Roughness evolution of sol-gel optical coatings by ion beam sputtering

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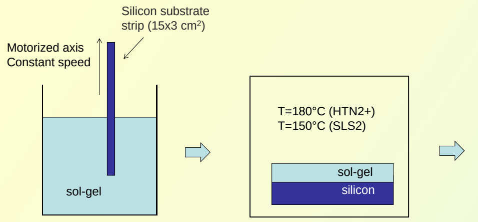
1. Abstract

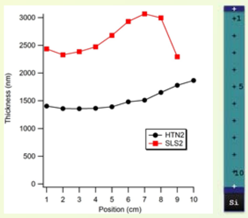
The surface roughness evolution of two silica-based sol-gel materials under 650 eV argon ion beam sputtering has been investigated. The liquid sol-gel solutions had been applied on silicon substrates using the dip coating technique and then thermally cured to obtain solid thin films. Their thickness had then been controlled over the samples surface using spectroscopic ellipsometry. The surface roughness of the sol-gel films has been measured using both interferometric profilometry and atomic force microscopy at different sputtering depths. Roughness increases significantly faster with sputtering depth in sol-gel layers than on bulk fused silica. Interestingly, the sputtering rates of the sol-gel layers are also observed to be much higher that of bulk fused silica. The development of micron scale holes with relatively stable interstices rules the surface roughness evolution. AFM measurements revealed a regular submicron scale lateral structure which nanometric amplitude is amplified under sputtering.

2. Sol-gel preparation



Dip coating

Curing in oven



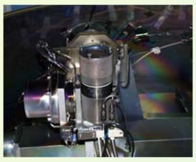
Thickness profile from ellipsometric measurements

2 Nanoxid® sol-gel tested → **HTN2+**, **SLS2**

<u>Measured sol-gel film thickness on dip coated Si samples length</u>
HTN2 :1.4 to 1.9 µm
SLS2: 2.0 to 3.0 µm

Samples for sputtering tests are taken (~3x3 cm²) from an homogeneous (thickness) part of the dip-coated strip

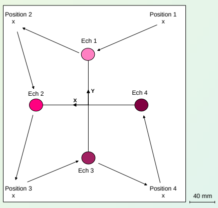
3. Ion beam sputtering



Ion Beam Figuring facility : *the ion source is mounted on a four-axis motorized system ; the sample-holder is installed on a fifth motorized axis .*

Experimental conditions

Ion source: Kaufman type, hollow cathode (HC), 3cm grid
Ion source – sample distance : 150 mm
Ion beam energy: 650 eV
Ion beam current: 60 mA
Ion beam current density on sample : 1.0 mA/cm2
Hollow cathode neutralizer (HCN): 120 mA
Gas flow: Ar 3.5 sccm (HC) and 4 sccm (HCN)
Working pressure: 1.7-1.9 10^{-4} mbar



Up to four samples are sputtered with different time during the same run.

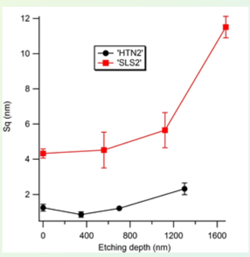
4. Experimental Results

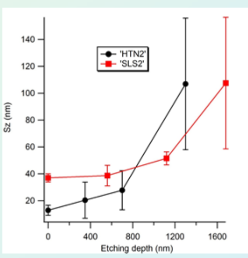
4.1. Ion beam etching rate

	HTN2+	SLS2	SiO2 (bulk)
Etching rate (µm/min) @ 650 eV Ar+, 1 mA/cm²	0.15	0.23	0.05

Measured by interferometric profilometry on the step created by masked areas on the sputtered samples

