

First Results of GI-WAXS

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New Insights in the Growth Behavior of Thin-Film-Technology

Klaus Attenkofer

Bernhard Adams

Marcel Demarteau

Seon Woo Lee

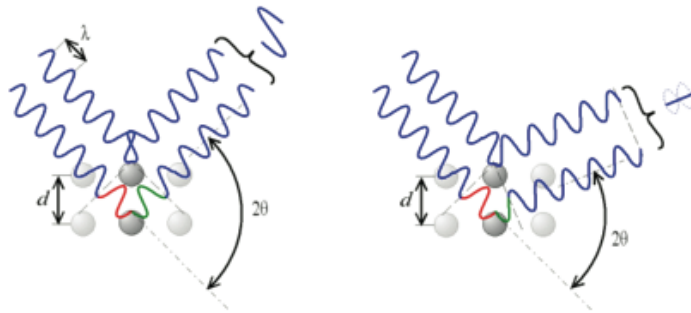
John Smeedley

Overview

- The technique and what can be learned
- The collaboration with BNL
- The last beamtime: What is possible
- The future

The Technique and What Can be Learned

Basics of (X-ray) Scattering

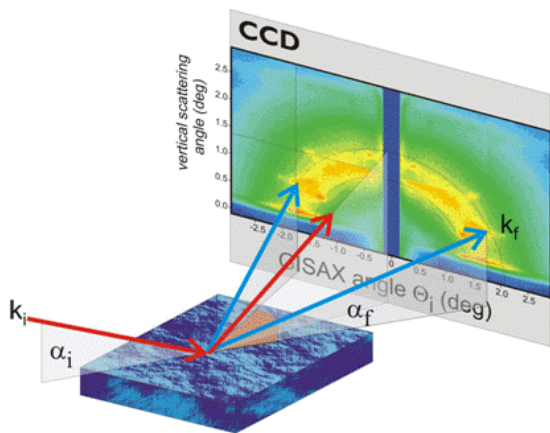


- The elementary process
 - Each atom scatters X-rays in 4π
 - An ensemble of atoms:
 - Crystalline form produces “bragg”-peaks
 - Amorphous materials produce a “Pair-Distribution-Pattern”

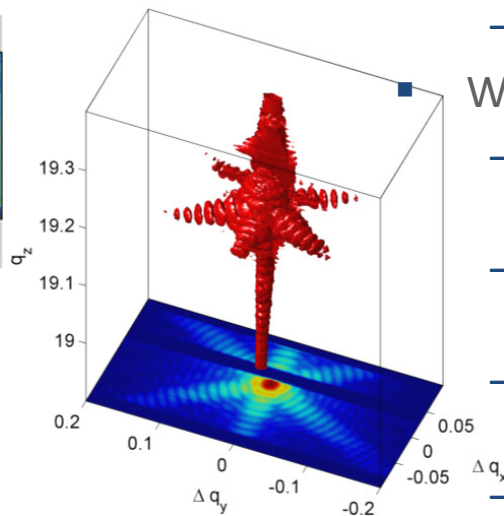
- Single wavelength diffraction:
 - Single crystal produces typically only one reflection (or none)
 - A powder of single crystals produce Rings

What information is in the diffraction pattern

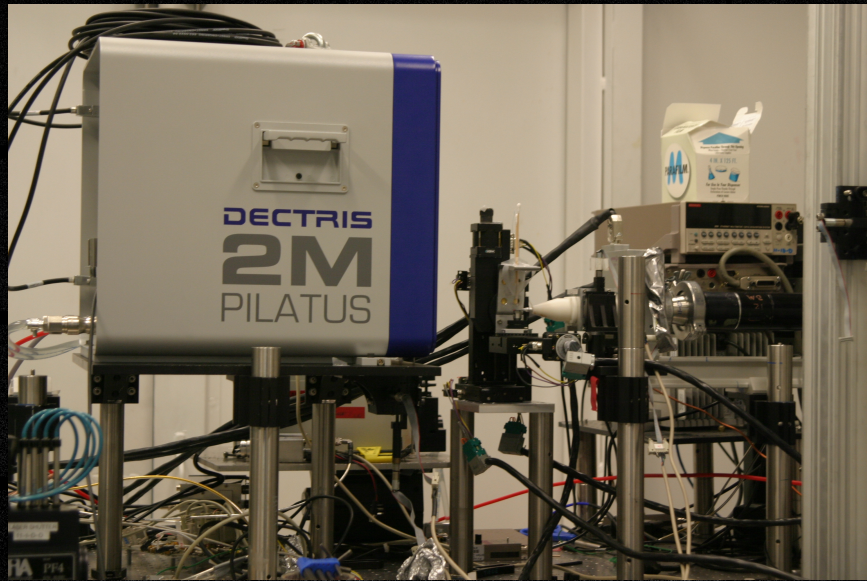
- 2-Theta position is a measure for the lattice-plane distance
- Phi-position reflects orientation of the crystallites
- Width and shape of the reflection reflects crystallite size and/or strain of the crystal
- Detailed analysis of peak-shapes will produce electron-density map of sample



