Thin Films and Coatings

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Characterization of amorphous and porous Silicon coatings by (S)TEM and EELS

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We present a new way to produce porous amorphous silicon coatings by glancing angle magnetron sputtering of Si targets in noble gas atmospheres. A similar process was previously applied to produce SiO_xN_y-coatings [1]. This allows the production of a closed porosity in contrast to the previously most common way of chemical etching in HF-based solution, which leads to open porosity. Our way is environment friendly and up scalable. By varying the deposition parameters such as type of noble gas, the pressure, RF-power as well as applying an additional bias or substrate heating we are able to change the microstructure, which influences the refractive index, of interest for the microelectronic technology and optical applications. The microstructural and chemical investigation of coatings by TEM should lead to a better understanding of the growth mechanism and the influence of the deposition parameters.

Because the pore size lies in the range of ≈ 2 nm to ≈ 50 nm TEM is the appropriate way to characterize the pore size distribution. But TEM also suffers from the overlap of pores in projection so we apply tomography in addition to reveal the porosity in three dimensions. This helps to identify the closed pores and the complicated surface structure of the sample due to TEM sample preparation by ion milling. In addition we perform both, STEM-EELS and EFTEM experiments.

In Figure 1 shows STEM HAADF images of coatings deposited under (a) He- and (b) Ar-atmosphere. The coating is dense for the Ar- and porous for He-atmosphere. Pores align in columns towards the magnetron source. The corresponding EEL-spectra in (d) reveal only the Si plasmon peak for the dense coating while that of the porous coating in (c) contains two additional features, the He K-edge at ≈ 22 eV and the shoulder at ≈ 10 eV attributed to the pore-matrix interface plasmon [2]. Figure 2 shows an EFTEM spectrum image of another coating deposited under He-atmosphere with more separated. The spectrum extracted from position 1 at the wall separating two pores reveals the same shoulder and the image slice at 9 ± 2 eV is shown in (a) and demonstrates the excitation of the pore-matrix interface plasmon for the closed pores. The zero-loss filtered image is shown in (b) as well as a spectrum extracted from position 2 in between the major pores. This spectrum looks similar to that of the dense layer produced under Ar-atmosphere. Nevertheless the energy resolution and sensitivity of the spectra extracted from the presented EFTEM SI is too low to resolve the He-K-edge so we aim one getting more EELS data for quantification of He inside the pores [3].

1. V. Godinho, C. Rojas and A. Fernandez, Microporous and Mesoporous Materials 149 (2012) 142
2. M.-L. David, F. Pailloux, V. Mauchamp and L. Pizzagalli, Appl. Phys. Lett. 98 (2011) 171903
3. C. A. Walsh, J. Yuan and L. M. Brown, Philosophical Magazine A 80 (2000) 1507

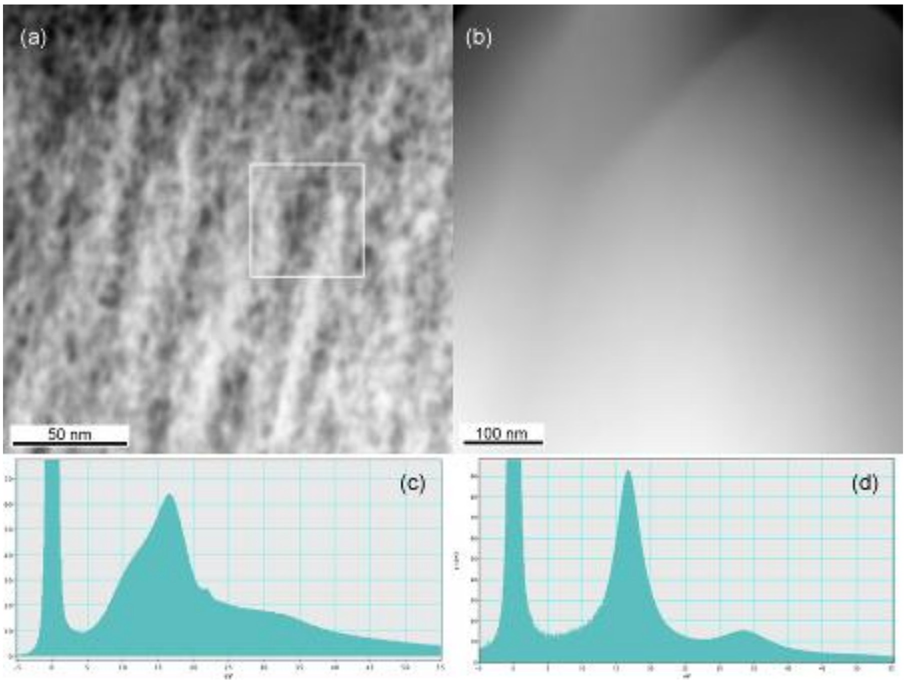

Figure 8. STEM HAADF images of coatings deposited under (a) He- (5·10-2 mbar) and (b) Ar-atmosphere ($7 \cdot 10^{-3}$ mbar). The corresponding EEL-spectra reveal only the Si plasmon peak for the dense layer in (d) while two additional features are present in the spectrum of the porous coating recorded in the area marked with the white square. At ≈ 22 eV the He edge is present and the shoulder at ≈ 10 eV originates from the interface plasmon [2].

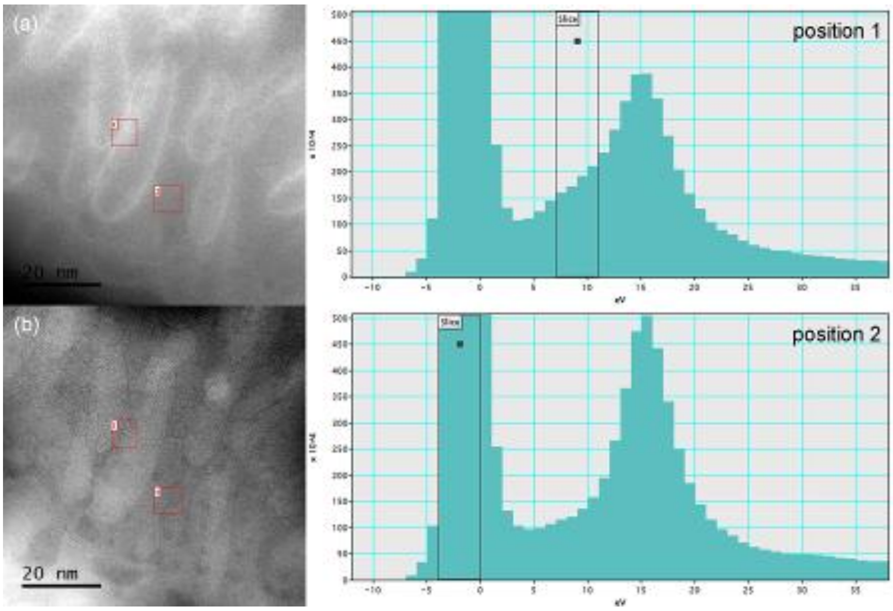

Figure 9. EFTEM spectrum image of a porous Si-coating prepared under He-atmosphere. The image slice at 9±2eV (a) reveals plasmon at the inner surface of the pores. The zero loss filtered image is shown in (b). The spectra extracted from position 1at the pore wall reveals the same shoulder as the spectrum in Figure 1 (c). For comparison the spectrum at position 2 is shown which reveals only the Si-plasmon peak as the spectrum of the dense coating in Figure 1 (d).