

# Small Angle Scattering - Introduction

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Angle & size related thru Braggs' law:

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$d$	$2\theta$	$q$
10 $\text{\AA}$ (0.001 micron)	$8.84^\circ$	$0.628 \text{ \AA}^{-1}$
50 $\text{\AA}$	$1.77^\circ$	
100 $\text{\AA}$ (0.01 micron)	$0.88^\circ$	$0.0628 \text{ \AA}^{-1}$
300 $\text{\AA}$	$0.29^\circ$	
600 $\text{\AA}$	$0.15^\circ$	
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High-angle x-ray scattering (usually  $\sim 2^\circ$ - $160^\circ 2\theta$ ) --> atomic scale structure

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Small-angle scattering --> structure of BIG things

# Small Angle Scattering - examples

WAXS and SAXS study of (m)TMXDI-PDMS siloxane-urethaneureas



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WAXS and SAXS study of (m)TMXDI-PDMS siloxane-urethaneureas



Hard segments --> regions with crystal-like order

Soft segments --> amorphous siloxane chains

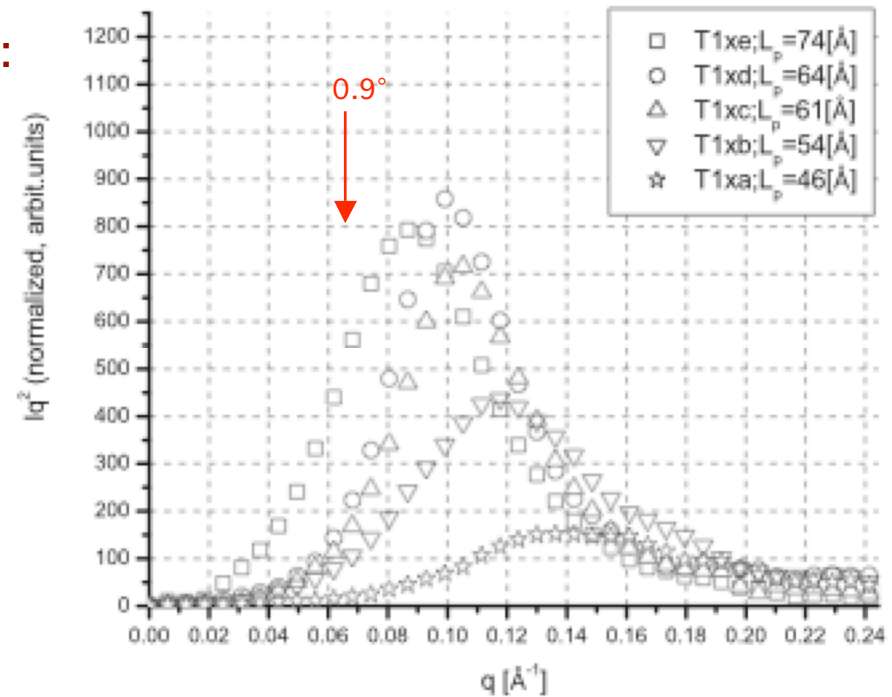
# Small Angle Scattering - examples

WAXS and SAXS study of (m)TMXDI-PDMS siloxane-urethaneureas



Saxs scattering curves for various NCO/OH ratios ( $a = 1.5/1$ ;  $e = 4.5/1$ ):