general GISAX-setup



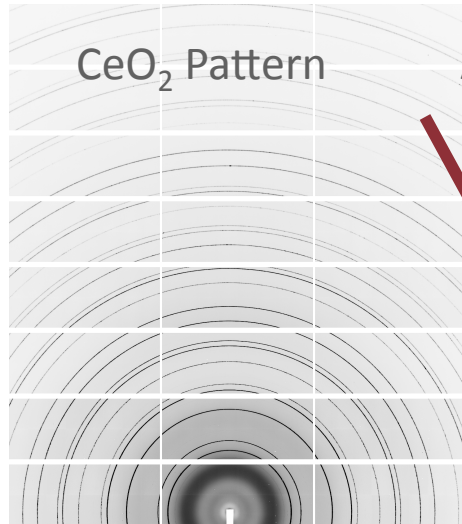
The Experimental Setup



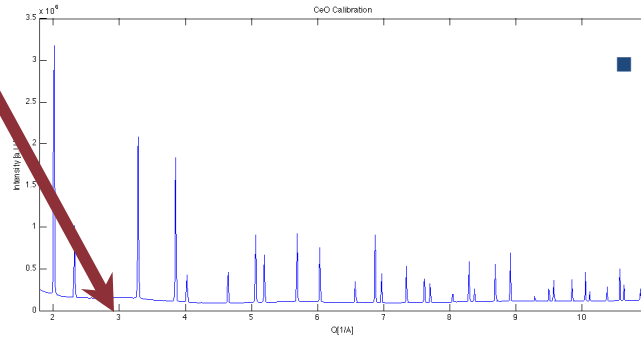
- X-ray parameters:
 - Energy: 22KeV
 - Size: 16 μ m x 400 μ m
 - Divergency : ?
- Sample manipulation:
 - Theta-motion
 - Phi-motion
 - X- and z-translation
- Data-acquisition:
 - Required time/picture: 0.1-1s



Data-Processing

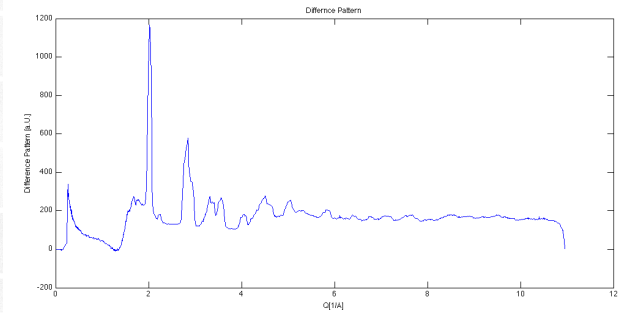
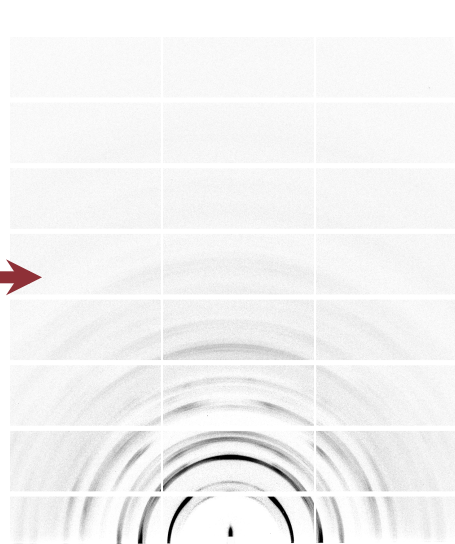
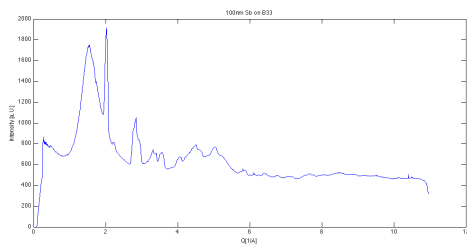
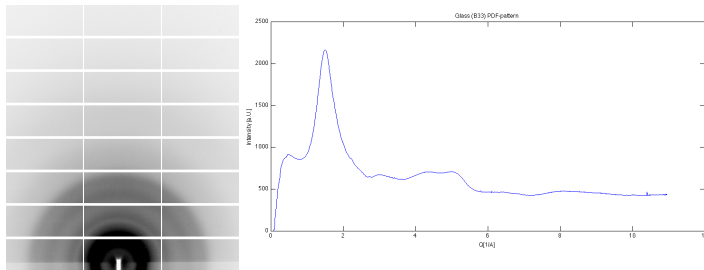


