Técnicas de dispersión de rayos X para caracterización de materiales y superficies

X ray scattering techniques for materials and surfaces characterization

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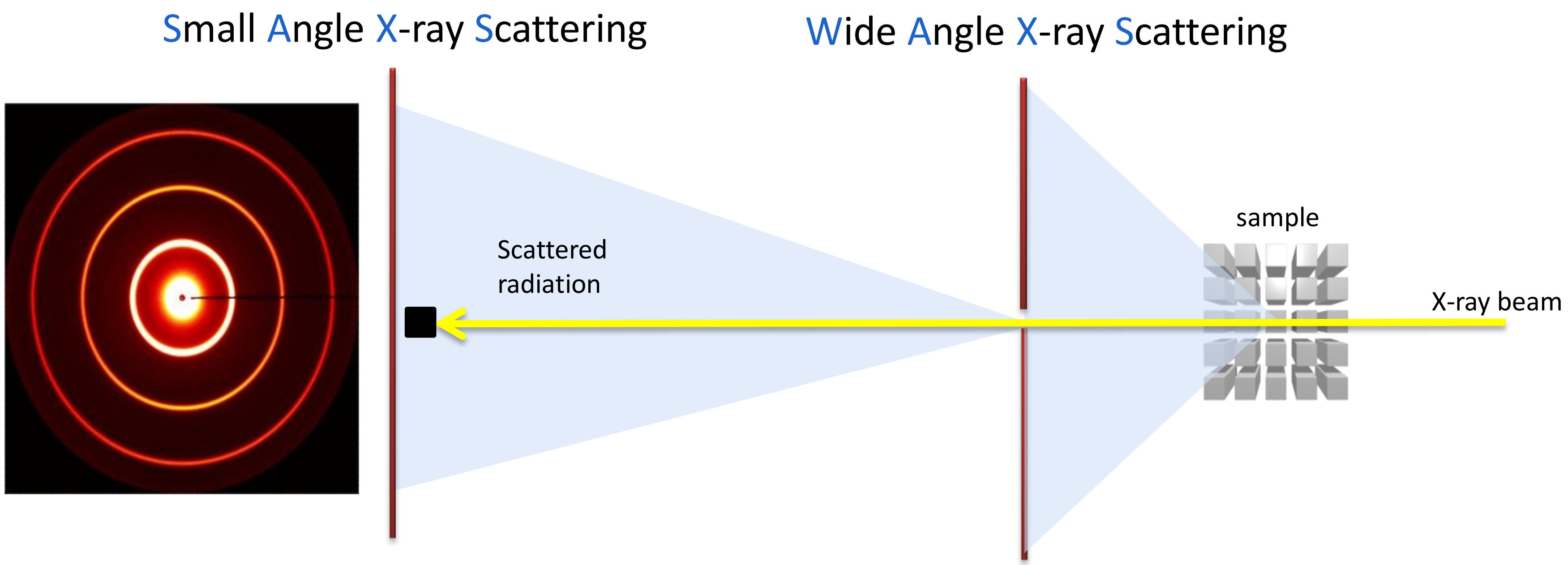
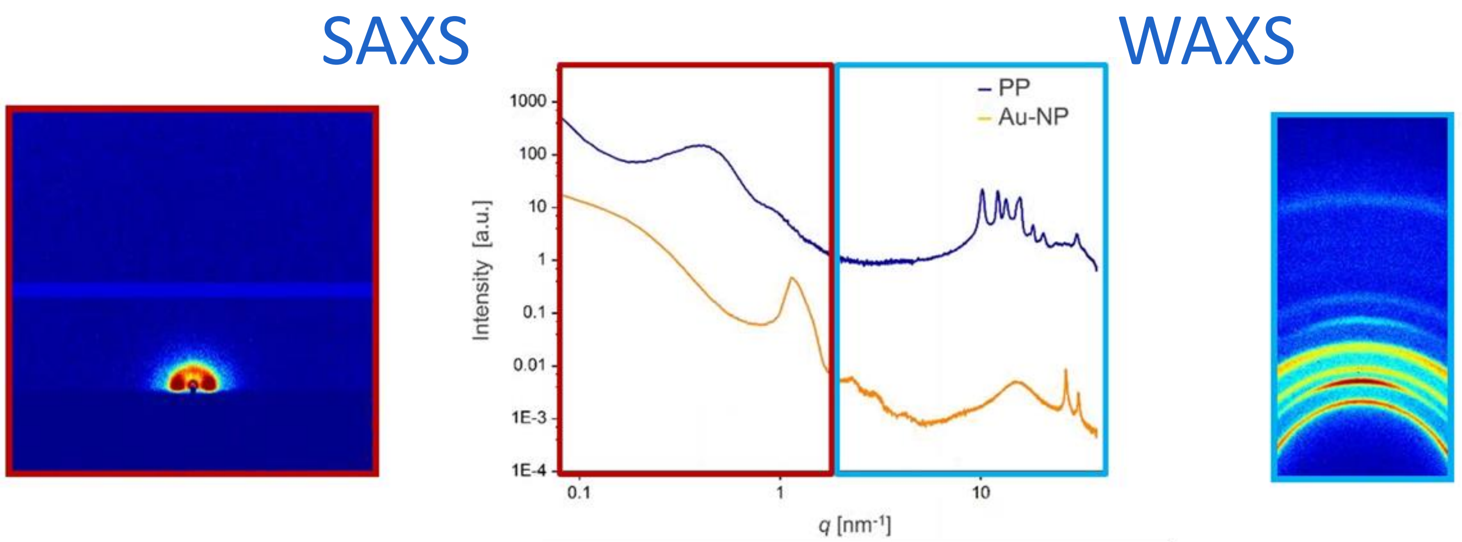
NCD-SWEET beamline