Size of the hard segment “crystalline” regions changes

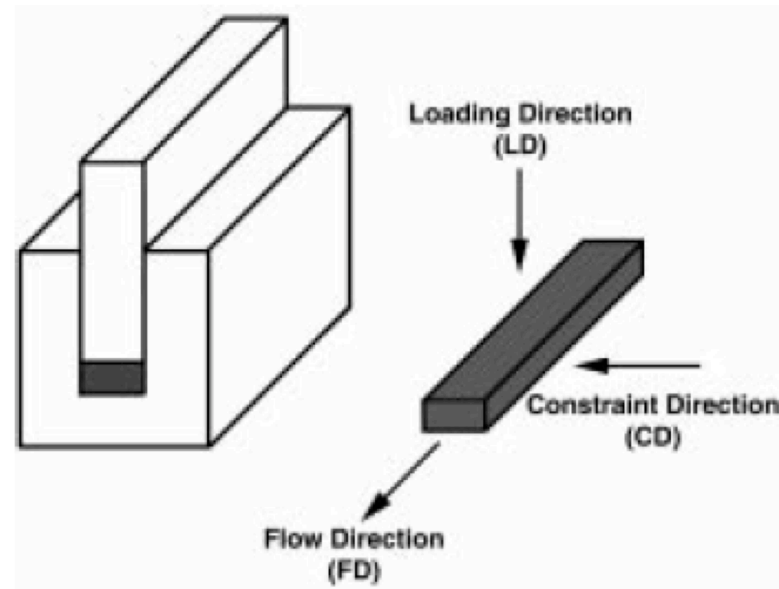




# Small Angle Scattering - examples

Microstructure orientation and nanoporous gas transport in semicrystalline block copolymer membranes

Polymer sheets of semicrystalline ethylene (E)/ethylene-propylene (EP) diblock E/EP and triblock E/EP/E copolymers made by channel die proessing

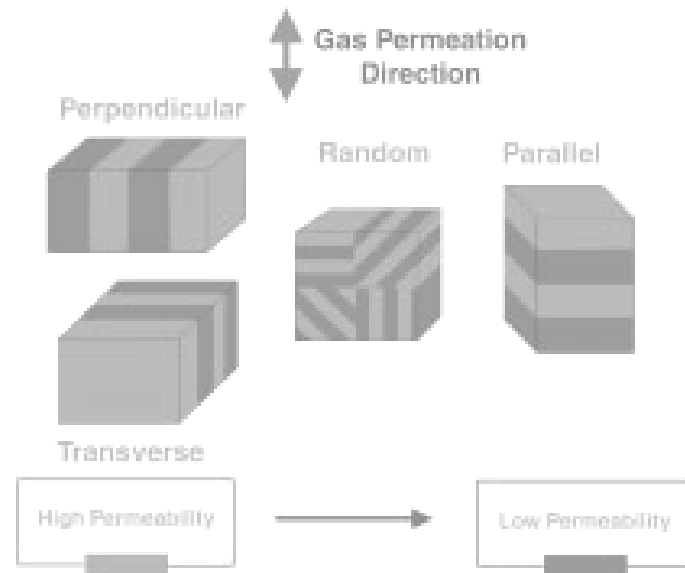


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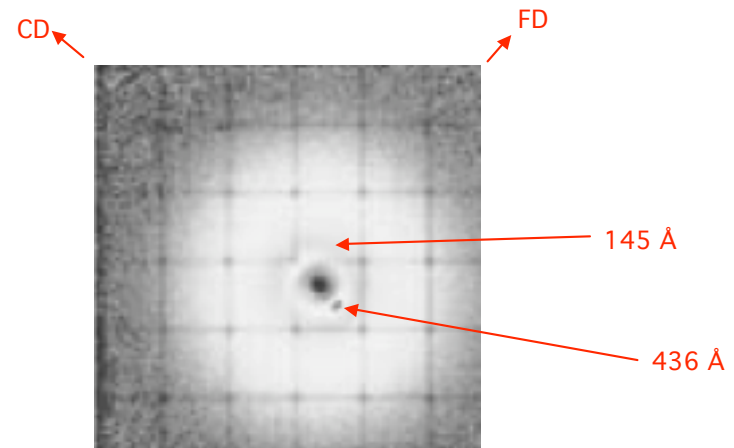
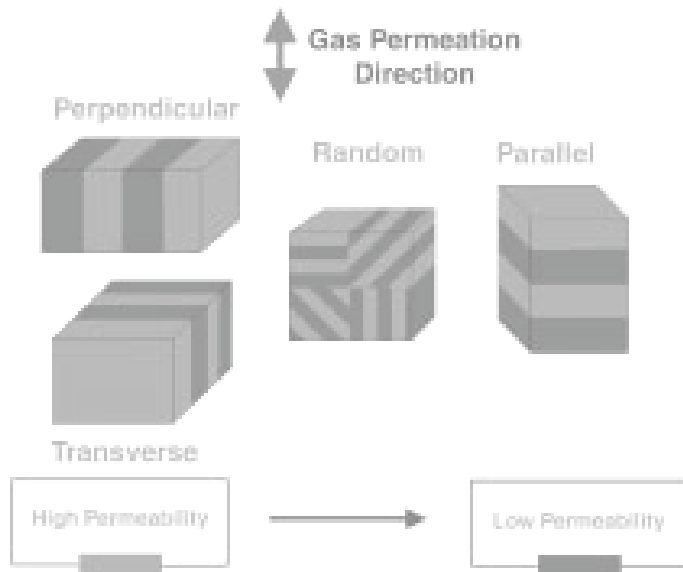
Sheets are stacked into several types of blocks w/ different gas transport props.