Azimuthal Integration and fit of peak positions

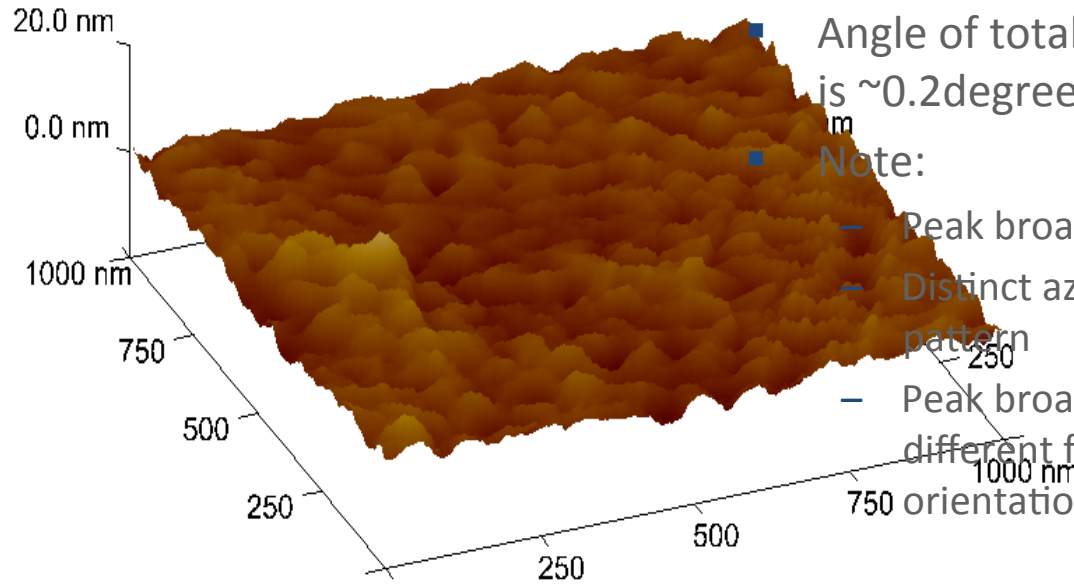
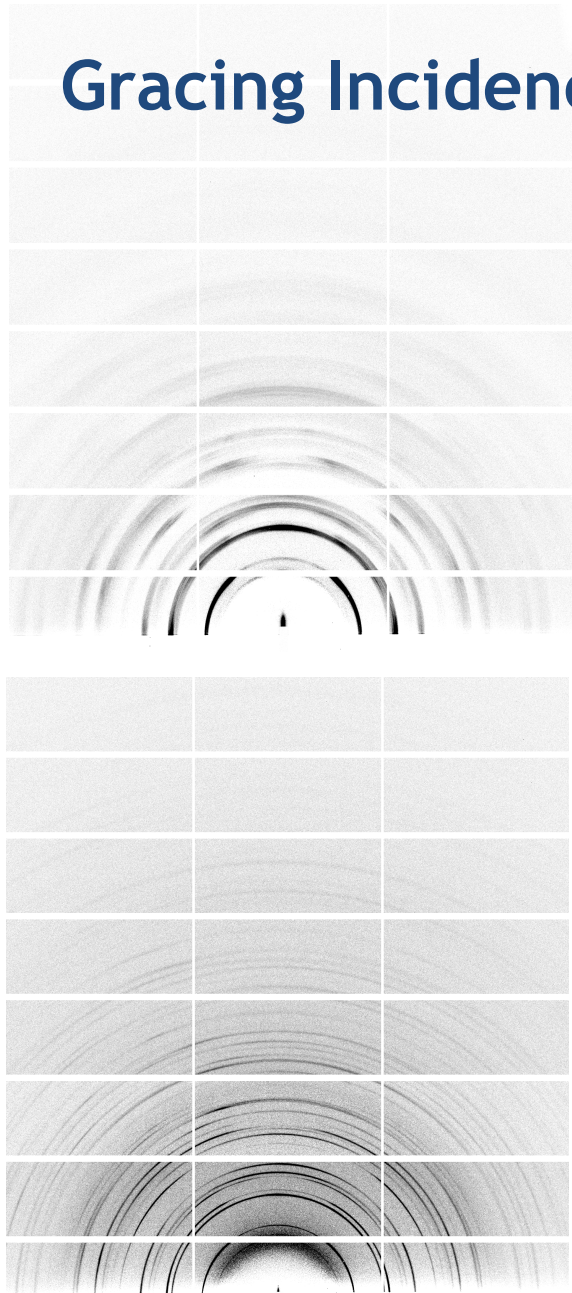


