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Reflectivity, imaging ellipsometry and scattering analysis of gold nanoparticles growing in a polymer matrix

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Introduction and research context

Research context and motivation :

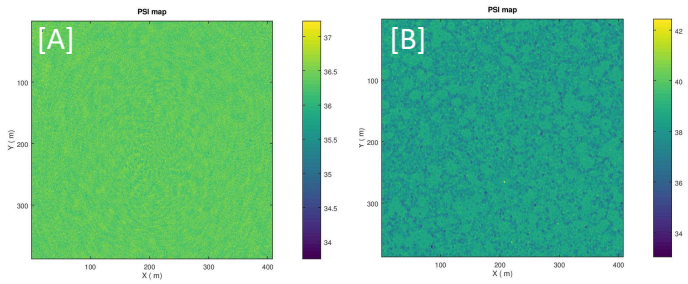
- Gold NPs embedded in PVA used as SATURABLE ABSORBERS in passive Q-switch systems : importance of roughness
- Optical properties of metal nanoparticles (NPs) determined by a collective oscillation of the conduction electrons : localized surface plasmon resonance (LSPR)
- Model system : Au^0 NPs in a poly(vinyl alcohol) (PVA) matrix and chemical reduction of the metal salt by thermal annealing of the film (λ_{spr} = 530nm)
- *In situ* route for synthesis : spontaneous encapsulation in a matrix during synthesis but less control on the shape/size of the NPs

Optical characterization :

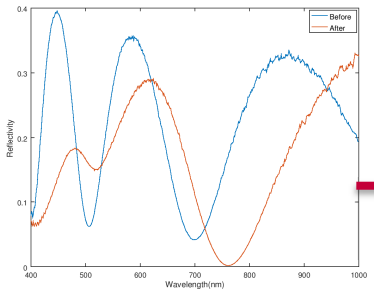
- Imaging ellipsometry (IE) measurements were carried out using an EP3 single wavelength ellipsometer operating at $\lambda = 658\ nm$ (Accurion GmbH, Goetingen, Germany). Ellipsometric angles (Δ and Ψ) images were recorded at an angle of incidence (AOI) $\theta_i = 42°$
- Spectroscopy reflectivity (SR) was studied by home-build spectroscopic reflectometer with CSS200 Thorlabs spectrometer (integration time : 12ms) and temperature control
- Optical scattering was analyzed by the bi-directional reflection distribution function (BRDF) with a EZContrast-MS (ELDIM) in collimated mode ($\lambda = 570 \pm 10\ nm$) with an angle of incidence of $\theta_i = -20°$

Experimental results

Monitoring of the gold NPs growth : LOCAL (IE) versus GLOBAL (SR) changes of optical properties

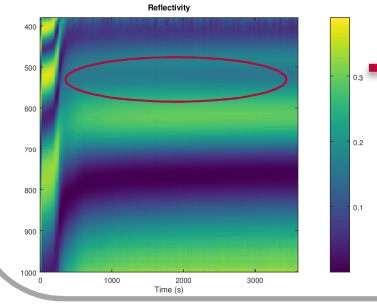


Imaging ellipsometry (IE) maps of Au-PVA (low doping level) film [A] before annealing and [B] after 60 min annealing at 140°C : gold NPs induce optical diffraction – Onset of depletion zones



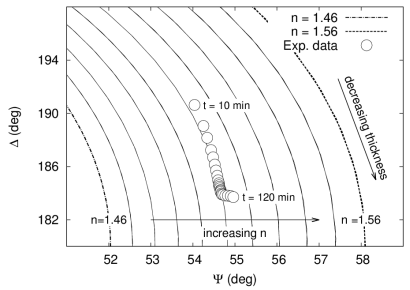
Spectroscopic reflectivity (SR) of [blue] Au-PVA film (high doping level) before annealing and [orange] Au-PVA film after annealing

Decreasing thickness of the film & decreasing reflectivity around 520 nm



Spectroscopic reflectivity (SR) of Au-PVA film (high doping level) during the annealing (changes in the first part of the experiment correspond to the glass transition of the PVA)

Constant angle of incidence curves at 658 nm and 42° incidence parametrized by film thickness (low doping level)

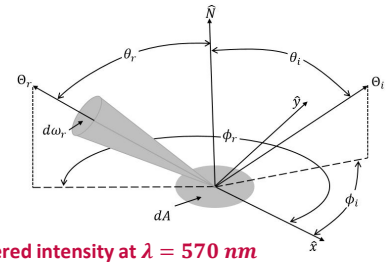


Left most curve : n = 1.46
Right most curve : n = 1.56
Open circles : Gold-PVA optical response

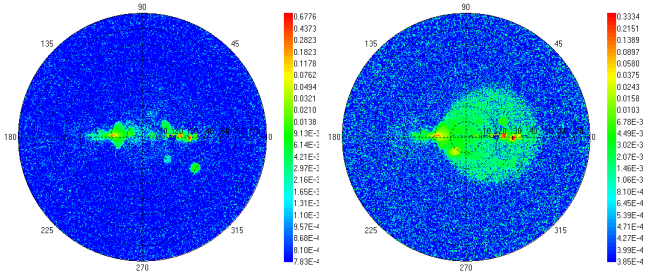
Decreasing thickness of the film & increasing of the refractive index

Bidirectional Reflectance Distribution Function (BRDF)

The BRDF of a surface is the ratio of reflected radiance to incident irradiance at a particular wavelength for all scattering angles



BRDF - Scattered intensity at $\lambda = 570\ nm$



Left : BEFORE annealing - Right : AFTER annealing (90 min, 135°C)

- Illumination from right to left
- Scattering strongly induced by NPs growth
- Important backscattering component

Conclusions and acknowledgements

- Variation of the film thickness and the refractive index during the annealing → growth of the Au-NPs
- Energy absorption around 520 nm in the SR map (LSPR) → encapsulated Au-NPs in the polymer matrix
- Light (back)scattered by the annealed sample due to the growth of the gold nanoparticles

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