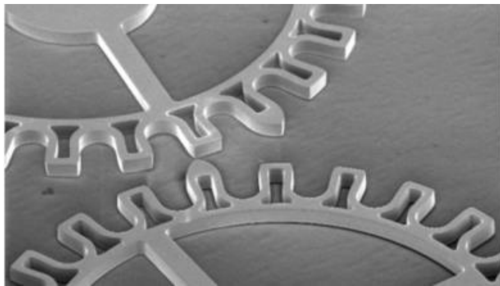
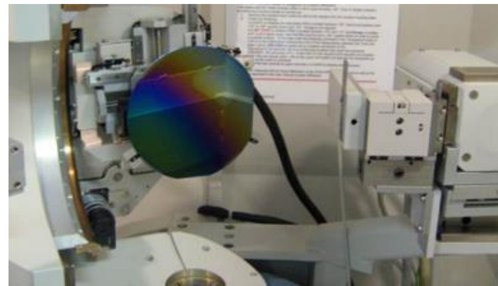
Advanced materials characterization using state-of-the-art microscopy and XRD techniques focusing on the relationship between micro-structural and mechanical properties

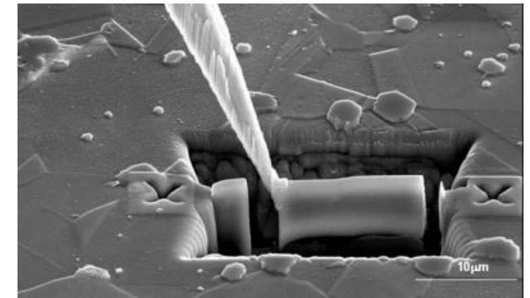
EIROforum WAMAS, CERN, 20th of November 2013

Laboratory for X-Ray Applications & Microscopy, Microsystem Technology

X. Maeder, A. Neels, M. Dadras, O. Sereda, M. Garganourakis, T. Bandi, M. Lebeouf, S. Biselli





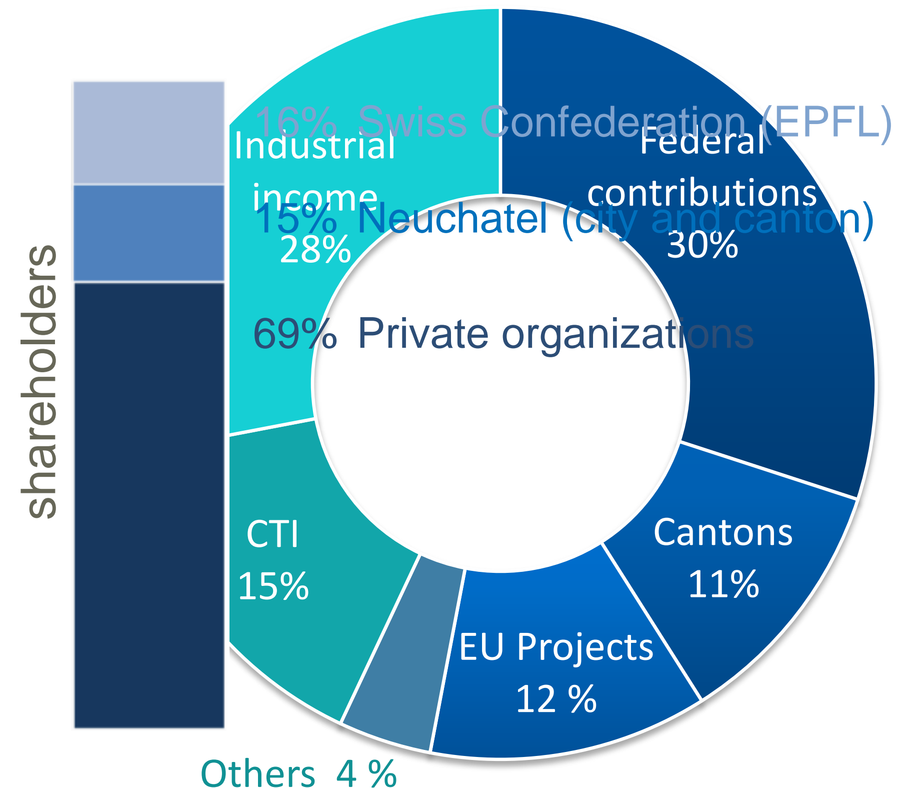


CSEM at a glance

- Incorporated, not-for-profit ***Research and Technology Organization (RTO)***, supported by the Swiss Government

- **A public-private partnership**
  - 31 % public
  - 69 % private

- **Key figures (2012)**
  - Revenues ~ CHF 70.0 mio
  - Employees ~ 400



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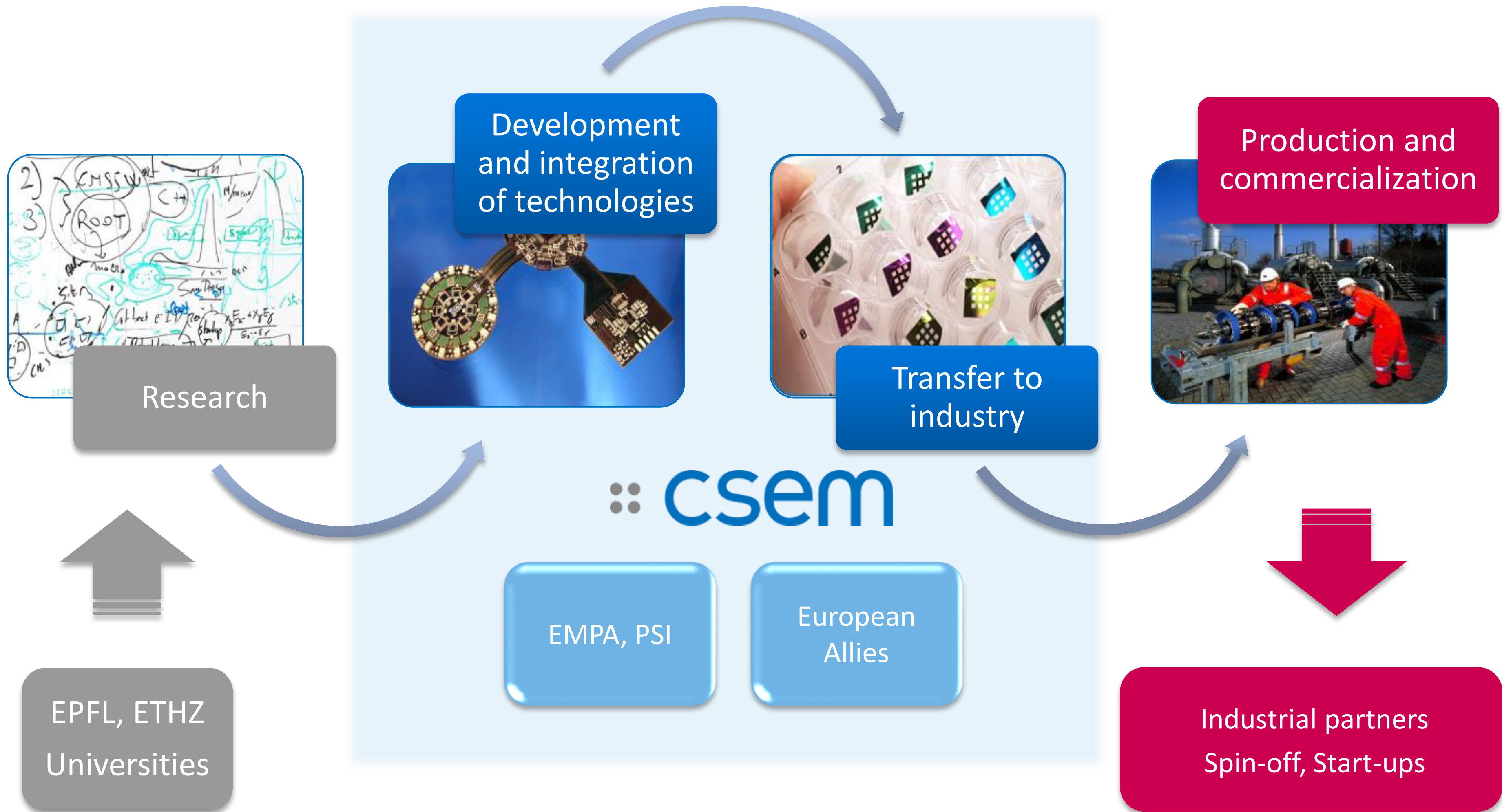
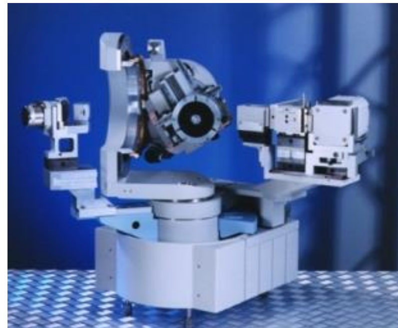
CSEM's positioning