Data-Analysis:

- Calibration with known standard (CeO₂)
- “Empty”-pattern (B33)
- Sb-on B33 pattern
- Result: Difference showing only Sb-film and changes on glass-substrate

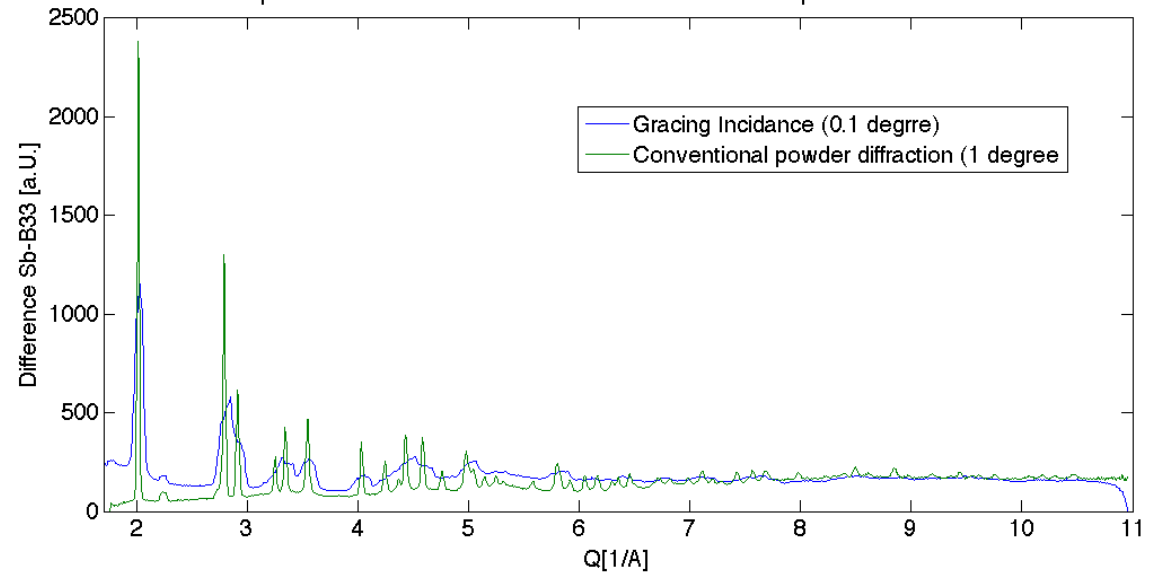


Gracing Incidence Techniques:



- Theta-angle determines Probe-depth
- Angle of total reflection is ~ 0.2 degrees
- Note:
 - Peak broadening
 - Distinct azimuthal pattern
 - Peak broadening is different for different orientations!

Comaprision Gracin incidence with "conventional powder diffraction"



The Collaboration with BNL (John Smedley)

- Setup of Insitu-setup
 - Splitting of cost
 - Splitting of development effort
 - Splitting learning-experiences
 - Growth of sample systems
- Data-analysis
 - ANL will take the lead
 - Splitting of Matlab-scrips
 - Collaboration with other groups for data-analysis (Byeongdu Lee, Dag Breiby/Martin Nielson)
- Beamtime logistics:
 - First beamtime at BNL and BNL
 - Proposals are in place (for 2 additional visits)
 - Next beamtime at BNL with insitu setup
- Additional support at BNL: Insitu STM/AFM setup

