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2ND INTERNATIONAL CONFERENCE AND EXPO ON CERAMICS AND COMPOSITE MATERIALS

SHERATON BERLIN GRAND HOTEL ESPLANADE, 10785 BERLIN, GERMANY

NEW X-RAY DEVELOPMENTS TO CHARACTERIZE CERAMIC MATERIALS

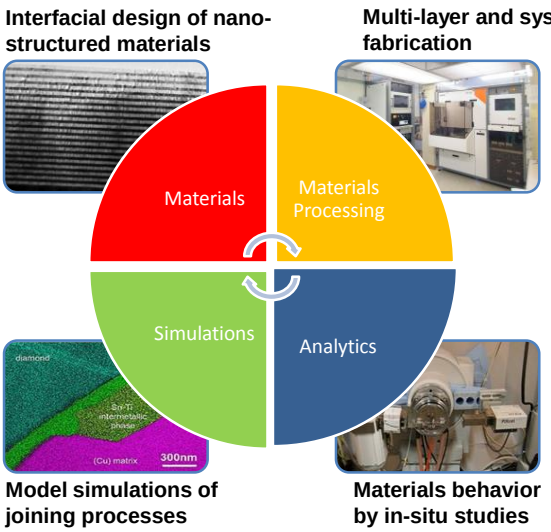
A. DOMMANN*, O. KUZIOR, A. FLISCH, S. HARTMANN, R. KAUFMANN AND A. NEELS,
EMPA, SWISS FEDERAL LABORATORIES FOR MATERIALS SCIENCE AND TECHNOLOGY,
CENTER FOR X-RAY ANALYTICS, SWITZERLAND



www.empa.ch/x-ray

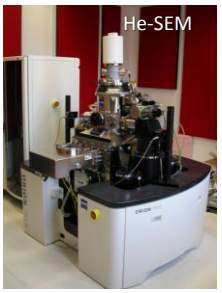
Accelerate progress by sharing know how

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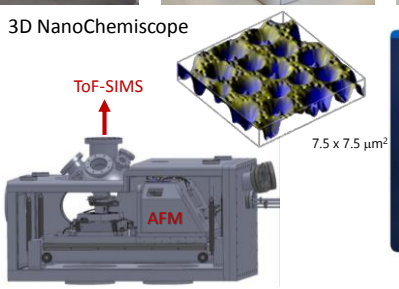
Surface Analytics







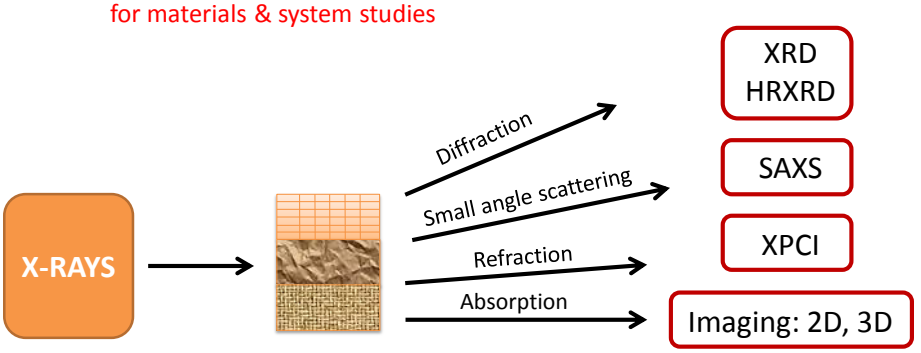






X-ray Analytics: Methods & Tools

X-ray tool & methods optimisation
for materials & system studies



Low to high Energies

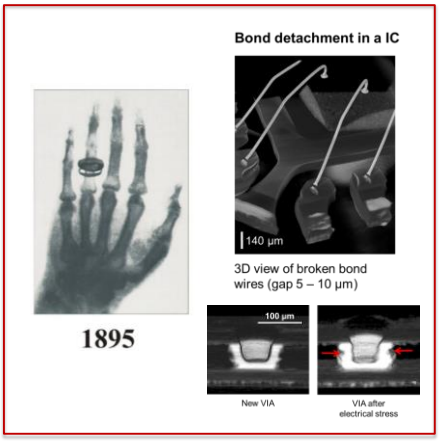
In-situ studies:
- Temperature
- Humidity
- Mechanical stresses

Detector developments

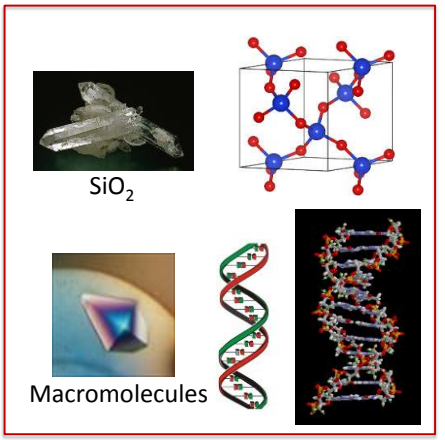
X-ray Analytics: Imaging & XRD

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Imaging
→ Seeing the invisible