Beamline dedicated to Small Angle X-ray Scattering (SAXS) technique, which provides information about differences on electronic density in the material. It can be combined with Wide Angle X-ray Scattering (WAXS).

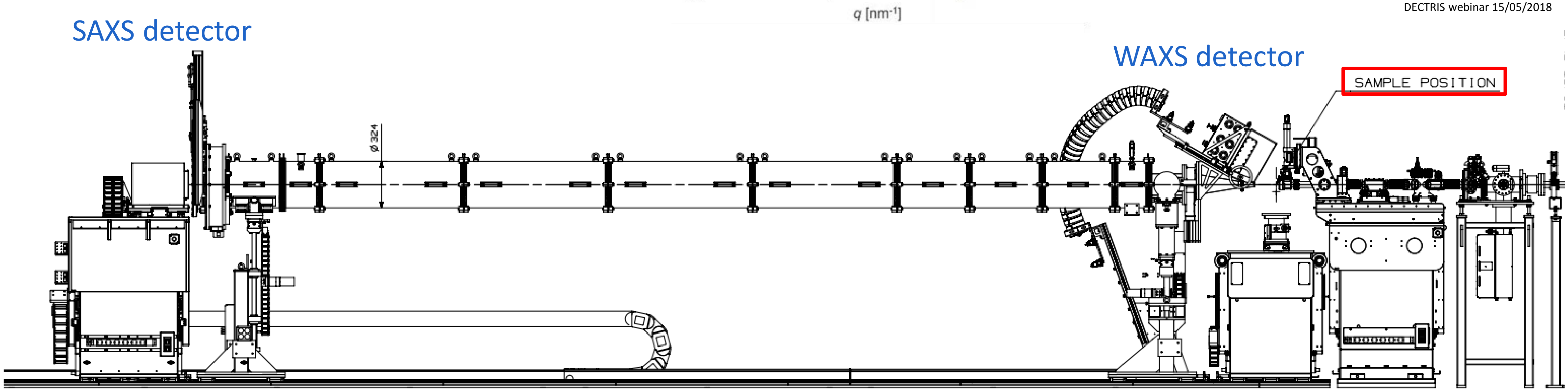
- SAXS proves structure information between 1 - 300 nm
- WAXS provides crystalline structure recording Bragg peaks



NCD-SWEET beamline



DECTRIS webinar 15/05/2018

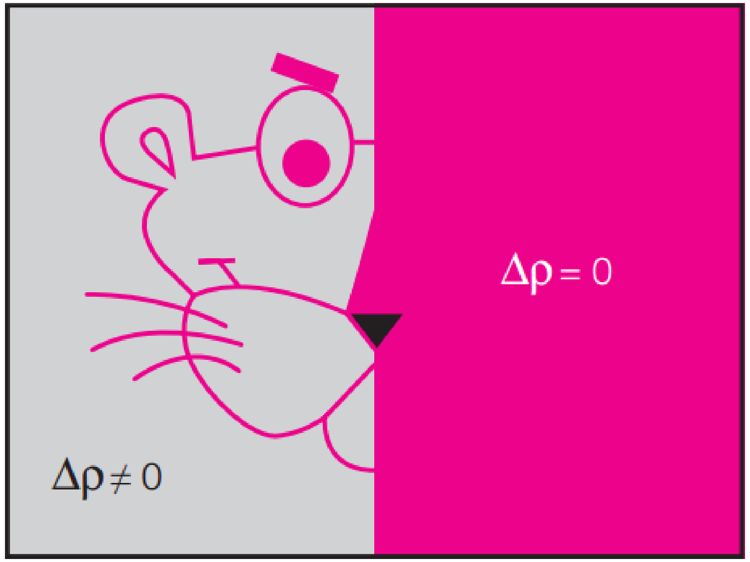


NCD-SWEET beamline

SAXS requires electronic contrast

SAXS is caused by differences on the electron density between a material and its surrounding matrix.