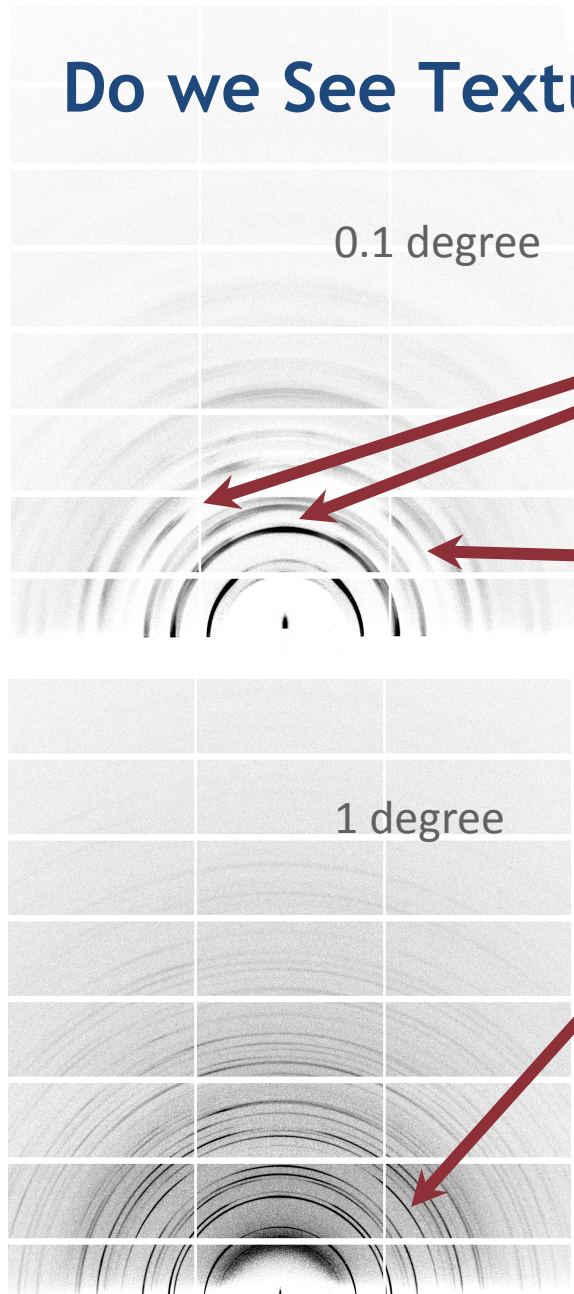
The last beamtime: What is possible

Overview

- Goals of beamtime:
 - Setup-optimization
 - Stability test of beamline
 - Substrate subtraction
 - Background reduction
 - Geometry
 - Creating first data
 - How to analyze data
 - What can we expect to measure
 - Scientific goals:
 - Phase determination?
 - Texture?
 - Can peak-broadening observed (distribution function.....)
 - Is it possible to characterize the lateral structure?
- Setup & samples:
 - Simple exsitu setup

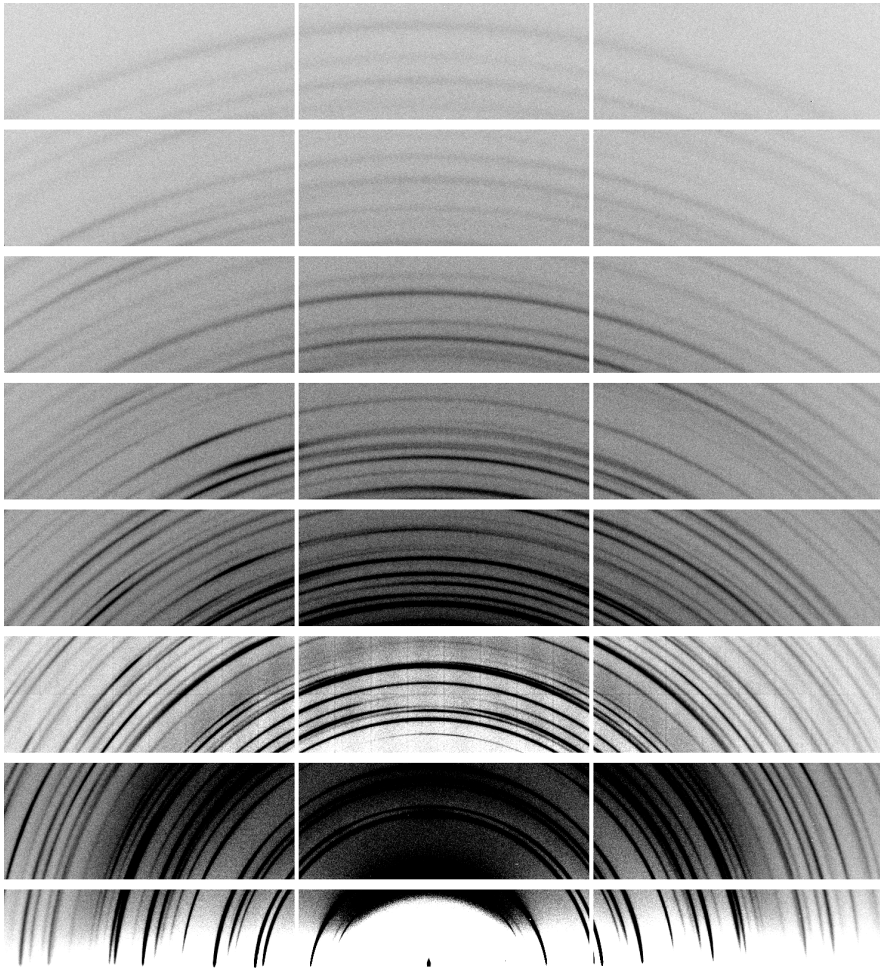
Property	Sb-on B33	Sb1 on Mo	Sb2 on Mo	Sb3 on Mo	Sb4 on Mo	Sb5 on Mo	Sb6 on SS
Sb-thickness [nm]	100	190	7	120	120	28	180
coating	TE	SP	SP	SP	SP	SP	TE
Substrate temperature	RT	RT	RT	150C	RT	RT	150C
Annealed	no	no	no	no	400C	no	no