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Microstructure orientation and nanoporous gas transport in semicrystalline block copolymer membranes

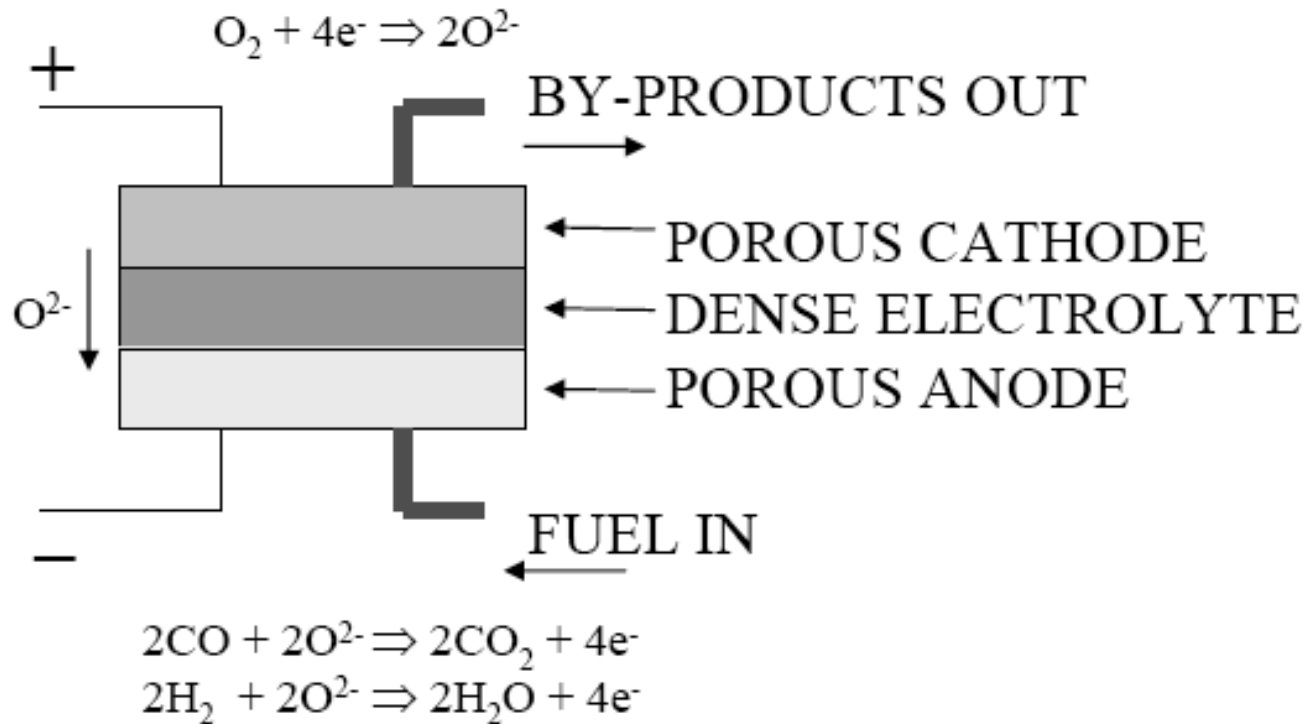
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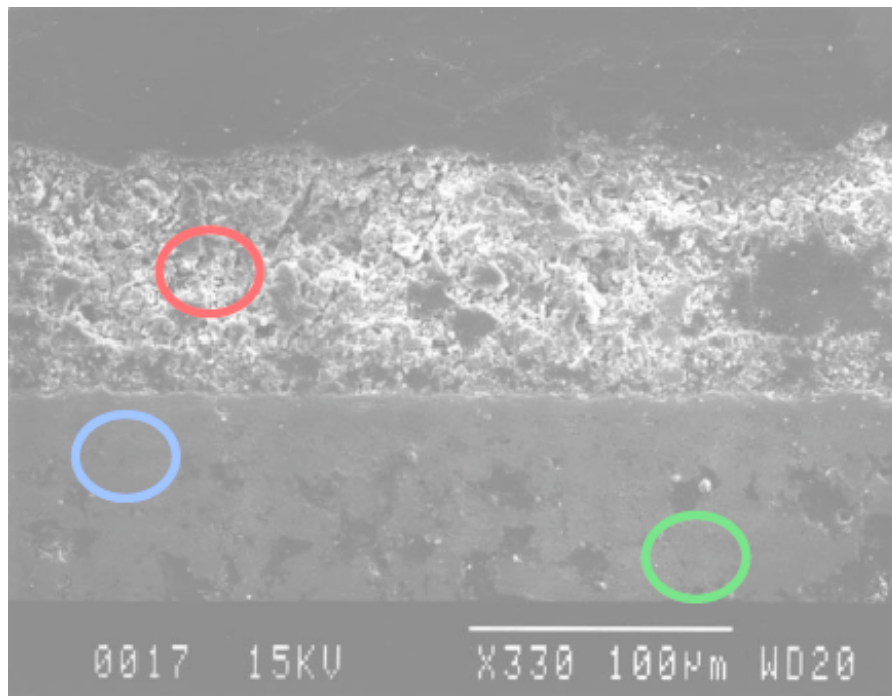
saxs image for perpendicular texture type