Closer to industry …

Reliability-Analytical Laboratory @ CSEM

Système de Management Certifié SQS ISO 9001



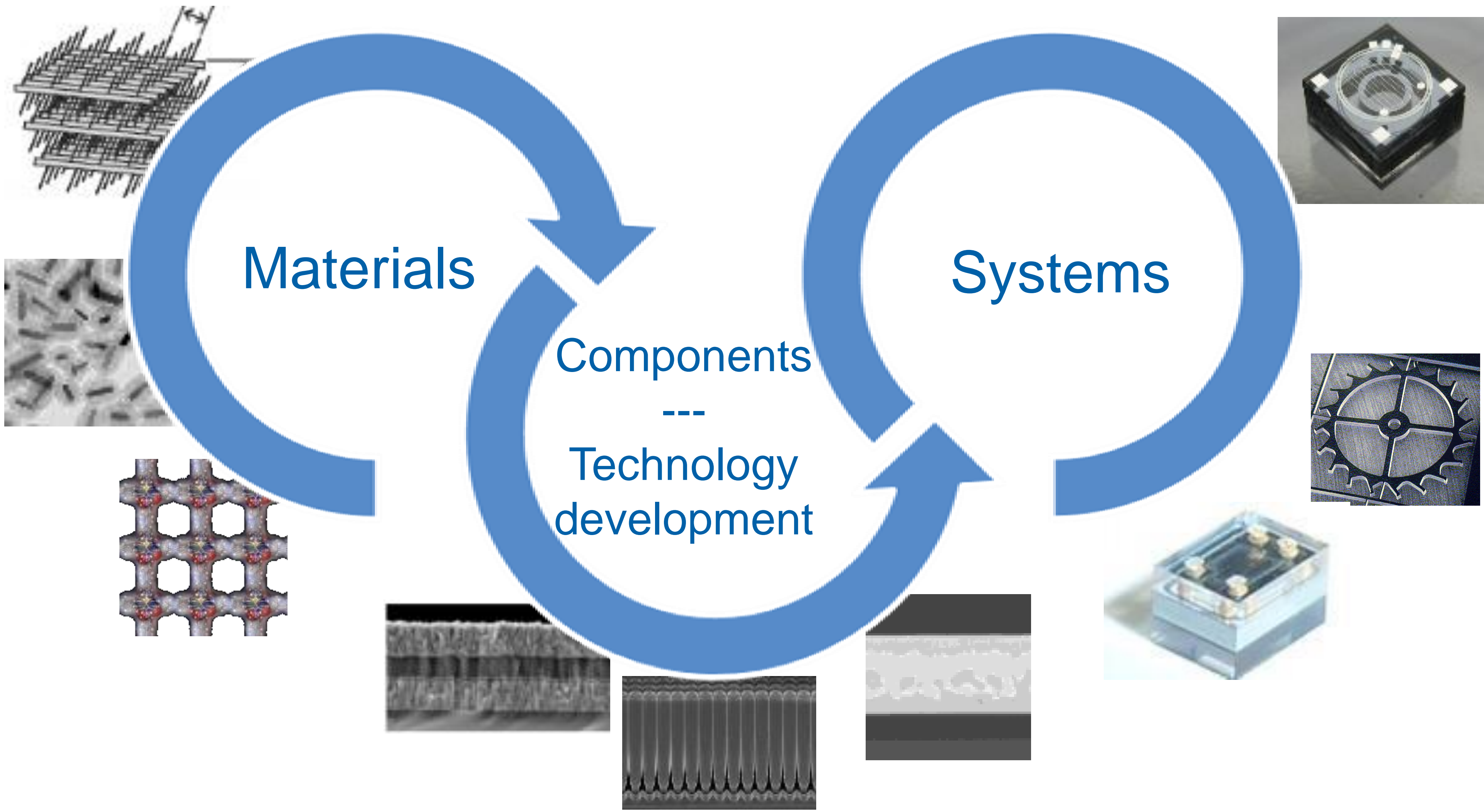
Two Units with complementary competencies

- **SMN: Service Micro & Nanoscopy**
- **XRD: X-ray Diffraction**

Our objectives:

- Technology support & services for our clients
- Establishment of new qualification methods
- Development of new analytical tools for process support
- Reliability and Testing of Microsystems

Our Domain of Activities



Technology Development Support

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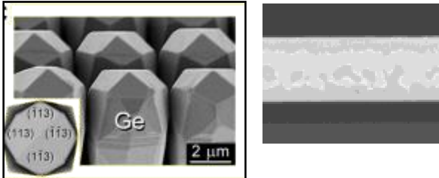
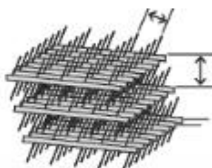
Materials & Components

- Semiconductors & intermetallics



- Oxide & nitride coatings

- Organic thin films



- ….

Properties

- Structural

XRD methods & Microscopy

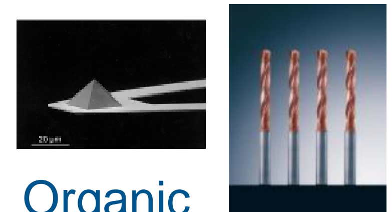
In-situ techniques:
- Temperature,
- Humidity,
- Reactions,
- Mechanical forces

- Mechanical
- Electrical
- ….