When the monster came, Lola, like the peppered moth remained motionless and undetected. Harold, of course, was immediately devoured.



The contrast in SAXS is the difference of electron densities (shown as pink color) between particle and environment. A pink panther in a pink room is invisible with the exception of the nose which remains visible due to its non-zero contrast.[1]

[1]*The SAXS Guide*, 3rd edition. Anton Paar GmbH 2013

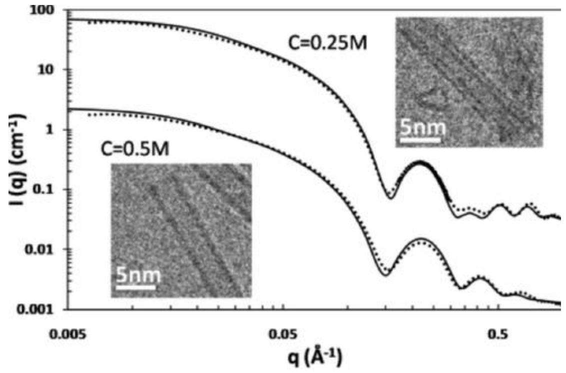
Why do we need SAXS?

Transmission Electron Microscopy

- Real Space image
- High resolution for small details
- Small analyzed area
- Pictures are unique but not always representative
- Small details are visible
- Sample preparation required
- *In* situ conditions limited

Small Angle X-ray Scattering

- Reciprocal Space image
- Low resolution
- Large analyzed area
- Average data are obtained
- Small details are not visible
- Minimum sample preparation
- Wide range of *in situ* conditions



SAXS applications

Microstructure mapping in friction stir welds of 7449 aluminium alloy using SAXS

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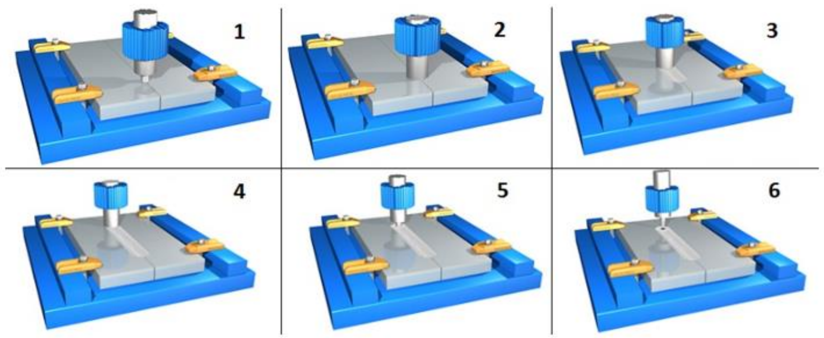