Do we See Texture and Depth-Information

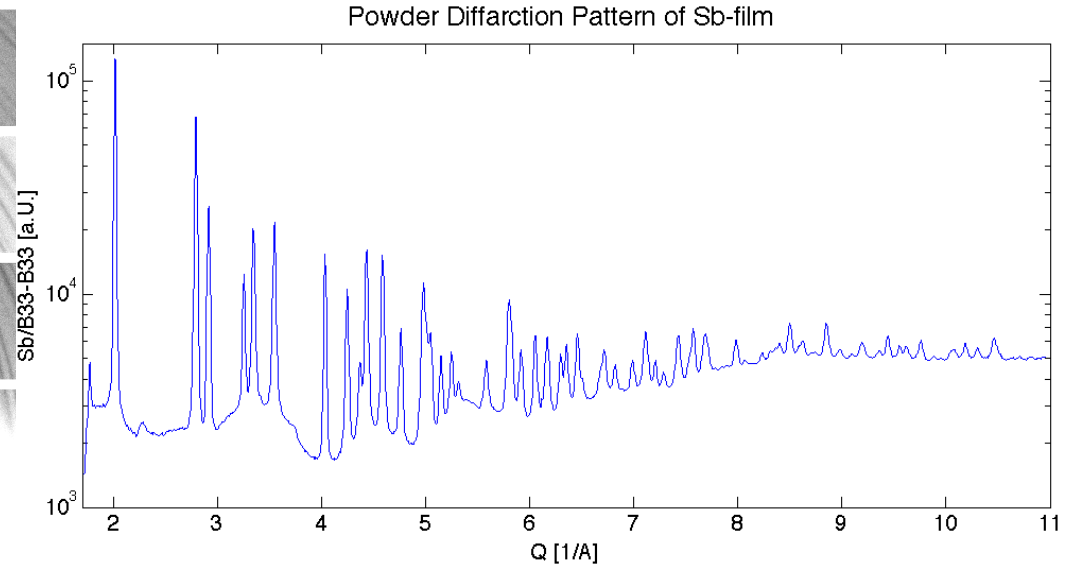


- Clear Texture
- Peak broadening for grazing incidence
 - Either
 - Strain of grains on the surface
 - Smaller grain size on the surface (broadening depends not on azimuthal angle!)
 - No indication for crystalline SbO
 - In principle amorphous SbO can be detected (analysis has to be improved)
- Peak-analysis shows that
 - Only crystalline Sb exists
 - Quantitative analysis (pole-figure) is on the way (Collaboration with Dag Breiby/Martin Nielson)
 - Grain size analysis should be possible

Phase-Purity and Amorphous Contributions

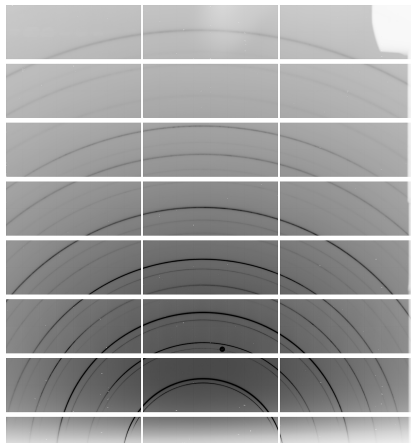


- All peaks can be coordinated with crystalline planes of Sb
- Remaining Background cannot be contributed to B33
 - PDF of amorphous contributions
 - Systematic error due to beam-instabilities

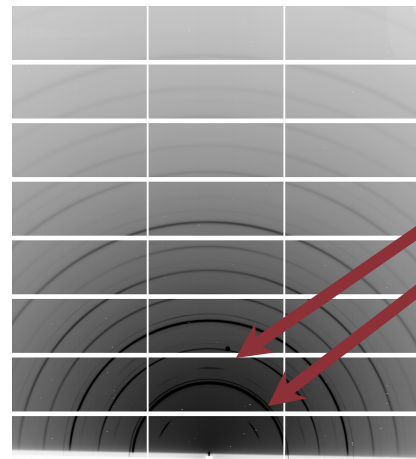


Comparison of Various Sb-Thickness on Mo Substrate (Sputtered no heating of substrate)