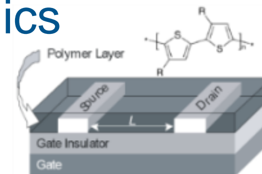
Systems / Application

- Microsystems

- Tools



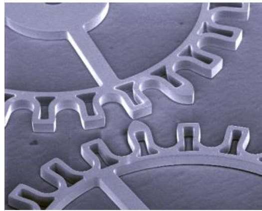
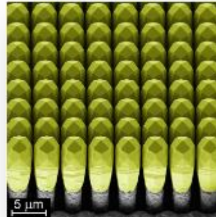
- Organic electronics

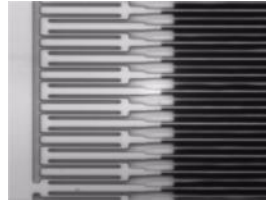
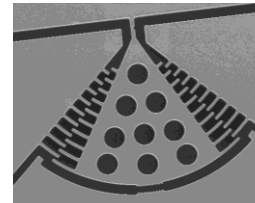
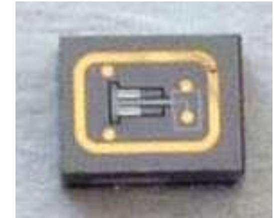


- ….

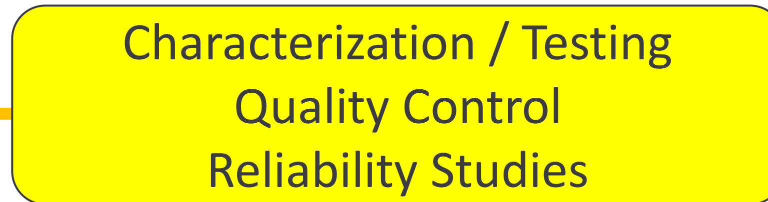
Technology Development Support

Qualification of subcomponents, components and devices

Concept Phase → Design Phase → Components Fabrication → Components Assembly & Packaging → Micro-system



Micro and Nanoscopy

Philips CM200
(Resolution 0.14 nm)

Philips XL30 ESEM-FEG , Resolution 2nm

Nanoscope ICON

-EDS
-EBSD
-insitu deformation testing
-several cooling and heating stages

XRD Application Laboratory

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PANalytical X'Pert PRO MPD; MRD; STOE STADI P; IPDS I; IPDS II

Coatings: Metal oxides, nitrides
Thin films: Molecular self-assembling organics
Interfaces: Joining materials
Microsystems: Quality Control and Reliability

Analysis:

- Phases
- Stresses (HRXRD)
- Defects (HRXRD)
- Texture
- Grain sizes and distributions

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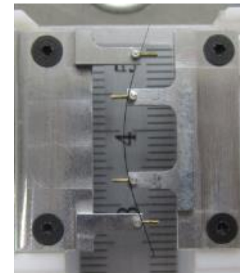
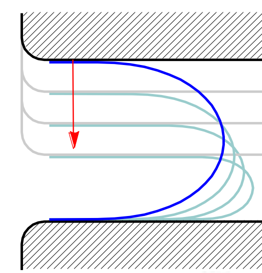
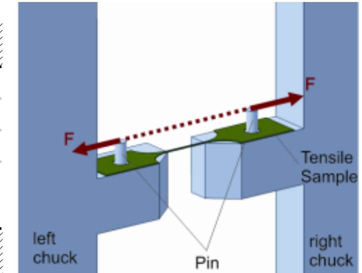
Reliability and Testing of Microsystems @ CSEM

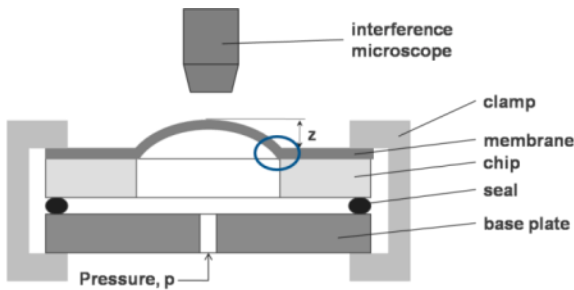
Materials Analysis: Stresses & Defects, Mechanical behavior, Microstructural properties

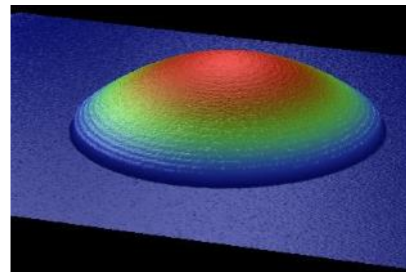
- ✓ Elastic modulus
- ✓ Poisson's ratio
- ✓ Fracture/failure mechanisms
- ✓ Fracture toughness
- ✓ Electrical properties (migration,etc.)
- ✓ Interfacial strength
- ✓ Coefficient of thermal expansion(CTE)
- ✓ *Crystal structure*
- ✓ *Grain size*
- ✓ *Surface roughness*
- ✓ *Doping*

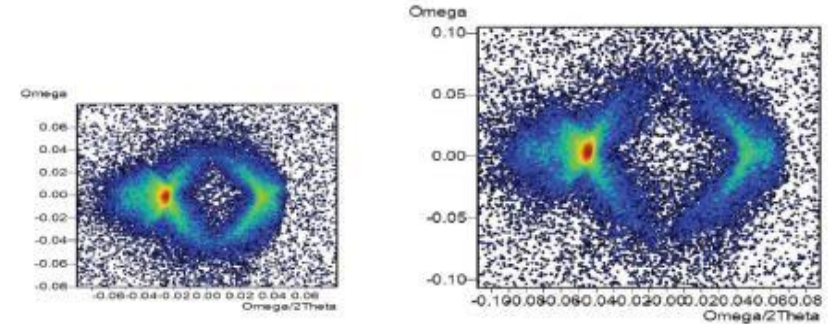








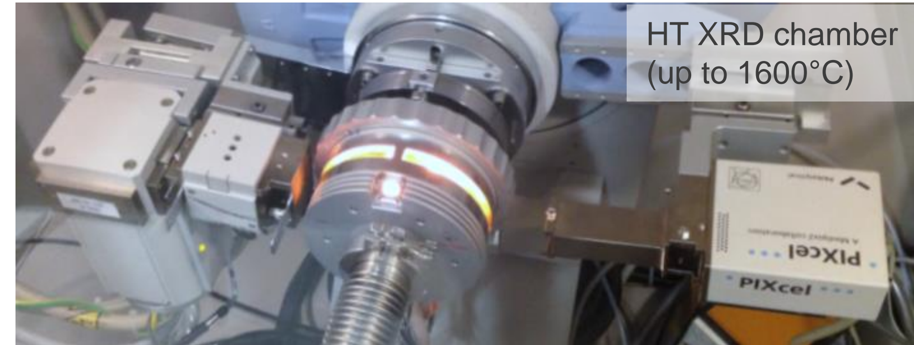




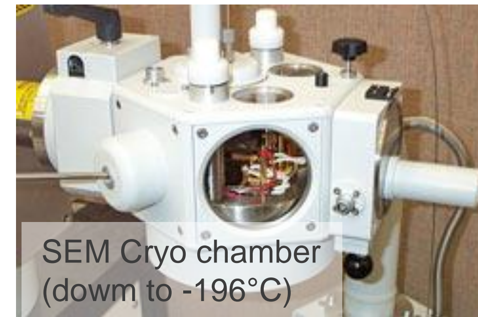
Reliability and Testing of Microsystems @ CSEM