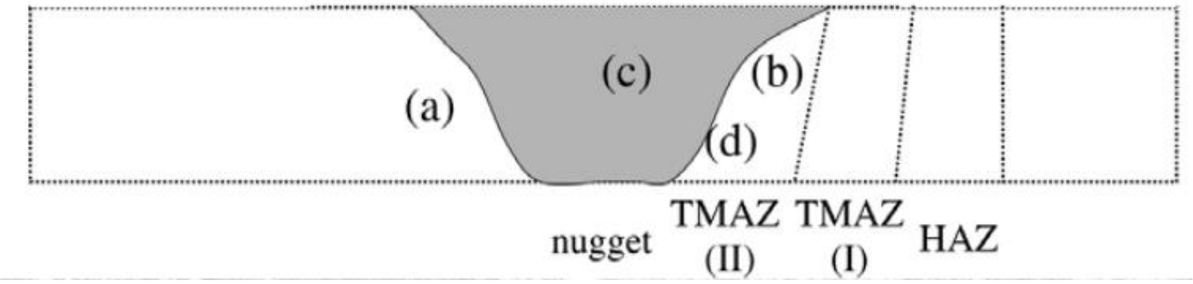

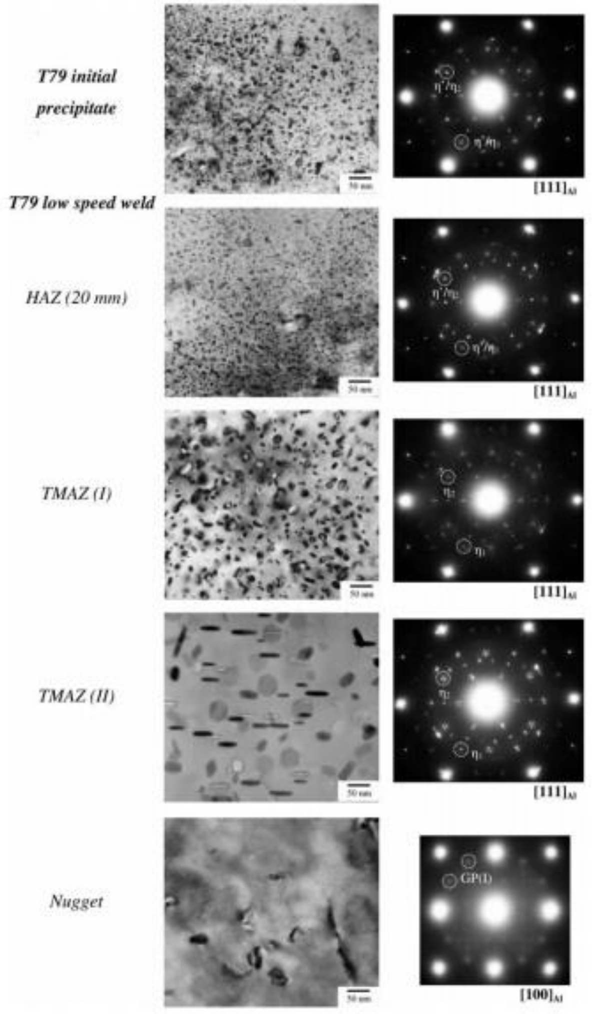

Fig. 6. TEM micrographs of precipitates in the different regions of the T79 low-speed weld. Bright-field images were taken in $(111)_{Al}$ two-beam conditions close to a $[110]_{Al}$ zone axis.

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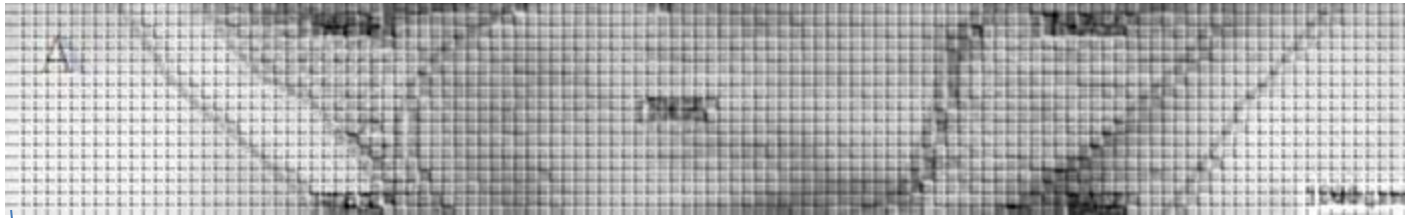
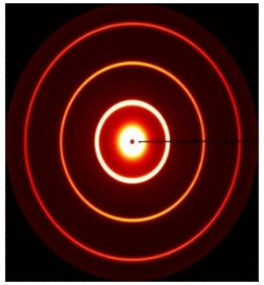
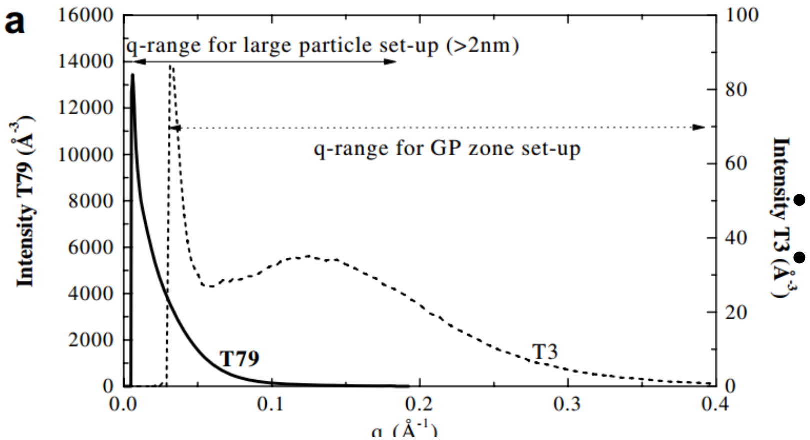
SAXS applications







