

Porous Silicon Composite Material

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Application Note

Porous silicon materials are under intense investigation for optoelectronics applications. The development of these materials is of commercial interest as they are compatible with IC fabrication, and their ability to be integrated into microelectronic silicon based technologies will allow production of low cost optoelectronics components and systems. One such application has been investigated through the study of a composite material composed of an oxidised porous silicon film filled with DR1 dye. Several organic molecules with conjugated chain structure (such as Disperse Red One, DR1, molecules used here) are known to possess nonlinear optical properties, which can be used to manufacture optical devices such as modulators or switches. The ordered mesoporous silica matrix makes it an ideal host for the introduction of these small organic molecules, and an efficient way towards manufacturing non-linear optical devices.

In this study Spectroscopic Ellipsometry (SE) was used to characterise material properties during the various manufacturing stages for producing the composite material. The optical properties and the anisotropy of the composite material were determined from the SE data analysis with the aim of characterising its nonlinear behaviour.

Sample Preparation

Porous silicon is typically prepared by anodic electrochemical etching. The silicon wafer and a platinum electrode are immersed in an electrolyte which is commonly a mixture of water, ethanol and HF. On application of an appropriate current (around $100 \text{ mA}\cdot\text{cm}^{-2}$), a $5 \mu\text{m}$ porous silicon layer is formed at the surface of the wafer. In this example the porous silicon layer is fully oxidised by a wet oxidation process. Following this Disperse Red One (DR1) molecules diluted in a THF solution are introduced into the porous material. A uniform distribution of DR1 molecules inside the porous silica layer is obtained.

Composite Porous Silicon Material Characterisation

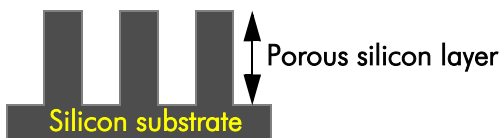
Ellipsometric measurements were made using a UVISEL NIR Spectroscopic Phase Modulated Ellipsometer. Ellipsometric data were acquired at an incident angle of 70° across the range $0.75 - 4.5 \text{ eV}$ with a step of 0.005 eV .

A wide spectral range is necessary to characterise of the composite structure owing to its strong absorption at UV and visible wavelengths. The optical properties and thickness of the structure were extracted from the SE data analysis, with the optical constants of the different films determined using Lorentz oscillator dispersion formulae.



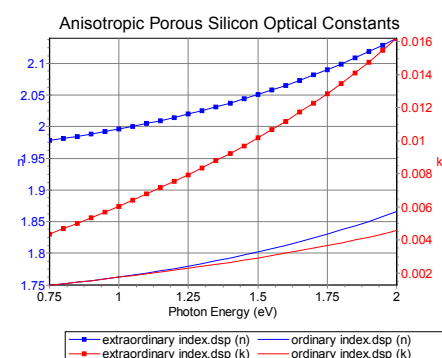
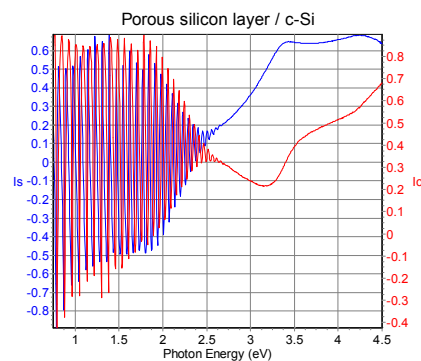


Characterisation of a 5 μm porous silicon layer

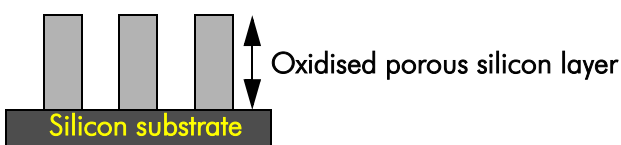


STEP1: Before oxidation

The spectroscopic measurements exhibit two different parts. Between 0.75-2.5 eV the porous silicon layer is transparent and the thick film introduces a large number of interference fringes, whilst at higher energies the film becomes completely absorbing. The optical properties of the layer were extracted over the reduced transparent range. The columnar structure of the layer introduces uniaxial optical properties, with the optical axis perpendicular to the surface. One can observe that the birefringence is quite strong with $(n_o - n_e) > 0.2$ in the 0.75 – 2 eV range due to the high level of doping in the silicon.