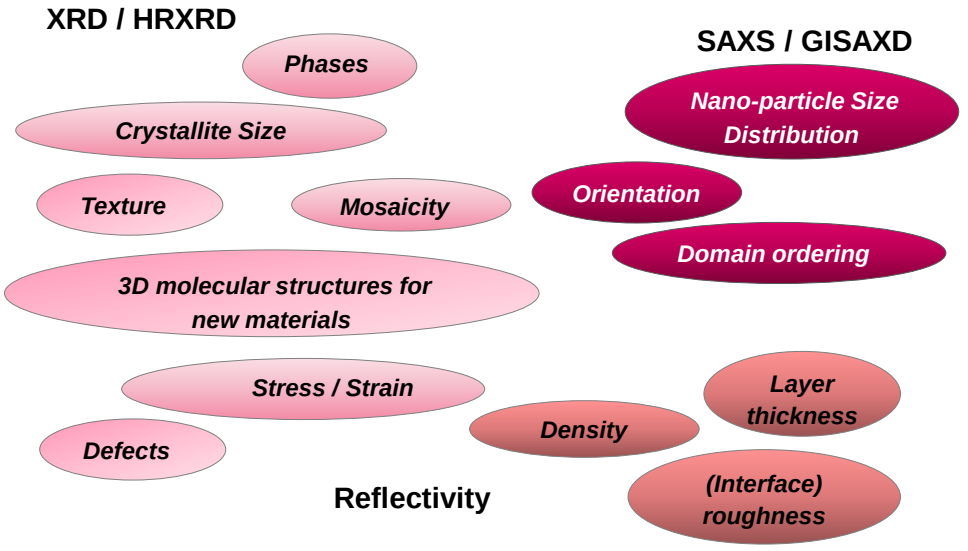


Atomic and Molecular Structure
→ Where are the atoms



Structural properties addressed by X-ray techniques

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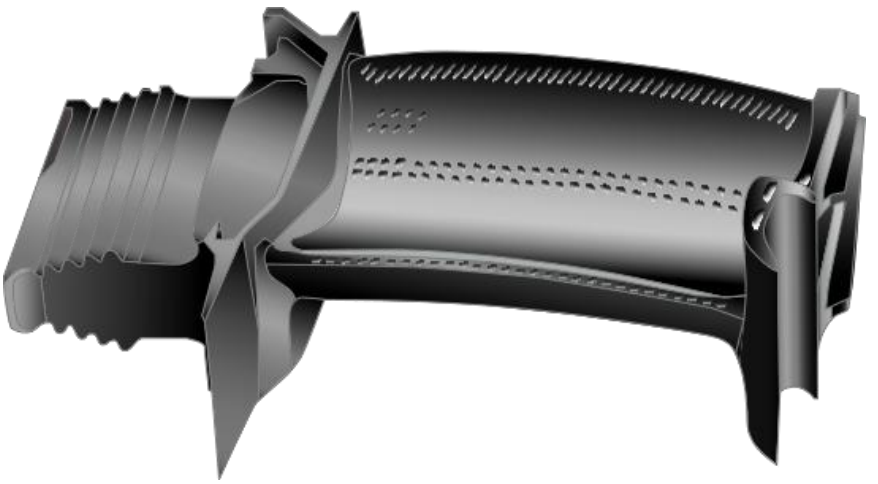
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- Hard Coatings: $(Al,Cr)_2O_3$ & $(Al,Hf)_2O_3$



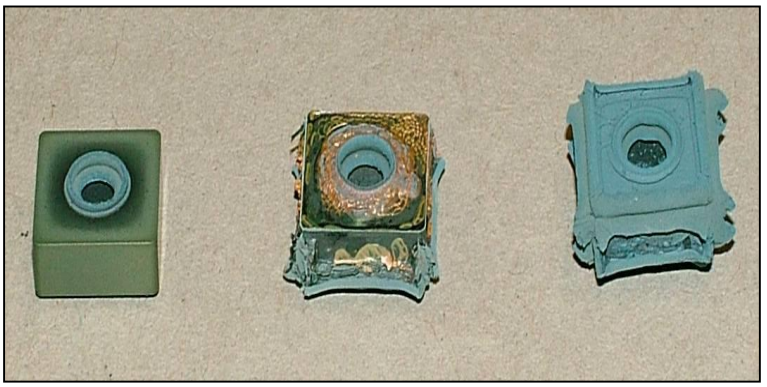
THERMAL BARRIER COATINGS (TBC)

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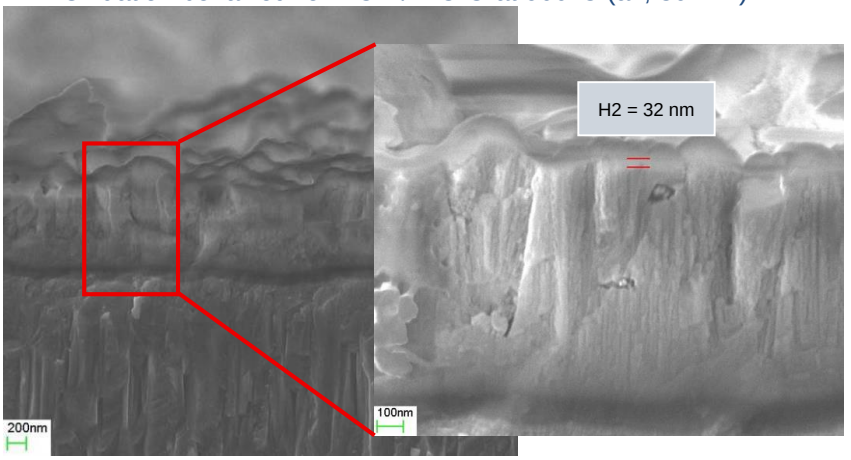
Thermal Stability