- Particle radius
- Volume fraction

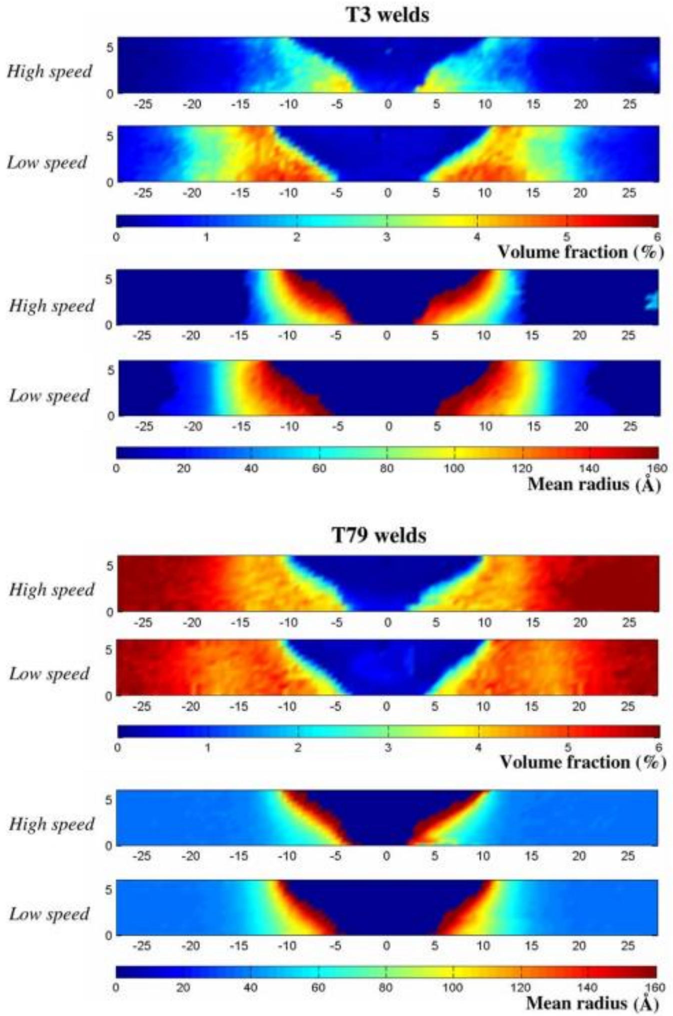


Fig. 3. SAXS maps (volume fraction and size) of η precipitates for the T3 and T79 welds under high and low welding speeds. Note that the friction stir welding tool shoulder diameter was 23 mm.

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…

(ii) The response of the base material to friction stir welding includes all the stages of precipitate evolution: dissolution, growth, coarsening and even nucleation of new phases. Coarsening is the most detrimental effect as it prevents the material from recovering its mechanical properties, either by natural ageing or by post-weld heat treatments. …

(iii) The welding speed (which determines the heat generation per unit time and welded length) is a key factor in controlling the extent of the precipitate evolution irrespective of the initial state (T3 or T79). …it affects the peak temperature but also the temperature gradient. In this respect a lower welding speed will favour coarsening and a broader microstructurally affected zone.

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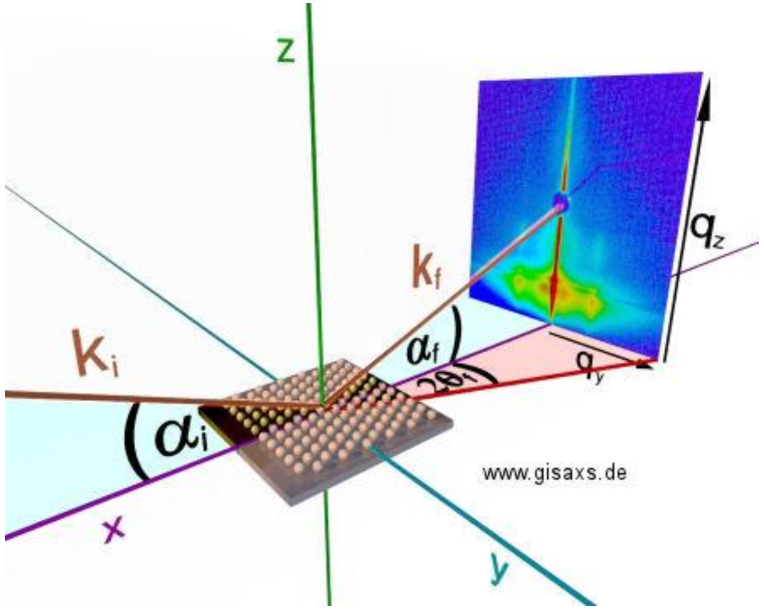
Surface investigations

Grazing Incidence SAXS (GISAXS) fulfill the necessity to investigate nanoscale objects at surfaces, buried interfaces or in thin films.



