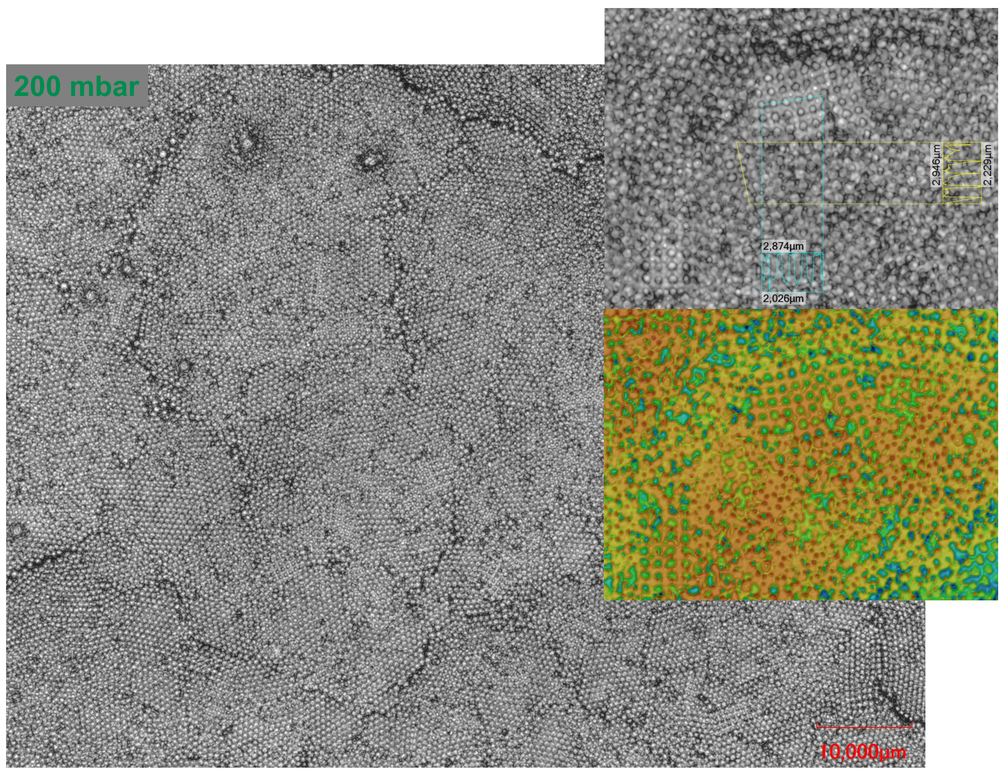
Pyrolysis of Polystyrene

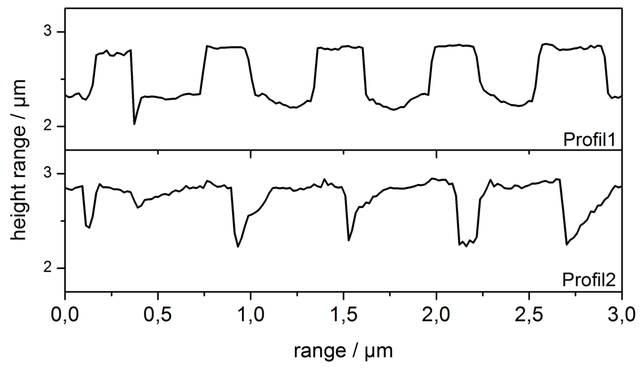


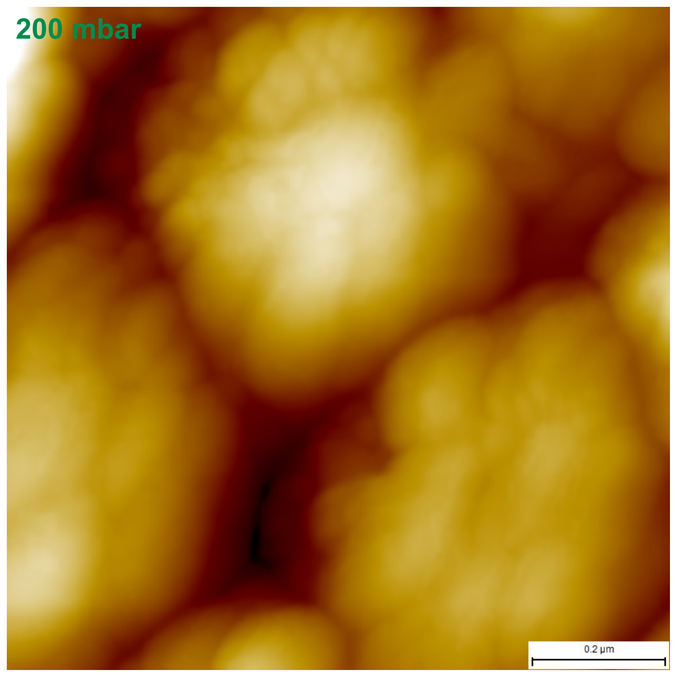




Due to local charge-up effects the O1s- and Si2p-peak shift partially.







Stoichiometry @ 200 mbar

O 1s (SiO_2)	43%
O 1s (CuO)	6%
O 1s (adv. C)	8%
Si 2p (SiO_2)	24%
Si 2p (SiN_x)	<1%
N 1s (SiN_x)	<1%
Cu 3p(CuO)	9%
C 1s (adv. C)	8%

Roughness
S_q = 1.5 nm

Pyrolysis
Temperature ramp 1.5 °C/min
Period of const. temperature
4.5 h @ 550 °C

Conclusion and Preview

"Hollow" SiO_2 spheres are prepared in a two-step DBD-deposition process with subsequent decomposition of PS. Preparation at 200 mbar comes to a well-shaped surface whereas 1000 mbar preparation leads to a rough and inadequately adjustable surface. For better characterisation of hollow spheres a profile made by FIB can be imaged by FESEM.

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