4.2. Roughness statistics (Sq, Sz) evolution of sol gel with sputtered depth



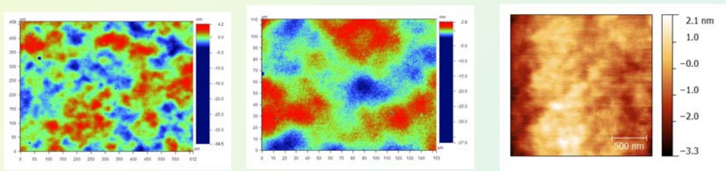


Roughness statistics averaged from 5 images measured by interferometric profilometry (Phase-Shifting interferometry, X40 magnification). Sq: rms roughness, Sz: peak-to-valley roughness

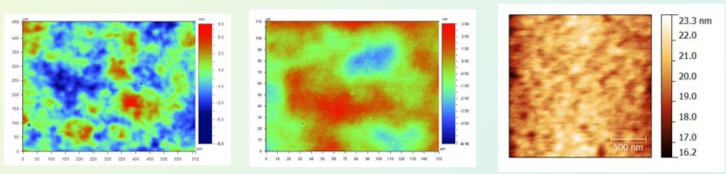
4.2. Topography evolution of sol gel : **HTN2+**

Measured by interferometricl profilometry
X10 (left) and X40 (right)

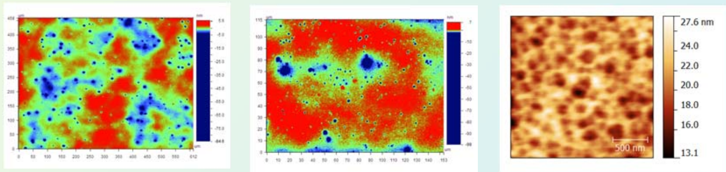
Measured by AFM
(2 x 2 µm² area)



Initial



After 0.7 µm sputtered



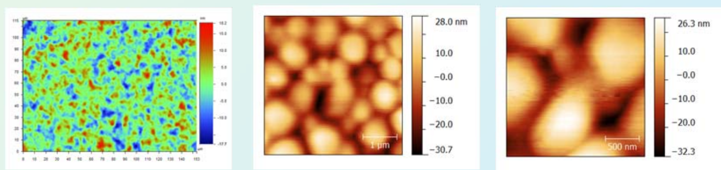
After 1.3 µm sputtered

4.3. Topography evolution of sol gel : **SLS2**

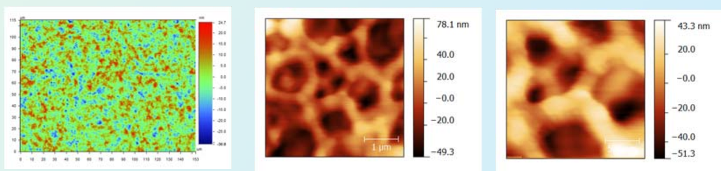
Measured by interferometric profilometry (X40)

Measured by AFM
(4 x 4 µm² area)

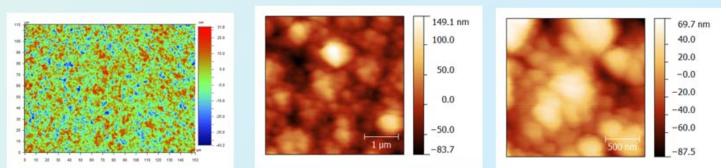
Measured by AFM
(2 x 2 µm² area)



Initial



After 1.1 µm sputtered



After 1.7 µm sputtered

5. Conclusion

- The roughness of both tested sol-gel films increased with etching depth.
- However the HTN2 initial roughness and roughness evolution with sputtering is lower (smaller grains) ; Sq stays < 2 nm for sputtering depths under 1 µm. But near the interface with the Si substrate, a lot of micrometric holes (inhomogeneities in sol-gel ?) are visible on interferometric micrographs.
- The much higher etching rate of sol-gel coating than bulk fused silica is also an interesting feature.

6. Perspective

- Improvement of HTN2+ sol-gel coating procedure to avoid micrometric defects.

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