Structure Investigation of Porous Silica-Titania Monoliths and Europium(III) Doped-Silica Coated Thin Films via SAXS

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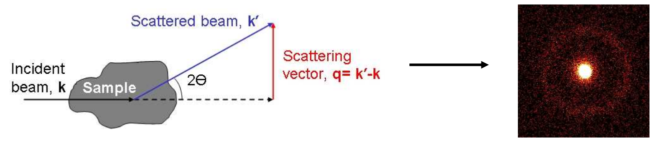
Introduction

Multifunctional materials with high porosity have a great potential in technical applications such as catalysis, chromatography or as sensors. Thin coatings, where lanthanide ions are incorporated into a stable inorganic silica matrix, are promising candidates to improve the thermal and photochemical stability of luminescence materials. These materials find a wide range of applications in our daily life such as in projection televisions and X-ray detectors due to the characteristic luminescent properties of the rare earth elements. Porous titania monoliths are of high interest, e.g. for water purification.

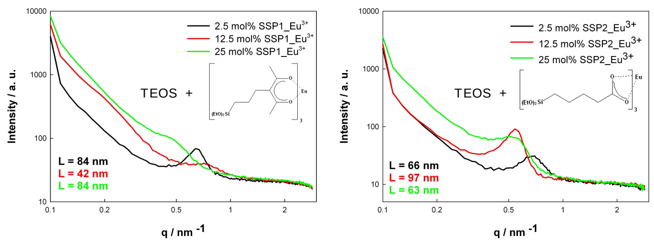
In this work, we report the structure investigation of porous silica-titania oxides as well as the surface texture determination of coated europium(III) doped-silica films. Both materials were prepared via a novel synthesis route using titanium- and europium(III)-complexed organosilanes, respectively. Tetraethyl-orthosilicate (TEOS) and tetrakis(2-hydroxyethyl)orthosilicate (EGMS) are used as additional silica sources. The main characterization methods are Small Angle X-Ray Scattering (SAXS), Grazing Incidence-SAXS (GISAXS) and *in-situ* SAXS.

Eu^{3+} Doped-Silica Coatings

SAXS Measurements:



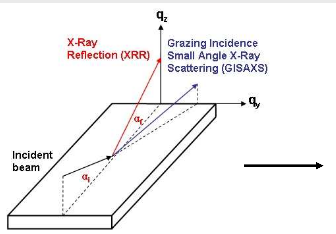
Schematic represention of a scattering experiment. From the scattering pattern one can obtain the size and shape of objects in a range between 1 – 100 nm.



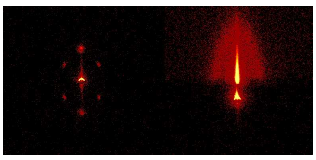
Scattering curves from Eu^{3+} doped-silica coatings. The full width at half maximum (FWHM) of the short range order peaks allow the calculation of the domain sizes L.

Scherrer's formula for small Θ [1,2]: $\Delta(2\theta) = \frac{K \cdot \lambda}{L \cdot \cos\theta} \xrightarrow{q=\frac{4\pi}{\lambda}\sin\theta} L = \frac{2\pi \cdot K}{\Delta q}$

GISAXS Measurements:



Schematic represention of a GISAXS experiment



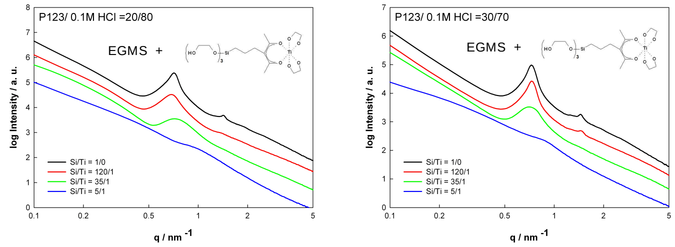
GISAXS Pattern: (left) A pure TEOS coated silica film. It shows perfect hexagonal ordering of the objects (right) TEOS and 25mol% SSP2-Eu^{3+}. The peak broadening is due to the small domain size.

GISAXS gives detailed information on surface texture, shape and orientation of objects on the surface.

Silica-Titania Monoliths

SAXS Measurements:

Porous silica-titania monoliths have been synthesized via sol-gel processing of EGMS and a single source precursor (SSP1). A lyotropic liquid crystalline phase of Pluronic® P123 acts as a structure-directing agent in dilute HCl (1 M).



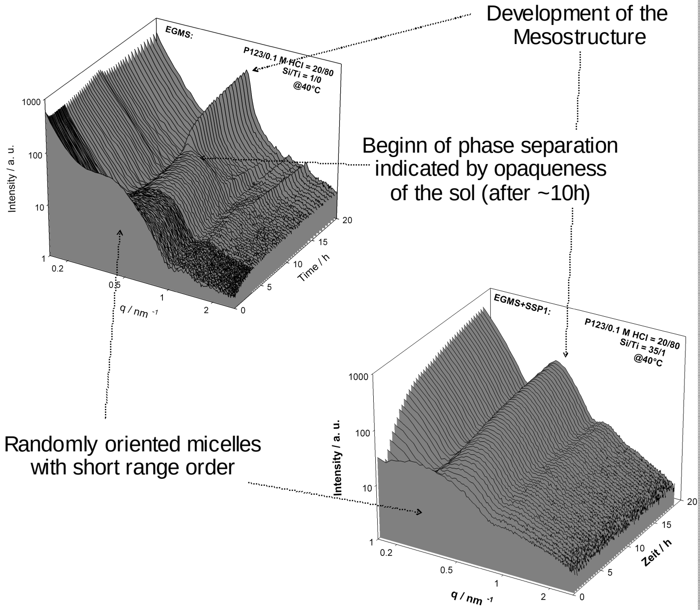
Scattering curves of the mixed oxide monoliths, showing the dependence of the structure on the amount of titanium.

With increasing amount of titanium-centers:

- peak shift towards larger q-values → smaller objects in real space
- peak broadening → indicates weaker ordering of the structure

in-situ SAXS Measurements:

In order to get a better understanding of the structure formation *in-situ* Small Angle X-ray Scattering (SAXS) experiments were carried out during processing, following the structural evolution in the mixture from the sol to the final gel.



Conclusion

- SAXS and GISAXS measurements were performed on Eu^{3+} doped-silica films.
- The domain sizes were calculated with the aid of Scherrer's formula.
- Silica-titania mixed-oxide monoliths were synthesized using novel single source precursors (SSPs). It is shown, that the structure ordering decreases with increasing amount of titanium-centers.
- *In-situ* SAXS Mesurements were carried out, in order to get a deeper under-standing of the structure formation process.

References:

[1] Warren B. E., 1941, Physical Review, Vol. 59, No. 9

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