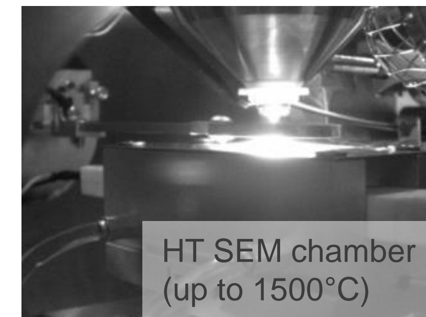
Environmental Effects

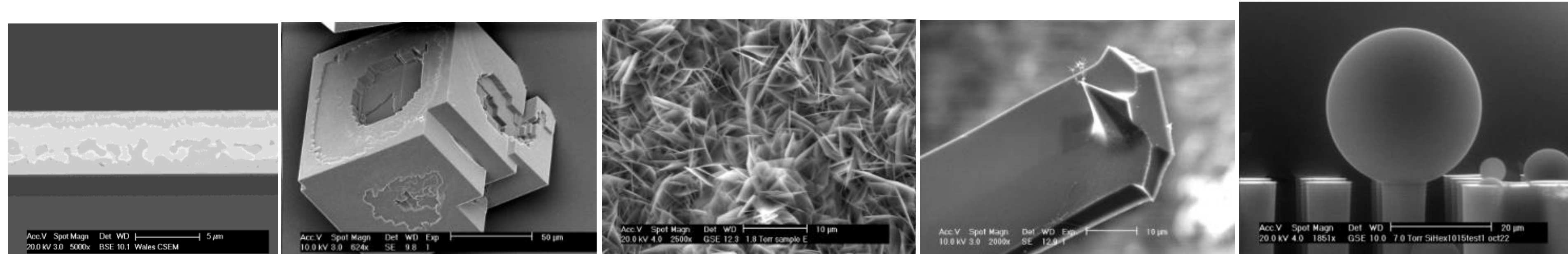
- ✓ Storage, humidity effects
- ✓ Radiation tolerance
- ✓ Chemical exposure effects
- ✓ Biocompatibility
- ✓ Effects of extreme heat or cold
- ✓ Effect of shock



HT XRD chamber (up to 1600°C)


SEM Cryo chamber (dowm to -196°C)


HT SEM chamber (up to 1500°C)



Reliability and Testing of Microsystems @ CSEM

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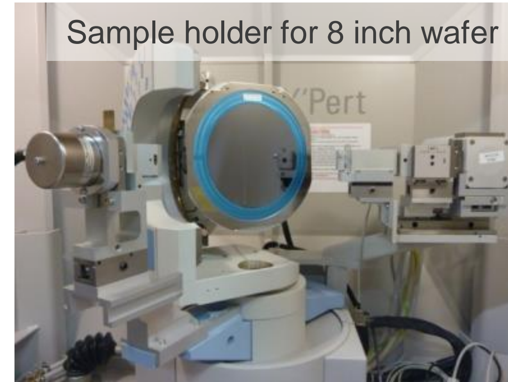
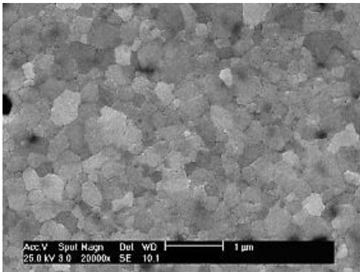
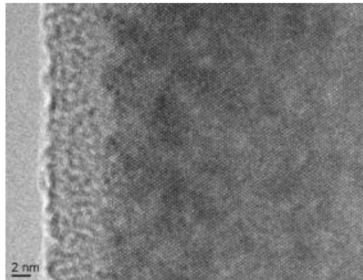
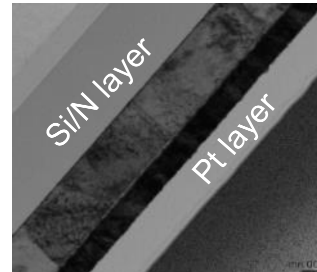
Device-Level Metrology

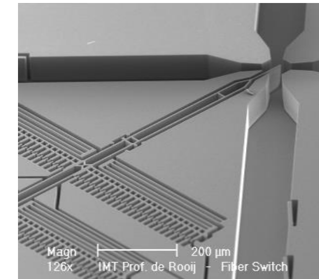
- ✓ *High resolution cross-sectioning*
- ✓ *Microscale crack propagation visualization*
- ✓ *Real time performance (movement) visualization*
- ✓ *Device design effects (corners, etch holes, etc.)*

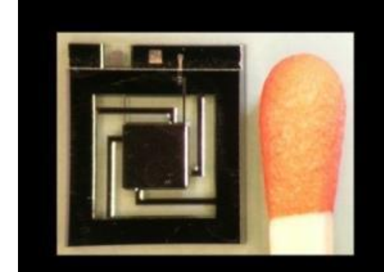












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Reliability and Testing of Microsystems @ CSEM

Device-Level Metrology

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Fabrication:

1) Si structuring: DRIE

Defects

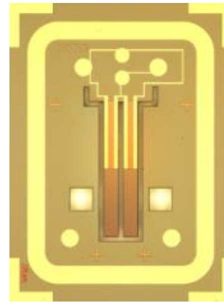
2) Layer deposition: AlN, …

Texture

Phase Analysis

Crystallite Sizes

Layer stress

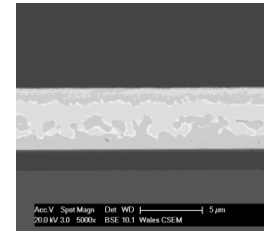


MEMS resonator

Packaging:

3) Sealing at 320°C

In-situ T-dependent XRD studies



Packaging Stress

Defect Mobility

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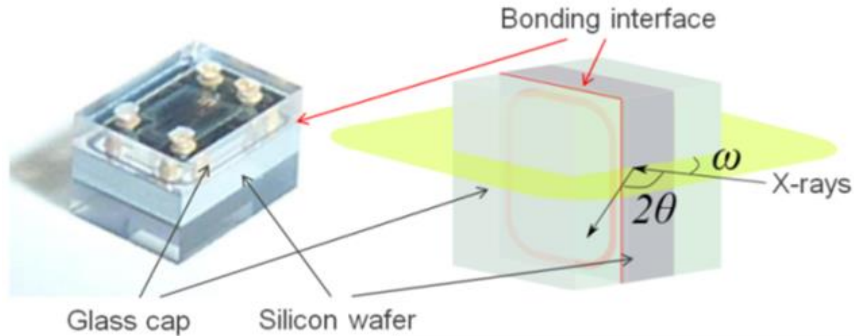
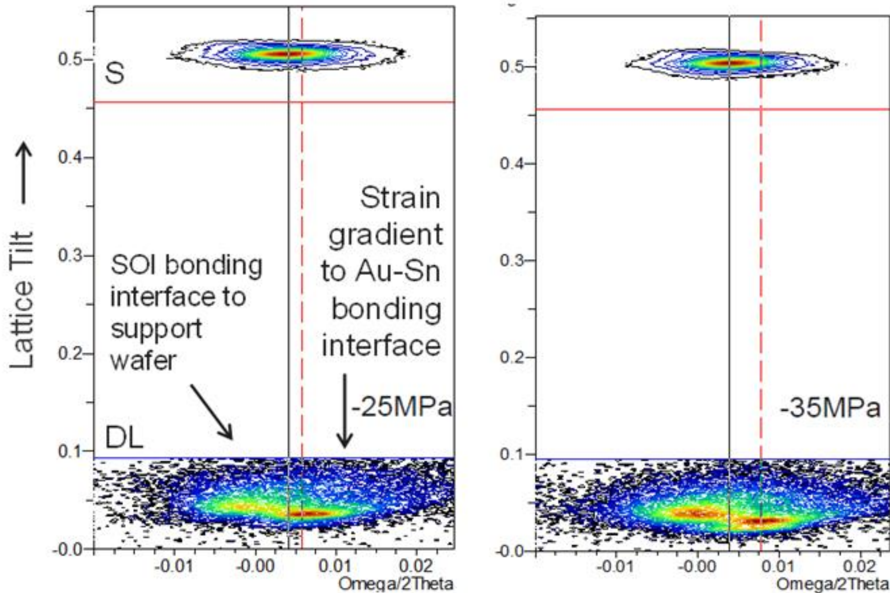
Device-Level Metrology

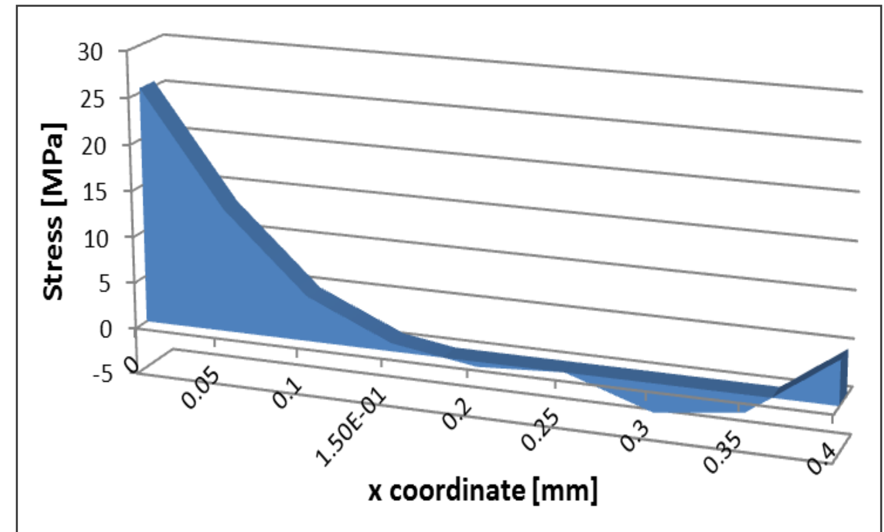


Packaged device

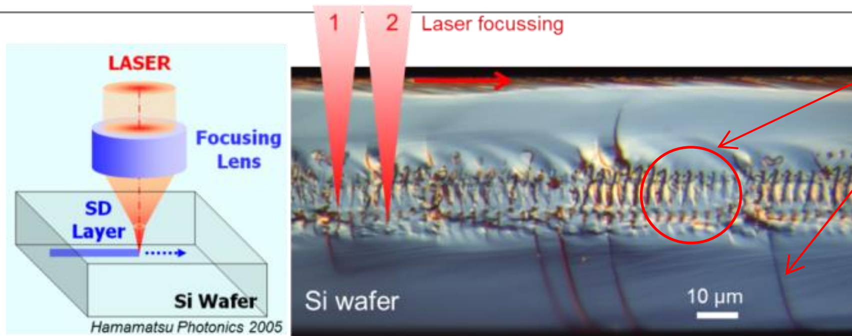
Device without cap



Bonding interface



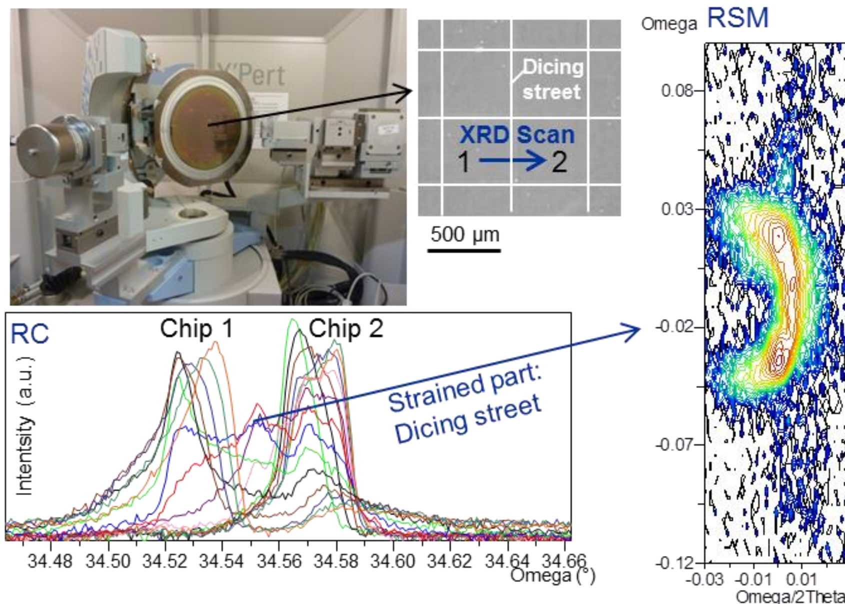
Qualification of Laser Processes for Chip Separation using HRXRD and Micromechanical Testing



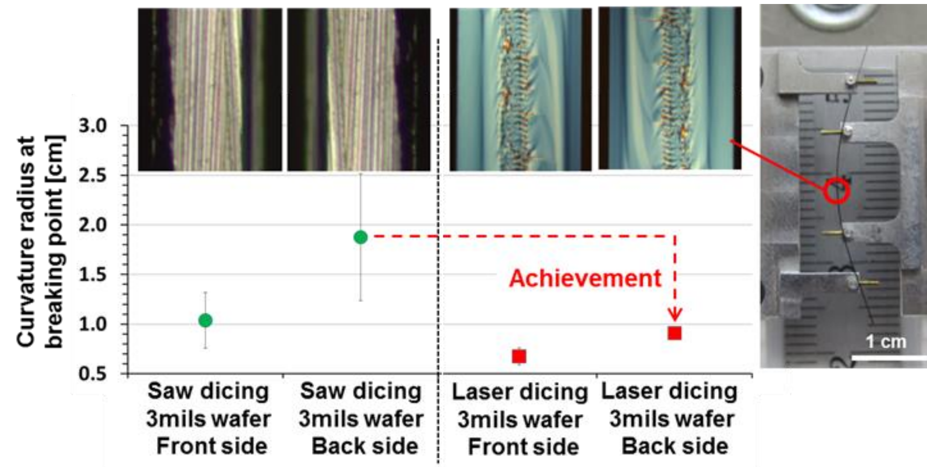
- **High defect zone and amorphization of Si**
- **Radial fractures**

How much do these defects affect the quality of the Si and therefore the reliability of the chip? What are the best laser parameters to minimize these defects?