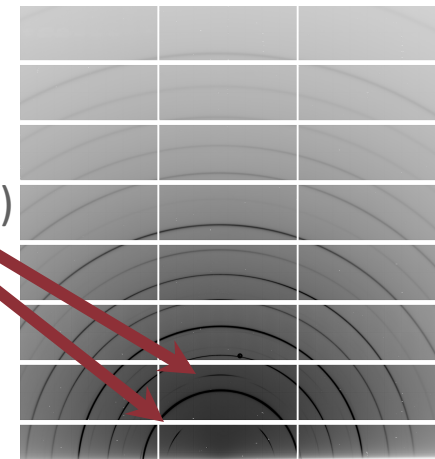
7nm Sb



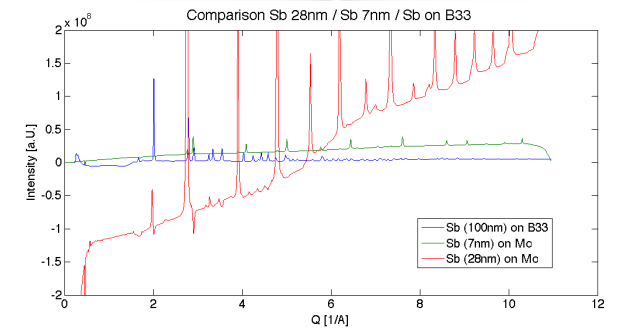
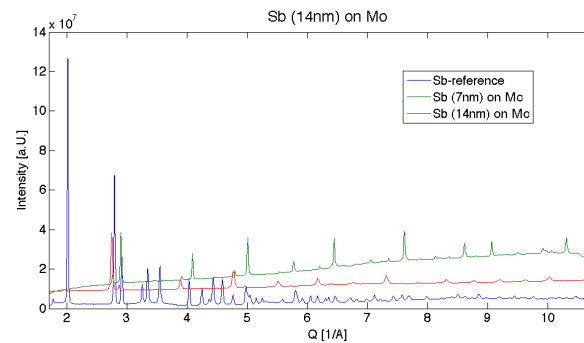
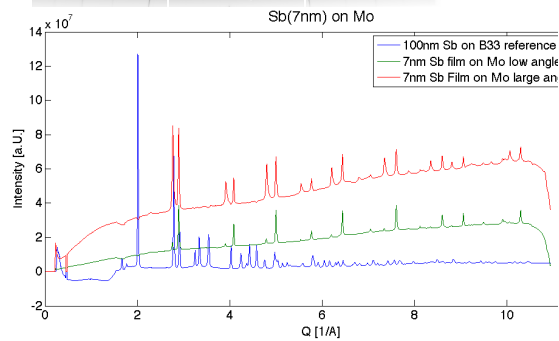
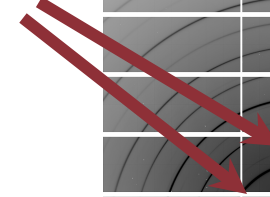
14nm Sb