GISAXS advantages:

- Non-destructive: hard x-ray beam
- Surface sensitive: grazing incidence
- Large sample area: large beam footprint
- Versatile: Wide range of conditions

GISAXS disadvantages:

- Electron density contrast
- Low surface roughness
- Multiple scattering contributions
- Difficulties on data analysis

Surface investigations

Mesostructured alumina as powders and thin films

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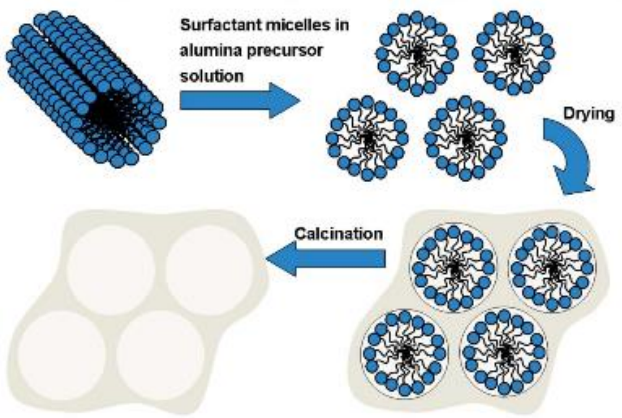

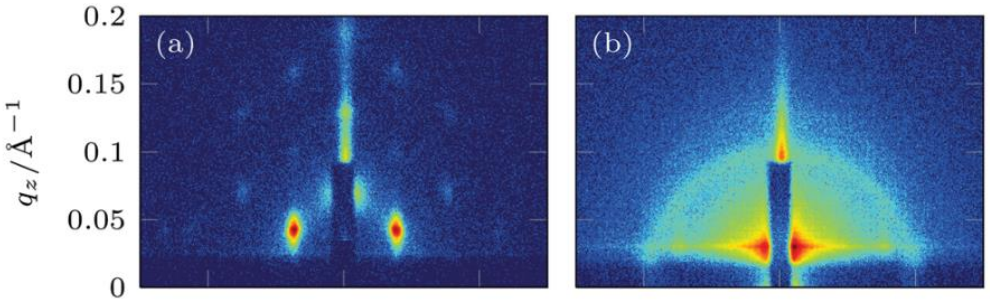

Experimental GISAXS patterns obtained from films prepared from P-123. (a) As-cast film with alumina only, (b) the same film after calcination to 400 ºC, leading to a collapsed and essentially isotropic structure.

Variations in mechanical and thermal properties of mesoporous alumina thin films due to porosity and ordered pore structure