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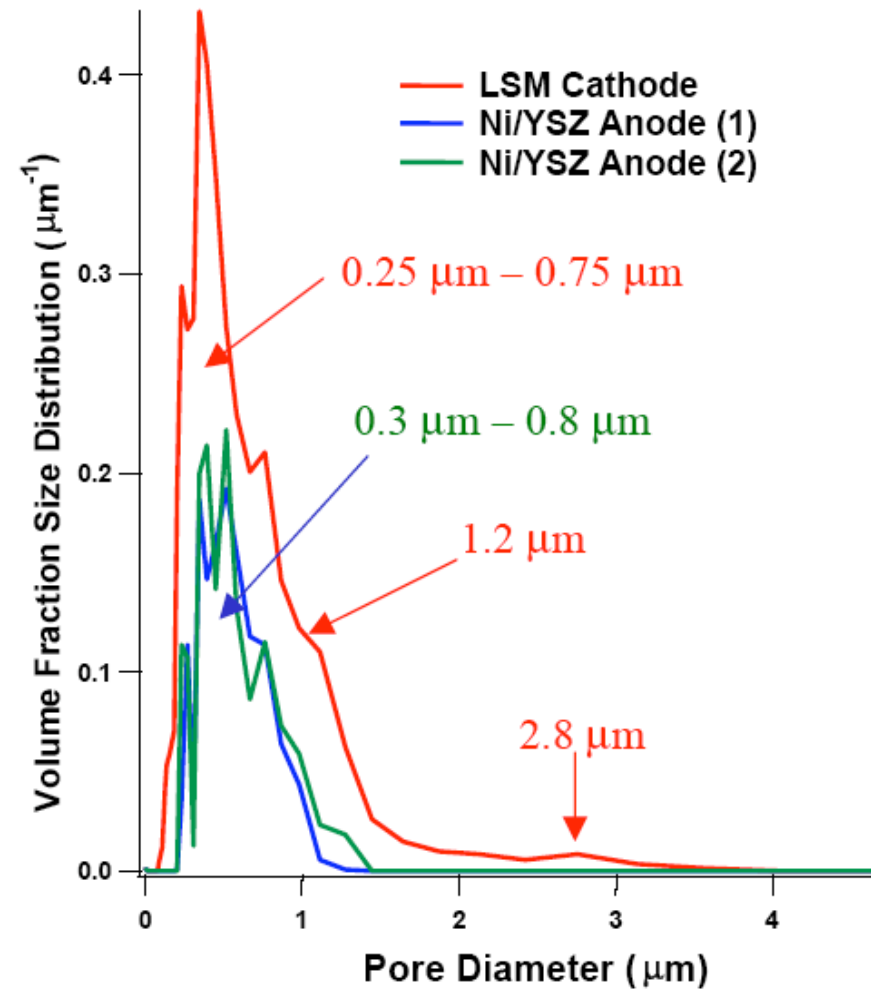
Nanometer to Micrometer Void Microstructure Characterization of SOFC Layers and Interfaces by Small Angle Scattering (SAXS) and Computed X-ray Microtomography(XMT)