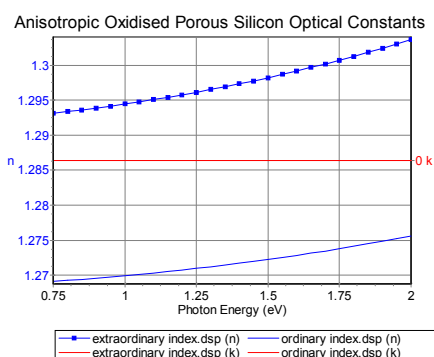
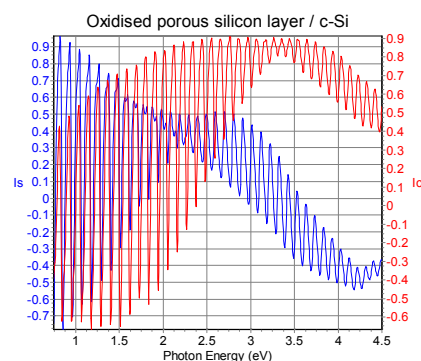


Characterisation of the oxidised porous silicon layer



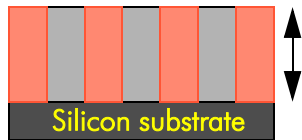
STEP2: After oxidation

The second step consists of the oxidation of the porous silicon layer in order to obtain a porous SiO_2 layer. The experimental data exhibit oscillations over the whole spectral range owing to the transparency of the layer. The measured optical properties show that the absorption is effectively negligible in the spectral range defined by step 1 and used for fitting the data. The found refractive index is lower than before and the birefringence is quite small with $(n_o - n_e) \sim 0.02 - 0.03$.





Characterisation of the oxidised porous silicon layer filled with DR1 dye



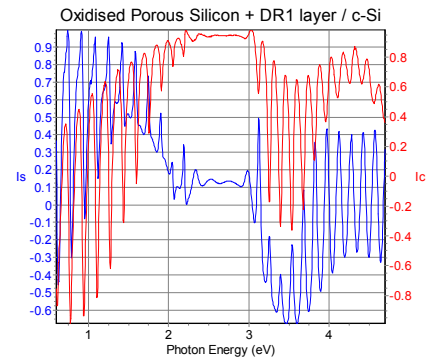
Oxidised porous silicon layer

roughness	803 Å
Thick anisotropic layer	55927 Å
SiO ₂	1180 Å
c-Si substrate	

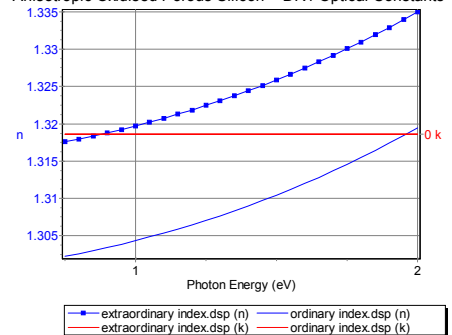
STEP3: DR1 filling

The absence of interference fringes between 2.25 and 3 eV is due to the strong absorption of the DR1 dye in this range.

A three layer model accurately describes the oxidised porous silicon sample filled with DR1. It takes into account a dense SiO₂ interface and also surface roughness described by a mixture of 50% void and 50% film material calculated using the Effective Medium Approximation. The oxidised porous silicon layer also exhibits weak uniaxial anisotropy with an optical axis perpendicular to the surface.



Anisotropic Oxidised Porous Silicon + DR1 Optical Constants



Conclusion

Complete characterisation of the composite material structure was successfully carried out using the UVISSEL Spectroscopic Phase Modulated Ellipsometer. Accurate and simultaneous determination of thickness, optical properties and anisotropy of the composite porous silica layer has been performed in the NIR/VIS range in order to investigate the nonlinear properties of the material.

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