Strain analyses with high resolution XRD



Micromechanical testing



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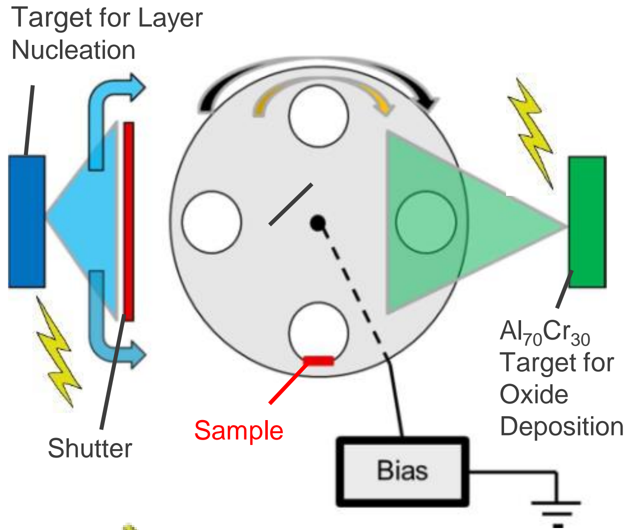
Thin film deposition and characterization:
Ex: Wear Resistant Hard Coatings

Reactive Cathodic Arc Deposition: Phases, Phase, transformations at HT, Texture, Stress, Grain size?



EMPA Materials Science & Technology

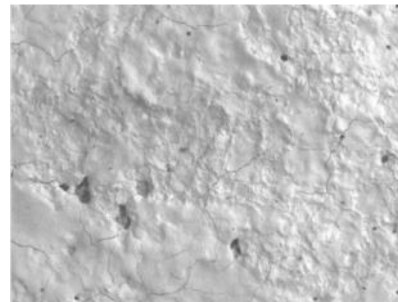
oerlikon balzers

ETH Eidgenössische Technische Hochschule Zürich
Swiss Federal Institute of Technology Zurich

Pure Al and pure Hf

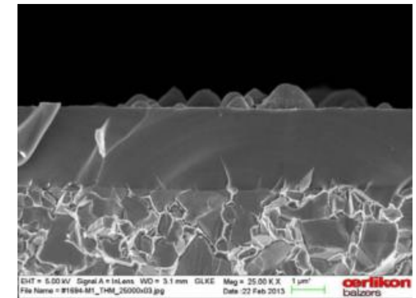
Cathodic arc process

O_2



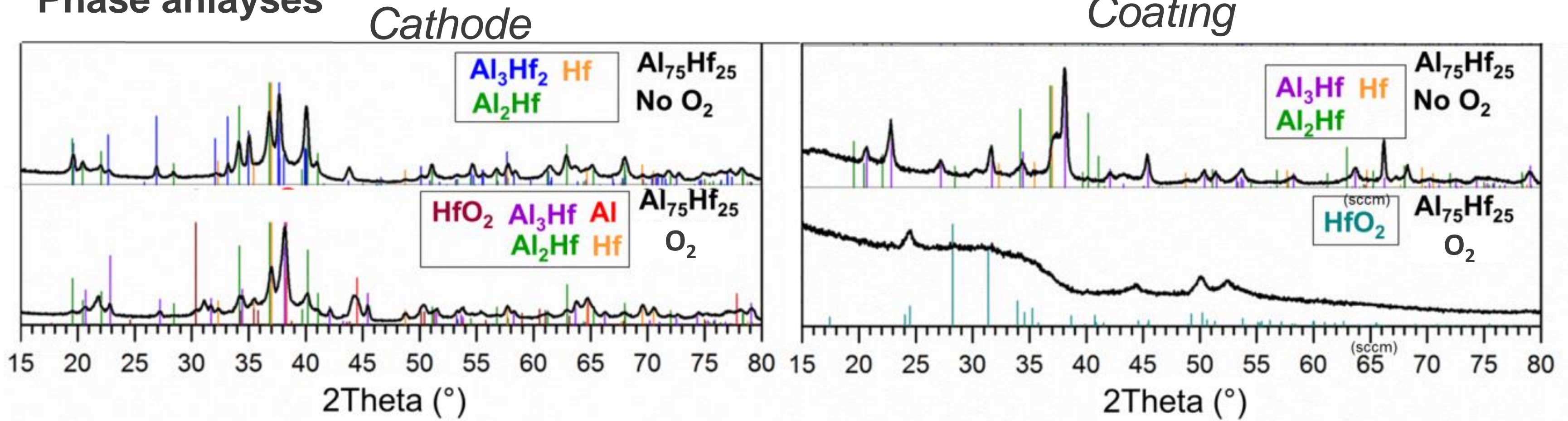
Intermetallics + Oxides

Deposition

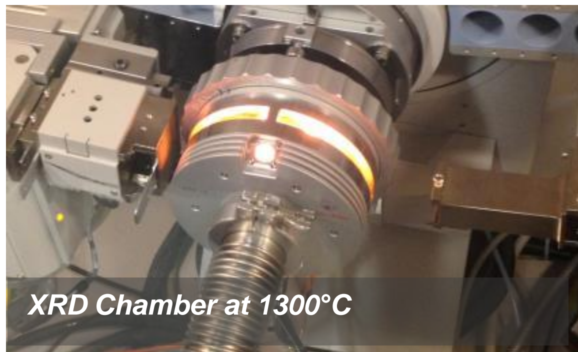


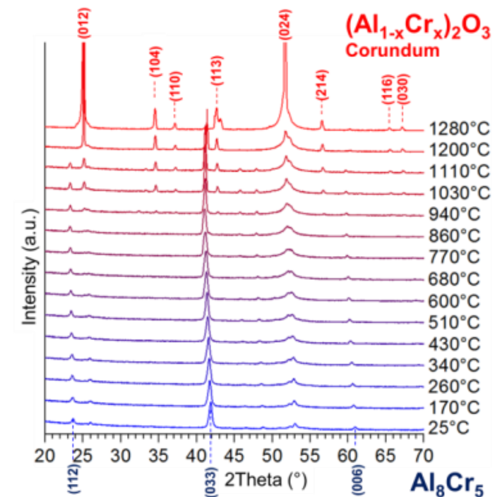
Thin film deposition and characterization:
Ex: Wear Resistant Hard Coatings