Preliminary Void Size Distributions  
obtained from Maximum Entropy  
Analysis of SAXS Data:



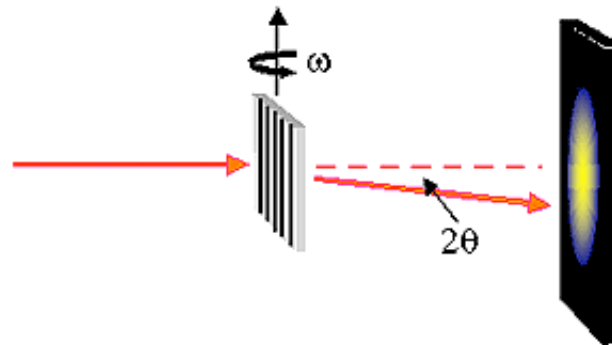
*SEM micrograph near interfaces.*



# Small Angle Scattering - examples

Critical Dimension Metrology of Nanoscale Structures with Small Angle X-ray Scattering

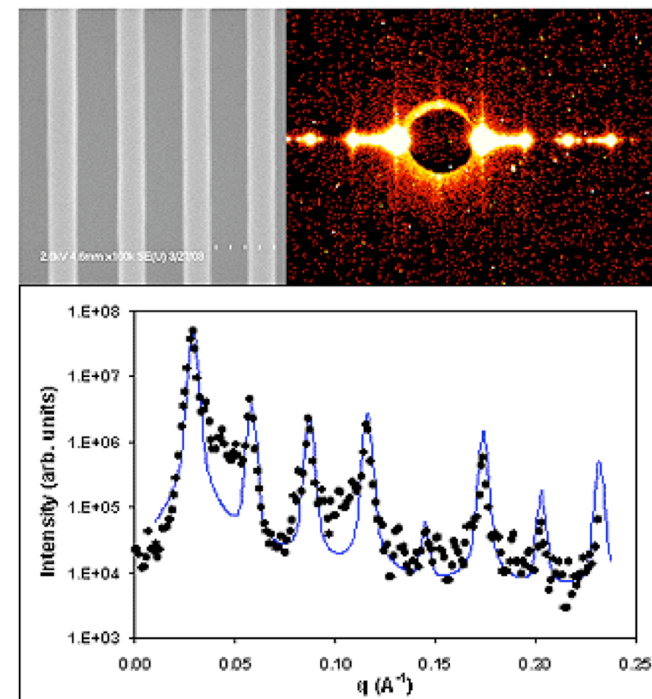
NIST developing transmission saxs method capable of angstrom level precision in critical dimension evaluation over (50 x 50) mm arrays of nanoscale periodic structures