$(Al,Cr)_2O_3$ TiCN uncoated



Thermal stability

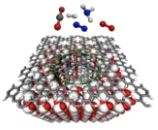
Oxidation behaviour of TiCN / AlCrO at 900°C (air, 30 min)



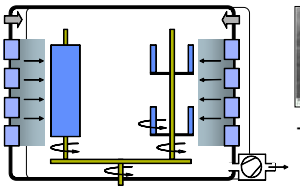
Support in process development: Hard coatings

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Dedicated Design of High Temperature Corrosion Resistant PVD Oxide Coatings for Automotive Applications

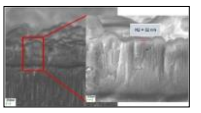


Simulations

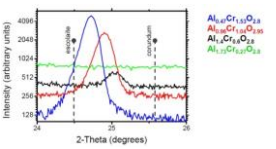


Understanding

Reactive cathodic arc evaporation (PVD)

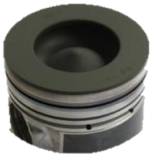


Testing



Characterization:

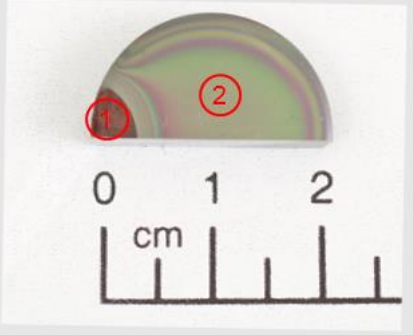
- XRD, *ex-* and *in-situ*
- RBS
- Mechanics



œrlikon balzers coating

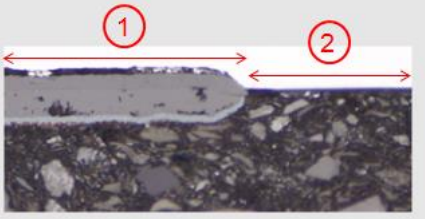
High Temperature Corrosion Resistant Al-Cr-O Coatings Deposited by Arc Operation

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Substrate: steel
Coating: $(Al_{0.7}Cr_{0.3})_2O_3$ (corundum)
Coating thickness: 2 µm

Annealing: ambient, 500°C, 100 hours

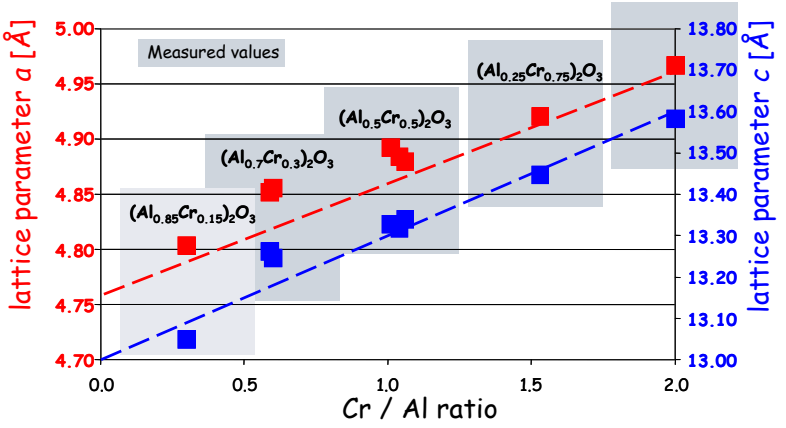


(1) Region without oxide coating after annealing resulting in a corrosion thickness of ≈ 30 µm
(2) No corrosion was detected in the coated region

œrlikon balzers coating

Crystal Structure Consistency with Vegard's law

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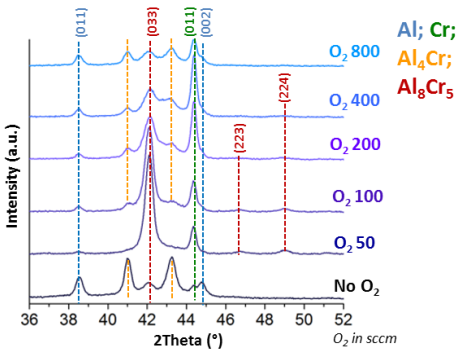
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balzers coating

Cathode surface vs layer composition

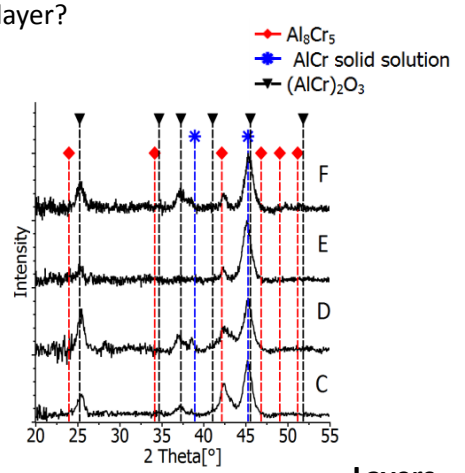
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- Phase transformation process at the cathode surface?
- How this influences the deposited layer?