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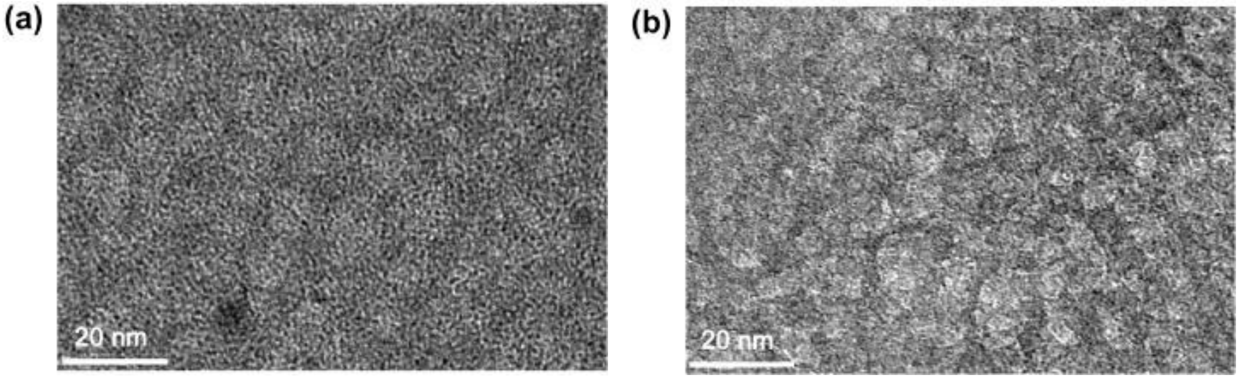
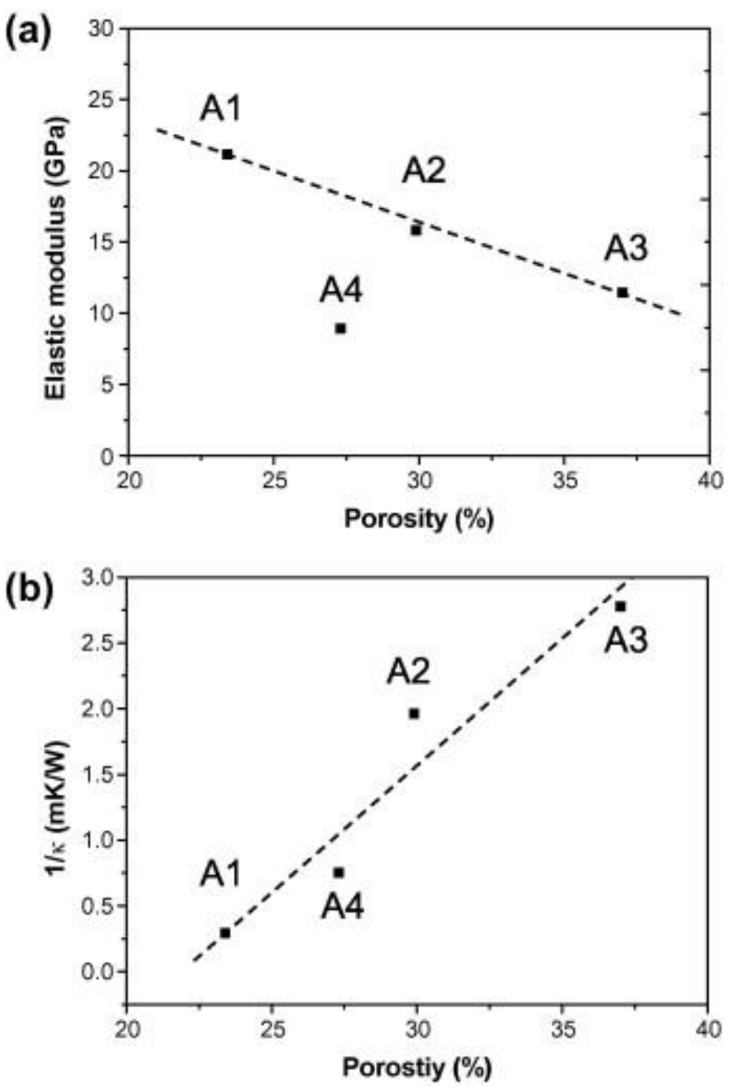
Table 1. Surfactant molar ratio (P-123/Al(OBu^s)$_3$) and porosity of mesoporous alumina films.

Sample	A0	A1	A2	A3	A4
P-123/Al(OBu^s)$_3$	0	0.07	0.14	0.21	0.28
Porosity (%)	5.4	23.4	29.9	37.0	27.3



Journal of Material Chemistry A ,2014, 2, 9727–9735
Journal of Colloid and Interface Science 345 (2010) 120–124

Surface investigations



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… The porosity of the mesoporous alumina films could be controlled between 5% and 37% by varying the surfactant concentration. The pore structure of the ordered mesoporous alumina films was confirmed as a body-centered cubic structure from GISAXS patterns. However, excessively high surfactant concentrations led to a collapse of the ordered pore structure, which causes a reduction in porosity, and a disordered pore structure. As porosity of mesoporous alumina increases, thermal conductivity decreases but mechanical properties are degraded. Ordering in the pore structure improves mechanical properties as the structure allows a better distribution of stress. Specially, the relations between pore structure and mechanical property/thermal conductivity, which were not merely noted in previous literatures, were analyzed in this work, and the fact that a pore distribution as well as porosity is important in mechanical and thermal properties of mesoporous materials was deduced from these relations.

In situ data acquisition

Effect of post-treatment on ordered mesoporous silica antireflective coating

Jinghua Sun,[ab] Ce Zhang,[ab] Cong Zhang,[ab] Ruimin Ding[a] and Yao Xu*[a]

"In situ 2D Grazing Incident Small-angle X-ray Scattering (GISAXS) is a powerful tool to investigate the structure of self-assembled nanostructure coatings."

"The in situ grazing incident small angle X-ray scattering (GISAXS) was used to investigated the structure evolution of the ordered mesoporous silica coating during the calcination process."

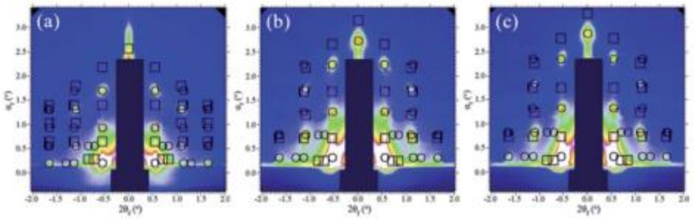

Fig. 4 The *in situ* GISAXS patterns of ammonia-treated coating CN (a) 50 °C, (b) 350 °C, (c) 350 °C maintaining for 3 h.