Phase anlayses



Insitu high temperature studies


XRD Chamber at 1300°C



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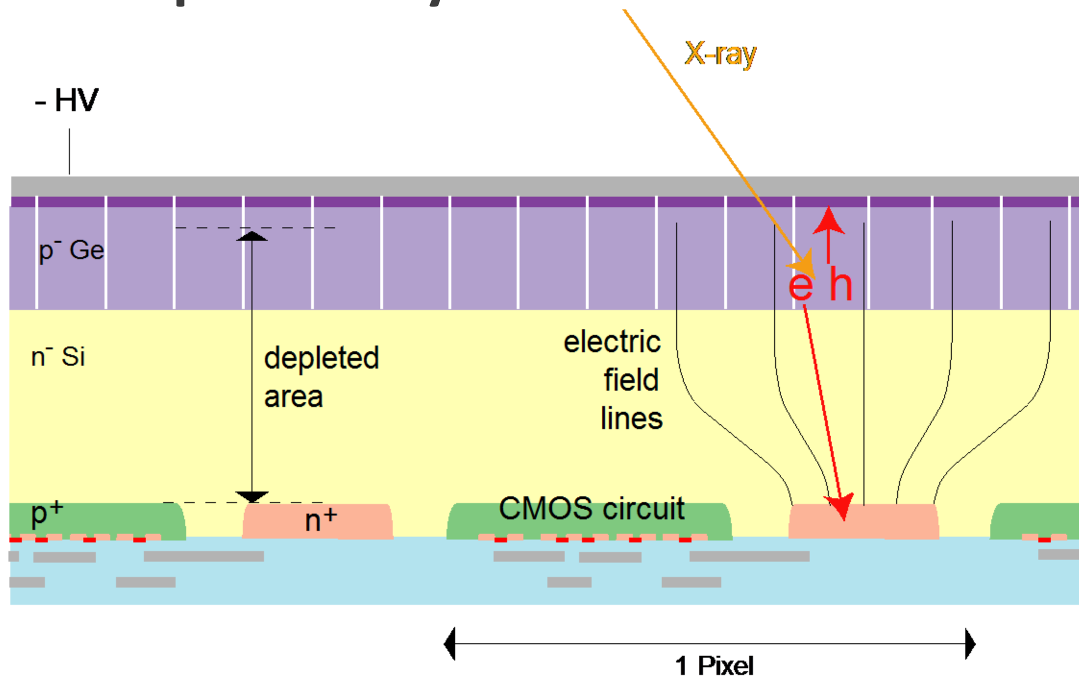
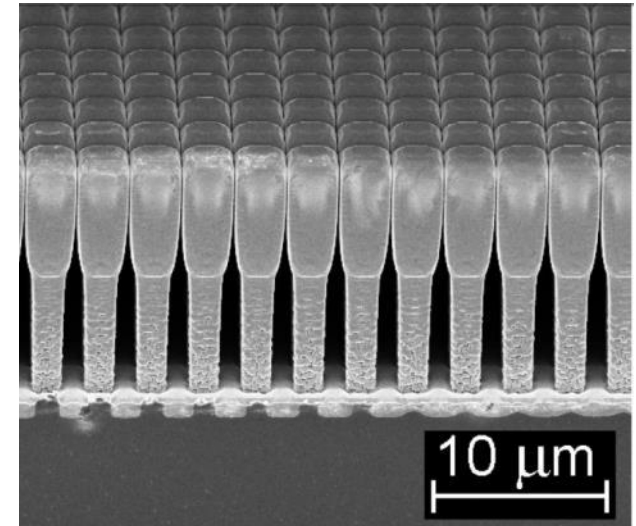
New Types of X-ray detection systems

Concept of X-ray Detector:





- Thick Ge layer (50 – 150 µm)
- Epitaxially grown with LEPECVD on backside of CMOS wafer
- Ge absorption layer is hence monolithically integrated
- No bump-bonding needed

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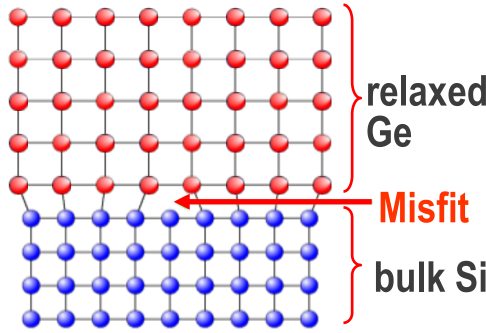
New Types of X-ray detection systems

NEXRAY

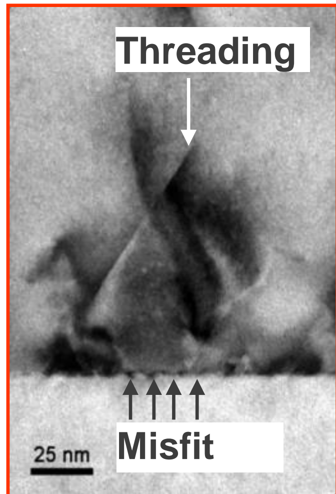
nano-tera.ch
swiss scientific initiative in health / security / environment systems

Challenge

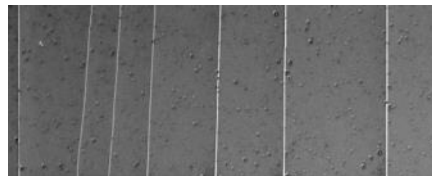
Innovation

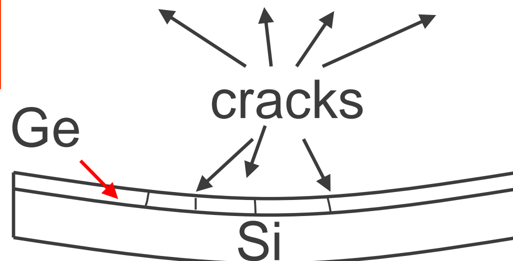


- Lattice mismatch (strain = 4.2 %)
- Mismatch of thermal expansion coefficients.

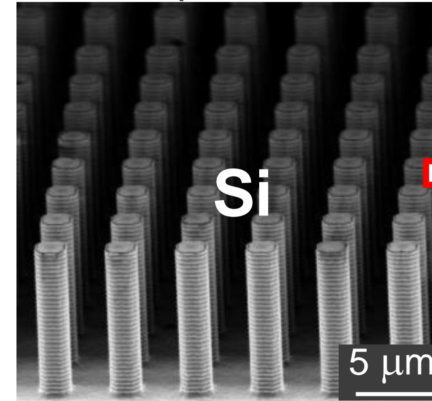


⇒ High density of misfit (MD) and threading (TD), wafer bowing and cracks

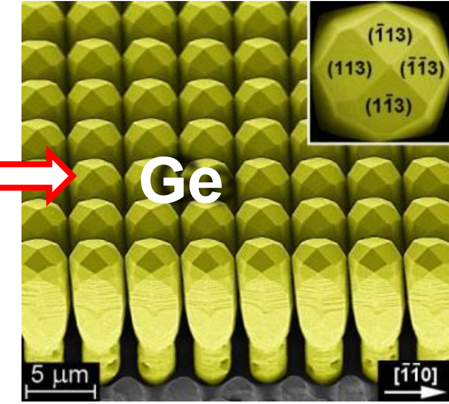




Micromachined Si pillars



Epitaxial Ge pillars on Si



Low Energy Plasma Enhanced Chemical Vapor Deposition (LEPECVD) of Ge on Si pillars