28nm Sb

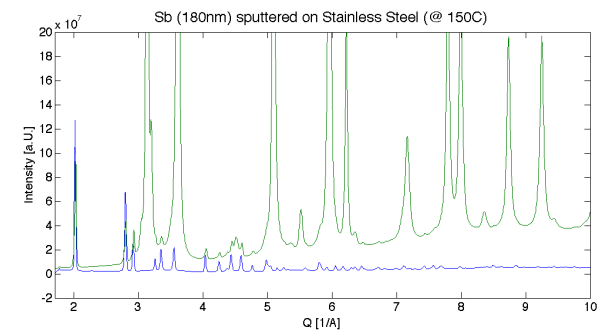
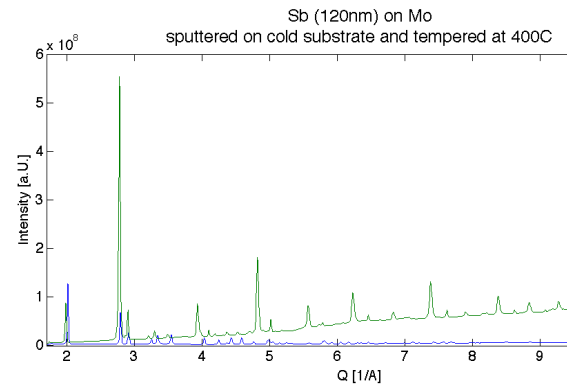
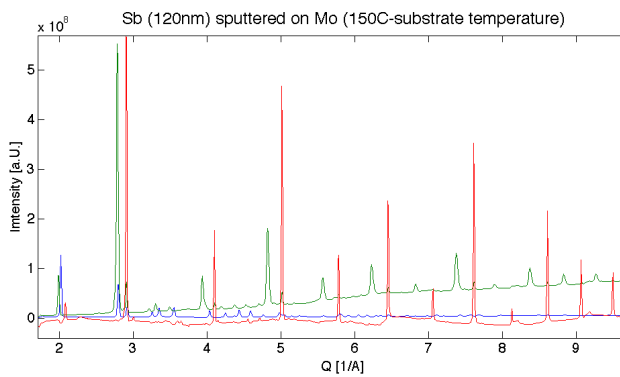
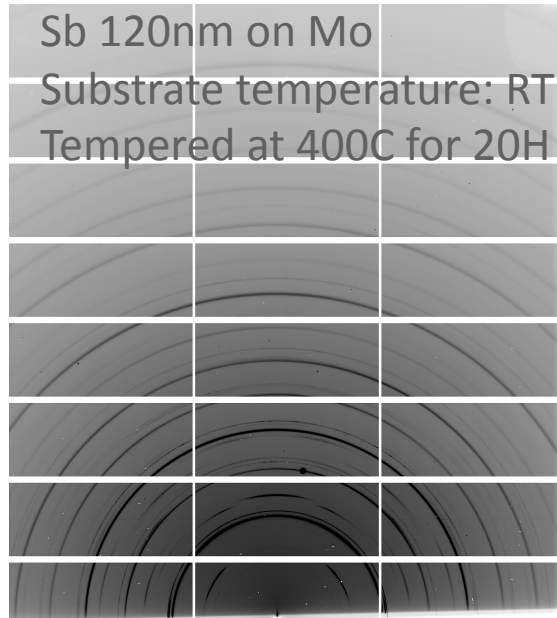
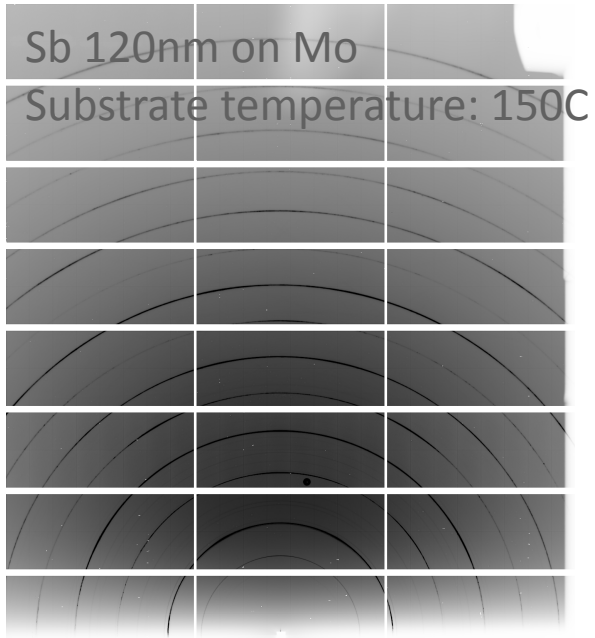


Sb
(Textured)



Mainly Mo-scattering
(perhaps some Sb?)

Influence of Substrate



The Future

- Experimental Setup:
 - Insitu setup:
 - first step will be glass-envelop
 - Later dedicated UHV-chamber
- Next samples:
 - Comparison of growth with different substrate
 - Epitaxial growth (similar to sapphire and GaN)?
 - Sputter versus thermal evaporation
 - Substrate temperature and temper
 - Co-evaporation versus sequential evaporation
 - Sb/K co-evaporated
 - Sb evaporated and inter-diffusion of K
- Data-analysis:
 - Automation of data analysis with matlab
 - Quantitative analysis of texture

Conclusion

- Gracing incidence technique was applied successfully to Sb-films similar to that which are used in cathodes
- Diffraction pattern allows to
 - Determine co-existing crystalline phases
 - Visualize texture (if existing)
 - Possibly:
 - Quantitative analysis of texture
 - Analysis of amorphous PDF background
- Preliminary results:
 - Sb (100nm) on B33
 - Thermal evaporation results in textured film
 - Only crystalline Sb is observable (no oxide)
 - Sb on Mo substrate
 - Below 7nm amorphous growth
 - Above 14nm polycrystalline strong textured growth for sputtered films
 - Only non-oriented growth for thermally evaporated films