Cathodes



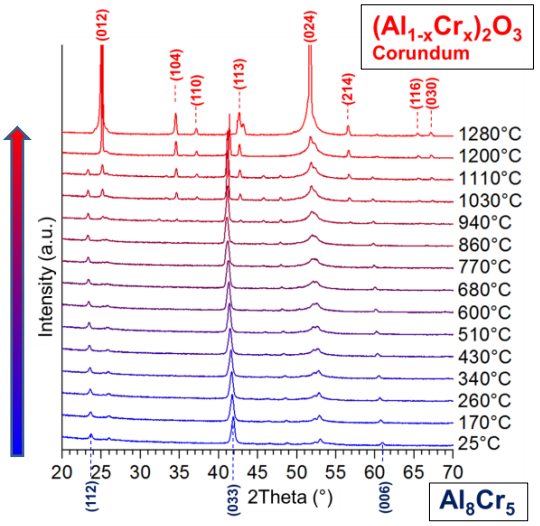
Layers

Al-Cr system:
XRD High temperature experiment

- Phase transformation at high temperature
- Progressive oxidation study
- Stability of the coating



XRD Chamber at 1300°C



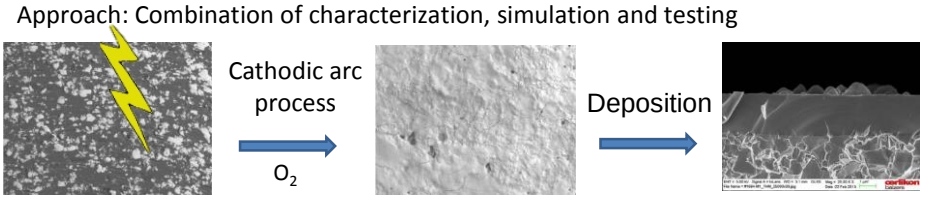
Wear resistant hard coatings

Oerlikon developed a complete new deposition method:

- Reactive Cathodic Arc Deposition

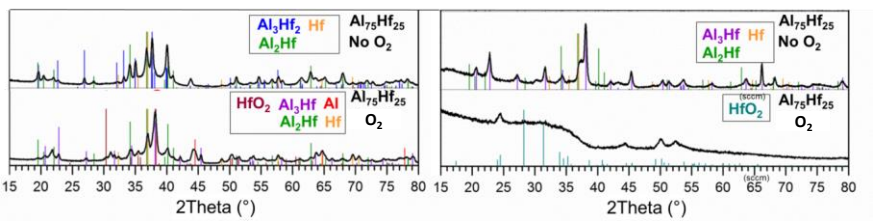
Need: fundamental understanding of this new deposition process

Approach: Combination of characterization, simulation and testing

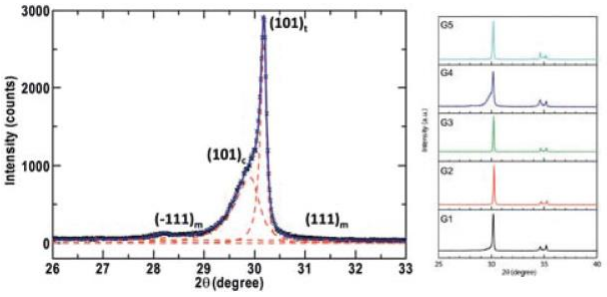


Pure Al and pure Hf

Intermetallics + Oxides



Post-hot isostatic pressing



(rigth) XRD patterns. The composition changes among the samples G1, G4, G5 is clearly visible. Grinding (G4) broadened the base of the tetragonal peak dramatically as expressed by the large amount of pseudo-cubic phase (56 wt%). HIP at 1350 °C after grinding lowered the amount of pseudo-cubic to 10 wt%.

(left) Fitting example for G4 using asymmetric Lorentzian functions with linear background function.

