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European Synchrotron Radiation Facility

High-Energy Scattering with Micro- and Nanobeams

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taylored hydrophobicity at the oil-water interface



$[\mathrm{H}(\mathrm{CH}_2)_m\mathrm{N}(\mathrm{CH}_3)_3\mathrm{Br}]$

Surfactants: m=16 CTAB cetyltrimethylammonium bromide m=18 STAB

 $\phi_{cmc} = 0.92 \text{ mM}$ (CTAB)

L. Tamam et al., PNAS 2011





Complete phase diagram from interface tension measurements







Interfacial structure determined by XRR: C₁₇/STAB



In-plane structure unknown !!



tayloring hydrophobicity at the oil-water interface with N'Ps



Surfactant: CTAB cetyltrimethylammonium bromide

 $\phi_{cmc} = 0.92 \text{ mM}$ (CTAB)

NP size: $r = 2.7 \pm 0.9 \text{ nm}$

D. Calzolari et al., submitted, 2011

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dense NP monolayer

 $\Delta E = 5 \text{ kT}$



dilute NP monolayer

 $\Delta E = 25 \text{ kT}$

In-plane structure unknown



Fuell cells: Hydration of a Nafion membrane

Degree of hydration of the proton exchange membrane (**PEM**) determines fuel cell performance



X-ray transmission measurements through the membrane-electrode assembly in a working fuel cell

V. Rossi Albertini et al., Advanced Materials 21 (2009)







Space/time-resolved water distribution in the PEM under working conditions. Beam 100 x 5 µm². Diffraction patterns were collected during a polarisation curve (inset: 40 mA steps of 30 mins duration) from 0 to 1 A.

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600





Vertical source < 20 μ m – almost theoretical value. After the double Laue monochromator, the source has an aspect ratio of about 8

Measured vertical transverse Coherence ~ 220 µm at 29.2 keV

Vaughan et al., Proc. Risoe Symp 2010

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Nano-diffraction



Nano-diffraction using Si nano-lenses gives a vertical spot of 80nm at 35 keV

- A scan of a 140 nm thick In Film gives ~160 nm wide peak.
 - Deconvolution indicates 80 nm beam height.
- In this configuration:
 - •Diffraction limit ~ 40 nm
 - •Geometrical limit < 20 nm (considering 17 micron source size as measured)
 - •Vertical vibrations in prototype assembly ~50 nm (measured)
 - •Band-pass broadening ~ 80 nm begins to dominate



Diffraction Mapping – Direct Mapping in 2d

Samples with rotational symmetry (many surfaces, films) can be mapped rapidly with line beams



Beams as small as **85x40000** nm² have been used in user experiments (*Paci et al., in progress*)



Diffraction Mapping – 3d Reconstruction

- Many samples for 3d reconstruction contain too many crystals to be mapped with a large beam
- In this case single layers are measured and combined to create a 3d grain map





e.g.,

Poulsen et al., Riso Symposium (2010) Ludwig et al., *Rev. Sci. Instrum.* (2009)



Beam-induced effects at high photon densities

Construction of a dedicated synchrotron scanning SKP



Assess beam-induced modifications in the electronic properties



Complex device for measuring the surface/interface potential with mV accuracy







High-energy beam on sapphire (0001)



Buried interface RTIL/sapphire (0001)

Cation [bmpy]⁺: 1-butyl-1-methylpyrrolidinium; Anion [FAP]⁻: tris(pentafluoroethyl)trifluorophosphate





What would we like to have

- Nanobeam 50-100 nm (pencil or line shape)
- Low divergence
- Large focal depth
- Efficient high energy x-ray detectors

Collaborators

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