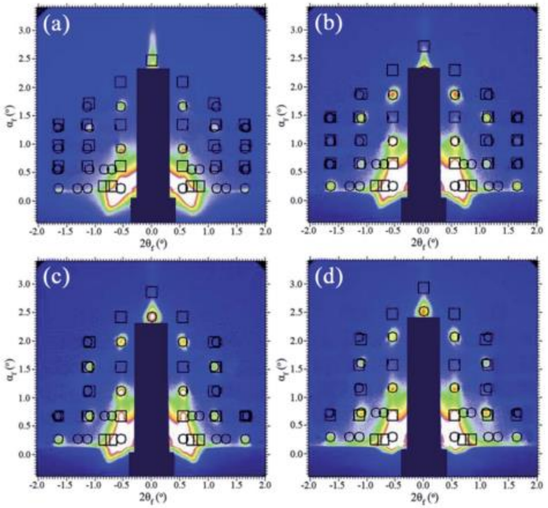

Fig. 5 The *in situ* GISAXS patterns of HMDS treated coating CNH (a) 50 °C, (b) 350 °C, (c) 350 °C maintaining for 3 h and (d) twice HMDS-treated coating CNHH.

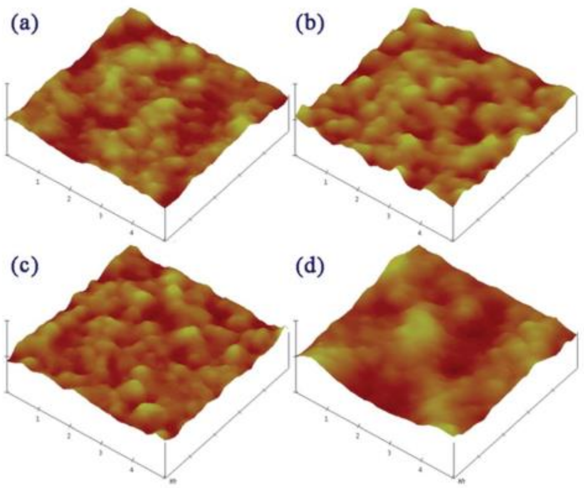

Fig. 7 3D AFM images of ordered mesoporous silica AR coatings: (a) untreated coating C, (b) ammonia-treated coating CN, (c) ammonia and HMDS treated coating CNH, and, (d) twice HMDS treated coating CNHH.

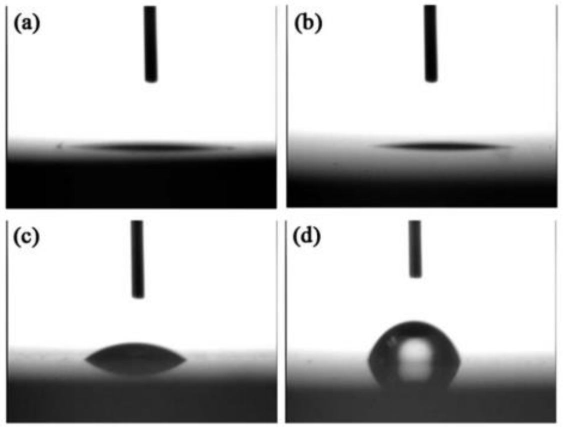

Fig. 8 Contact angle with water of ordered mesoporous silica AR coatings: (a) untreated coating C, (b) ammonia-treated coating CN, (c) ammonia and HMDS treated coating CNH, and, (d) twice HMDS treated coating CNHH.

C: mesoporous silica coating
CN: After Ammonia exposure (30 min) and thermal treatment (350 ºC, 3h)
CNH: After exposure to HMDS (hexamethyldisilazane)
CNHH: After a second exposure to HMDS (hexamethyldisilazane)

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Summary

NCD-SWEET beamline provides a set of tools for *ex situ* and *in situ investigations using* (Grazing Incidence) Small Angle X-ray Scattering ((GI)SAXS) in combination with (Grazing Incidence) Wide Angle X-ray Scattering ((GI)WAXS).

- (GI)SAXS provides information about scatters between 1 to 300 nm: nanoparticle size and shape distributions, pore sizes, characteristic distances of partially ordered materials, etc.
- (GI)WAXS provides structural information below 1 nm: crystalline structure