J. Kuebler[1], S.S. Scherrer[2], M. Cattani-Lorente[2], S. Yoon[3], L. Karvonen[3], S. Pokrant[3], F. Rothbrust[4]

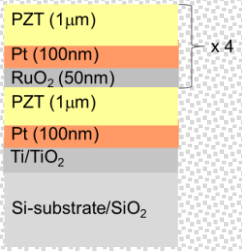
[1]Empa, Swiss Federal Laboratories for Materials Testing and Research Laboratory for High Performance Ceramics Ueberlandstrasse 129, 8600 Dübendorf, Switzerland

[2]University of Geneva School of Dental Medicine Department of Prosthodontics-Biomaterials 1205 Geneva, Switzerland

[3]Empa, Swiss Federal Laboratories for Materials Testing and Research Laboratory for Solid State Chemistry and Catalysis 8600 Dübendorf, Switzerland

[4]Ivoclar-Vivadent Research & Development Schaan, Liechtenstein

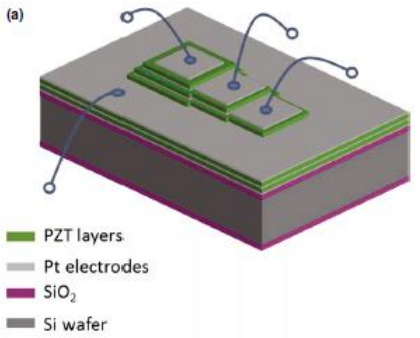
- Thin film multilayers: PZT
  - Piezoelectric materials for switches



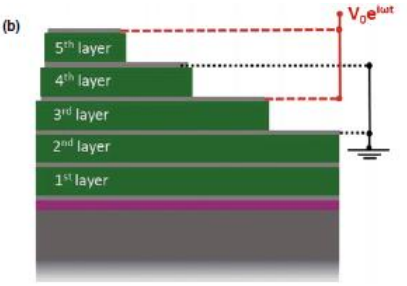
PZT layers by sol-gel deposition.

PZT multilayer systems

The five-layer lead zirconate titanate device after patterning the three topmost electrodes:



3D schematic view

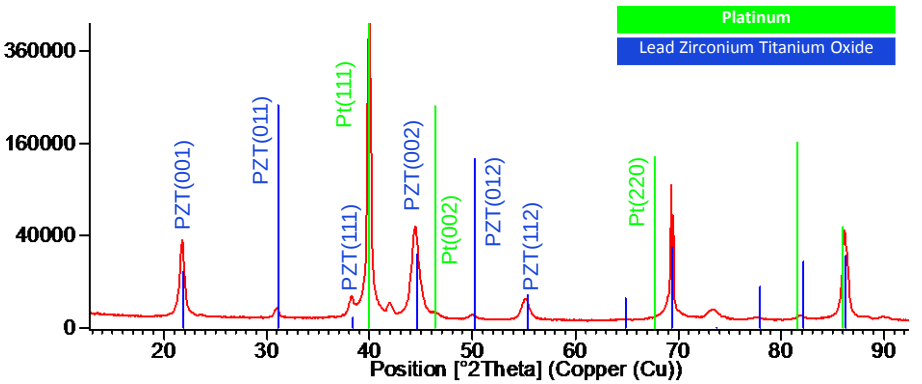


2D schematic of the cross section

J. Am. Ceram. Soc., 97, 2069–2075 (2014)

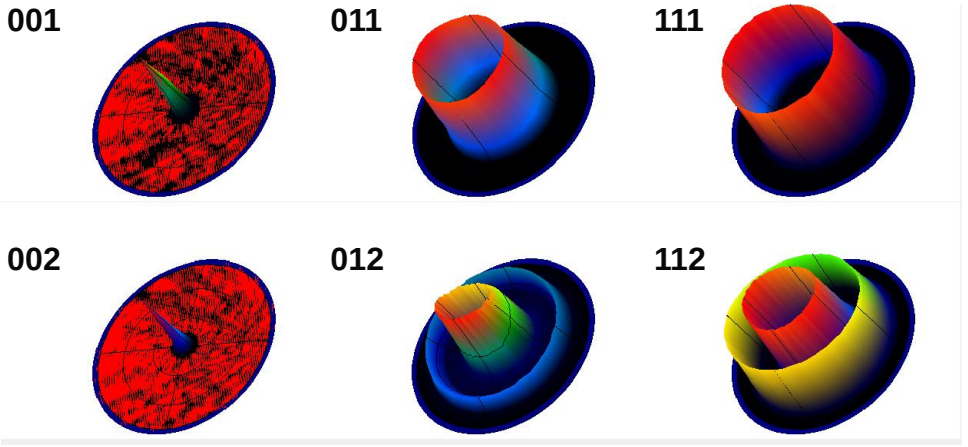
19

Phase analysis



Texture analysis: PZT pole figures

- PZT layers trough **sputtering**:
- Pole figures show the PZT fiber texture, the preferred orientation direction is (001).
- Much higher texturing compared to the sol-gel deposition process.



Texture developpement in multilayers:

- Only small loss in texture.
- Quantification trough texture index.