# Small Angle Scattering - examples

## Critical Dimension Metrology of Nanoscale Structures with Small Angle X-ray Scattering

SEM image of a photoresist grating on a silicon wafer & resulting 2-D SAXS image

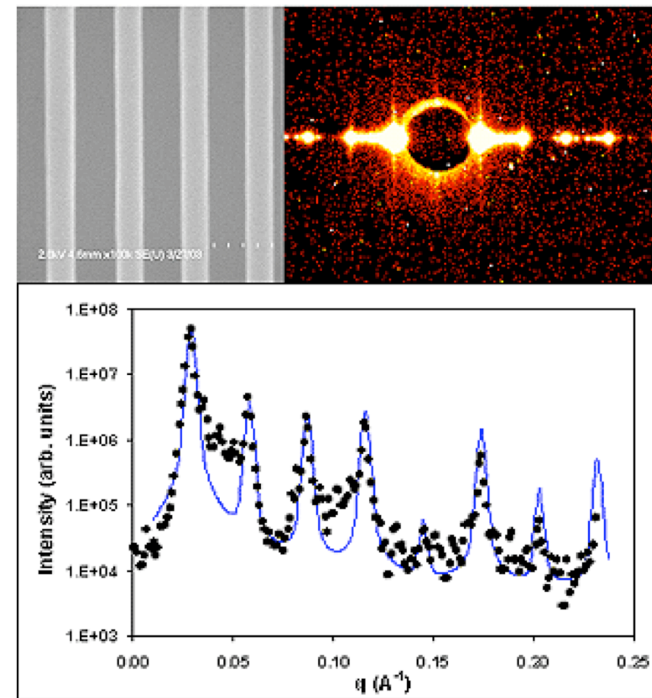


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SEM image of a photoresist grating on a silicon wafer & resulting 2-D SAXS image

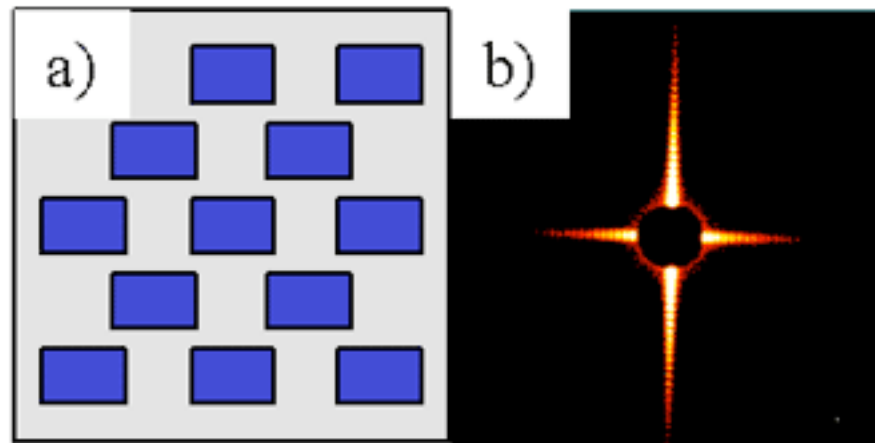
Streaks tell about deviations from ideal grating & defects such as long wavelength line edge roughness





# Small Angle Scattering - examples

Critical Dimension Metrology of Nanoscale Structures with Small Angle X-ray Scattering



- (a) blue rectangles represent etched regions in a film
- (b) resulting SAXS detector image

# Small Angle Scattering - examples

The measurement of the micro-fibril angle in soft-wood

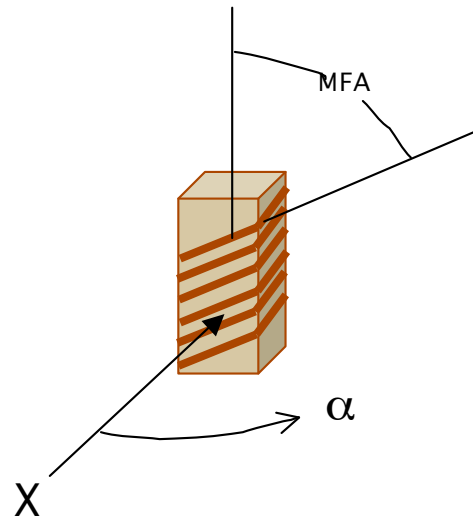
Wood cell wall consists of bundles of a crystalline arrangement of cellulose chains (microfibrils)

# Small Angle Scattering - examples

The measurement of the micro-fibril angle in soft-wood

Wood cell wall consists of bundles of a crystalline arrangement of cellulose chains (microfibrils)

Microfibrils align quite parallel in a spiral around cell wall, with spiral axis along long cell direction



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The measurement of the micro-fibril angle in soft-wood

Typical saxs patterns from Norway spruce - mean MFA of  $20^\circ$  - longitudinal cell axis vertical. (c) pattern recorded at  $\alpha = 0^\circ$ . (d) pattern recorded at  $\alpha = 45^\circ$