Falub et al. Science 2012

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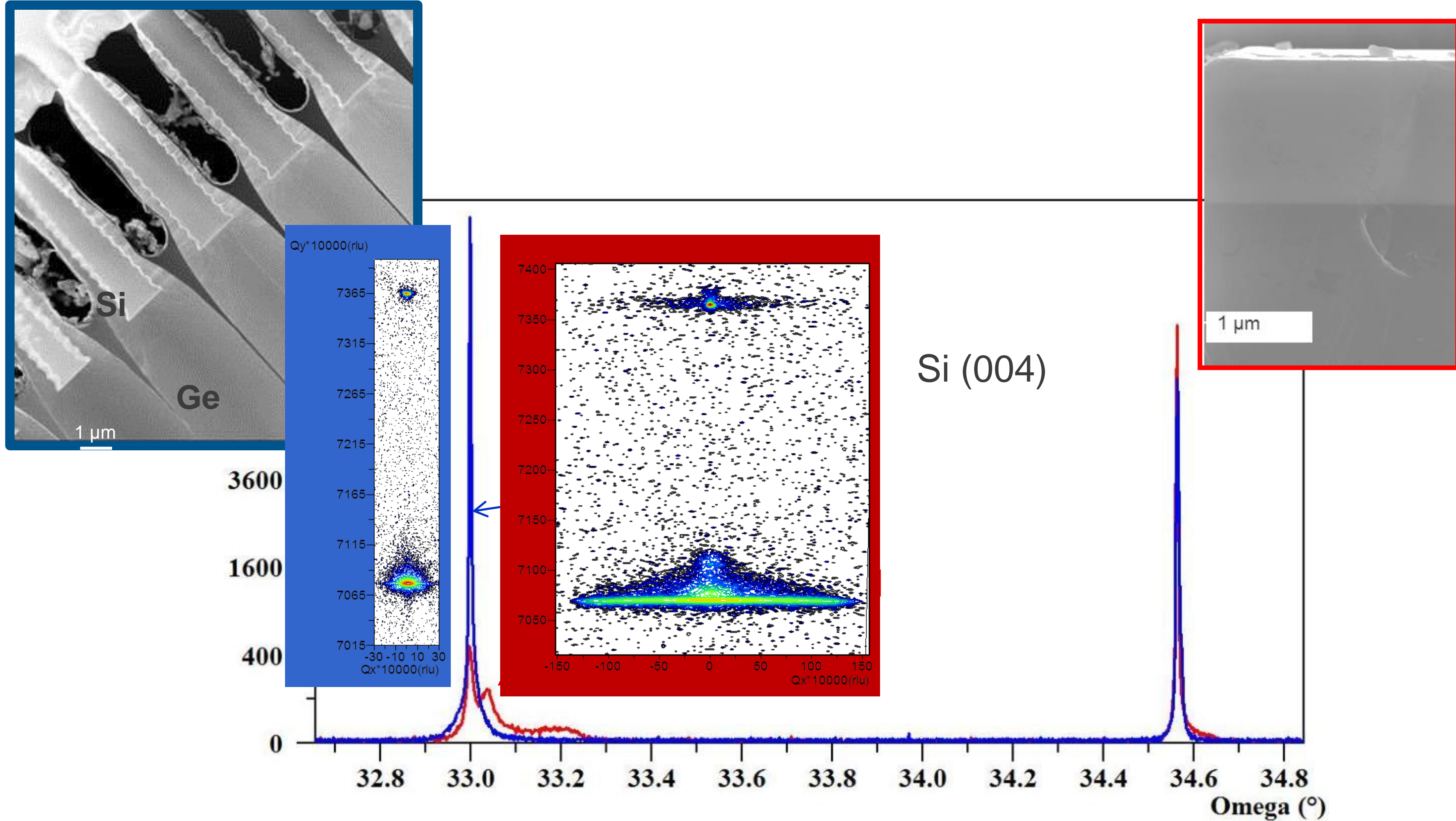
Strain analyses by HRXRD



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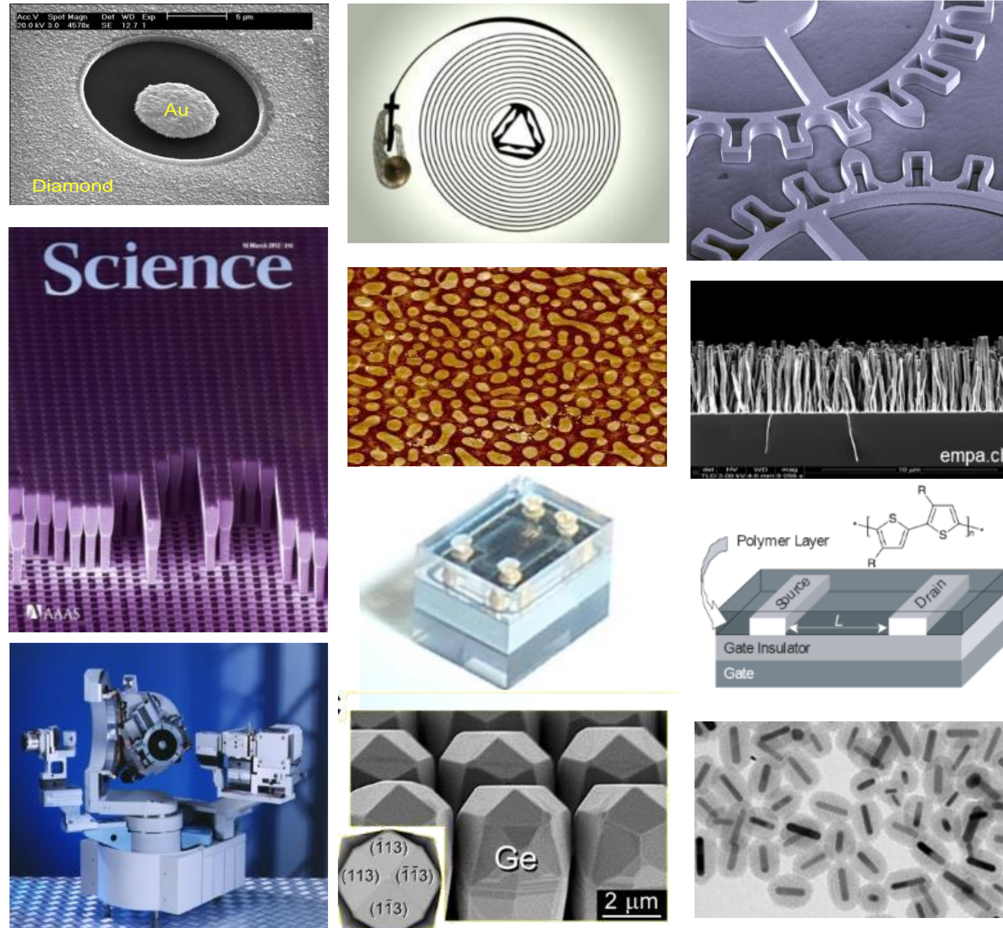
Technology development support :



Thank you for your attention