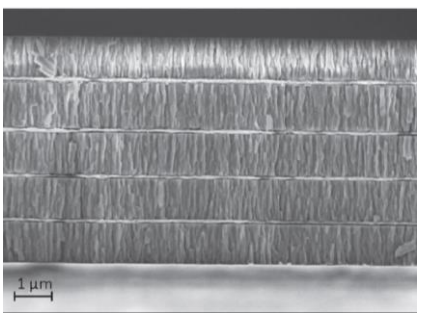
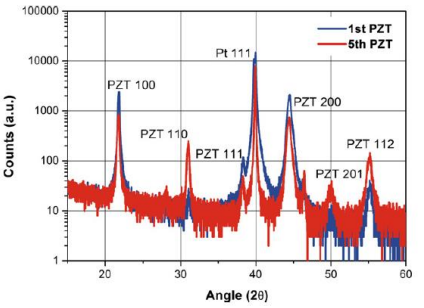


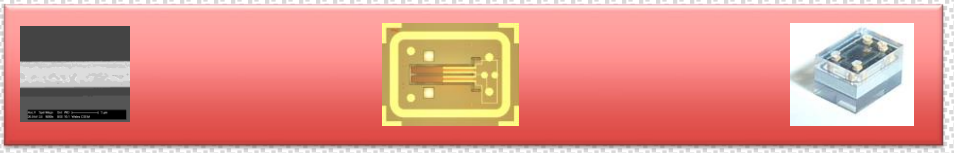
Table II. Texture Index (In Percent) of the Main PZT Peaks for the First and Fifth PZT Layers

Peak	P (PZT1—bottom)	P (PZT1..0.5)
(100) & (001)	86	53
(101) & (110)	0	4
(111)	12	7
(201) & (210)	0	21
(112) & (211)	2	15



J. Am. Ceram. Soc., 97, 2069–2075 (2014)

➢ HRXRD



➢ Epitaxial Systems

Analytical tasks to address for epitaxy films

- ➢ Strain
- ➢ Defects
- ➢ Layer thickness
- ➢ Lateral structure
- ➢ Chemical composition
- ➢ Lattice parameters

Thin film X-ray Diffraction: Geometries

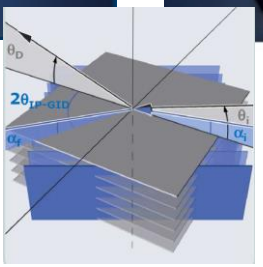
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Out-off-plane diffraction



In-plane diffraction



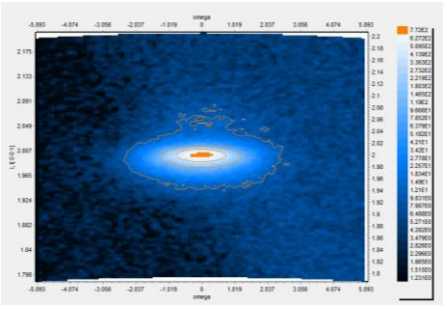


- Coplanar diffraction
  - (hkl) // sample surface

- Non-coplanar diffraction
  - In-Plane GID
  - (hkl) ⊥ sample surface

Thin epitaxial films: Out-off plane BTO(002)

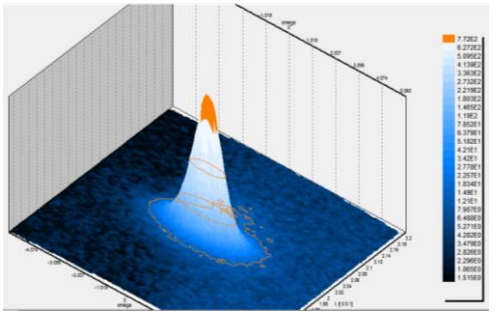
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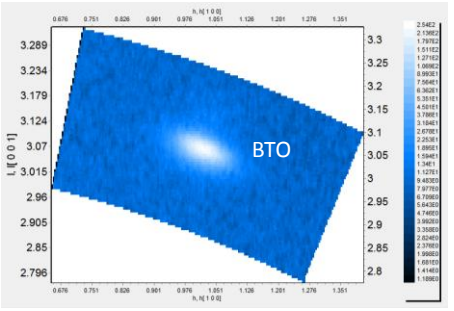
	d-spacing	FWHM	FWHM (RC)
BTO-Si	1.9975	0.45	1.401

$BaTiO_3$: reflection (002) on Si

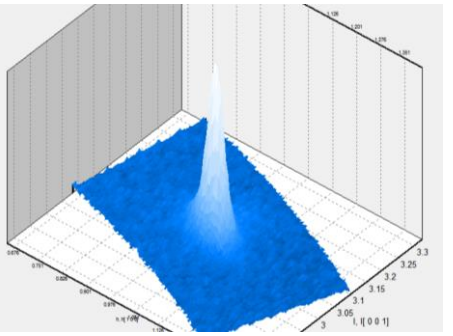
Out-off-plane diffraction



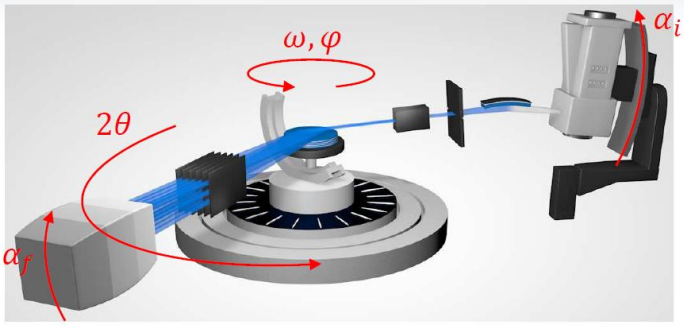
$BaTiO_3$: asymmetric reflection (103) on Si



$BaTiO_3$: reflection (103) on Si
asymmetrical reflection



In plane diffraction:



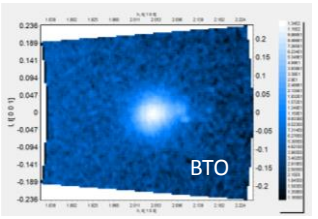
- Use of a 1D-detector rotated by 90° provide resolution perpendicular to the sample surface.

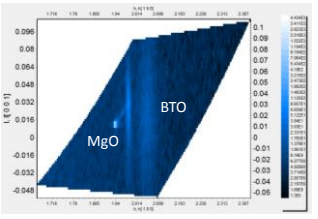
$BaTiO_3$: in-plane BTO(200) on Si and MgO

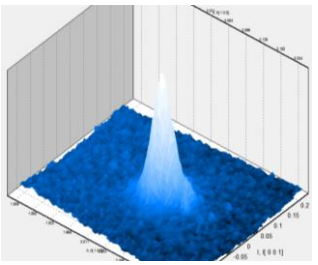
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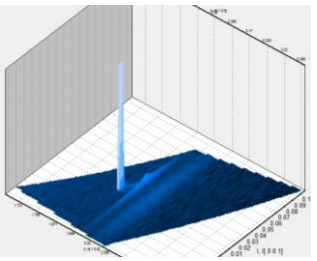
Si substrate

MgO substrate





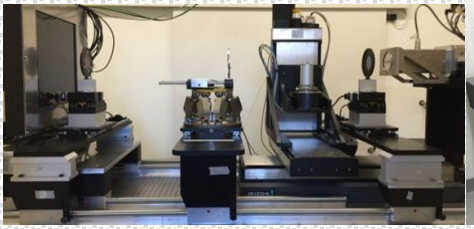


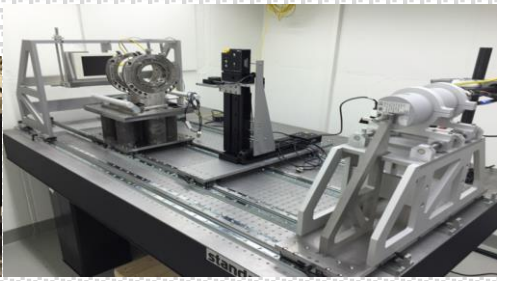


- High mosaicity for BTO on MgO

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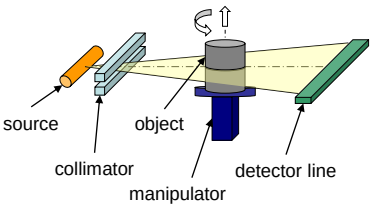
- X-Ray Tomography

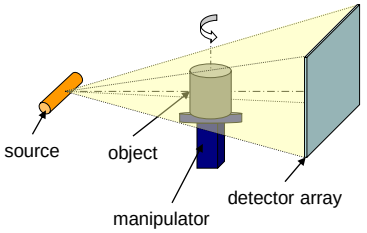




X-RAY CT SETUPS: FAN BEAM & CONE BEAM

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Industrial X-ray CT (450 keV)



Linac X-ray CT (4/6 MeV)



X-ray uCT (160/220 keV)

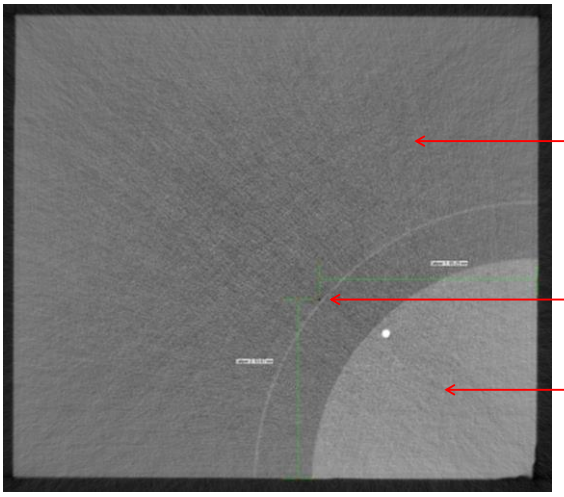


Cone beam CT (450 keV)

High energy X-ray imaging

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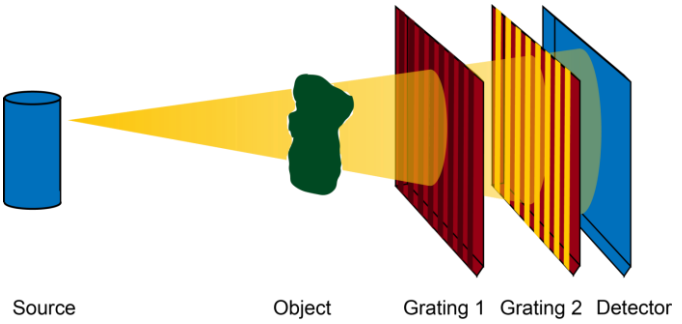
Insulator, region of interest (160 x 140 x 100 mm)



scan parameters:

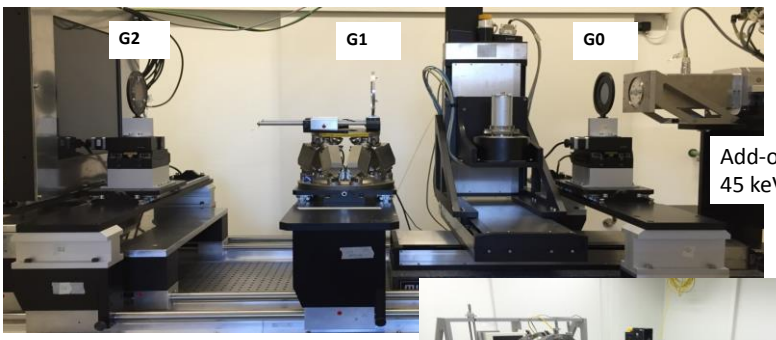
- X-ray source: 450 kV, 3 mA
- Pixel size: 0.15 x 0.15 mm

X-RAY PHASE CONTRAST AND DARK-FIELD IMAGING



- Measurement of X-ray phase-shift and scattering instead of absorption
- Dark field imaging: sensitive to sub-micrometre structures (e.g. pores)
- Virtual Institute for New X-ray Methods in Material Science

TWO XPCI SYSTEMS

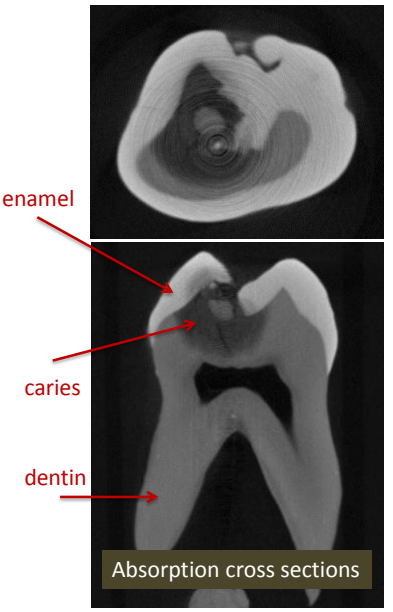


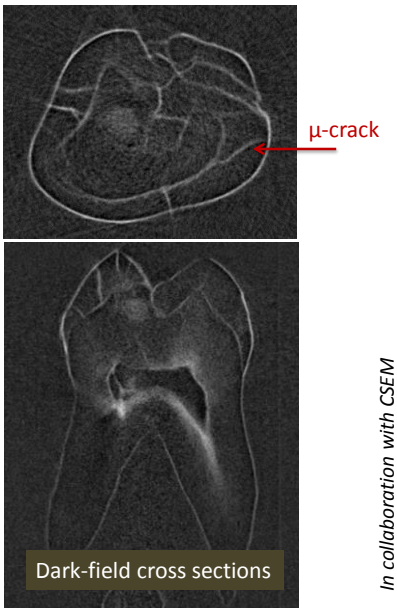
Add-on to µCT
45 keV design energy



New, dedicated setup
35 keV design energy

XPCI Tomography of a tooth





In collaboration with CSEM

Discussion

- **XRD and *in-situ* XRD**
  - support tool for layer deposition process optimization
  - System understanding and layer design
- **HRXRD**
  - For oxide materials and devices
  - Addressing in-plane diffraction for accessing directly in-plane structural parameters: strain, mosaicity
- **X-Ray Tomography**
  - For 3D information of oxide materials and devices
  - High sensitivity of the dark-field mode to small voids.

Curiosity, Flexibility, Communication

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Antonia Neels, Rolf Kaufmann, Tanja Kramer, Thomas Lüthi, Alex Flisch, Felix Reifler, Olga Kuzior, Gelu Rotary, Mathieu Plamondon, Mario Beltran, Kai Zweiacker, Michele Griffa, Yu Liu, Carina Stritt, Fei Yang, Jürgen Hofmann, Alex Dommann, Stefan Hartmann, Zoltan Balogh, Selina Kolokytha, Ramon Schmid, Kim von Allmen.



http://ecm30.ecanews.org/ecm2016/home.html

50 MICROSYMPOSIA
Biology Chemistry
Materials Mineralogy
Physics

PLENARY SPEAKERS


Ada Yonath


Jean-Marie Lehn

KEYNOTE SPEAKERS (to be completed)

Jan Pieter Abraham, Basel University, Switzerland
Bob Cernik, University of Manchester, UK
Birger Dittrich, Georg-August-University Göttingen, Germany
Robert von Dreele, Argonne National Laboratory, USA
Francesca Fabbiani, Göttingen, Germany
Makoto Fujita, University of Tokyo, Japan
Sandra W. Jacob, Novartis Pharma AG, Basel, Switzerland
Sven Lidin, Lund University, Sweden
Gabor Oszlanyi, Academy of Sciences, Budapest, Hungary
Simon Parsons, University of Edinburgh, UK
Werner Paulus, Montpellier, France
Martin U. Schmidt, University of Frankfurt, Germany
Peter Schurtenberger, Lund University, Sweden