RÅC Perspective Workshop – Photon Science
Hamburg 9/6, 2017

In situ and *in operando* high-energy X-ray characterization of hard protective tool coatings

Jens Birch
Thin Film Physics division
Linköping University

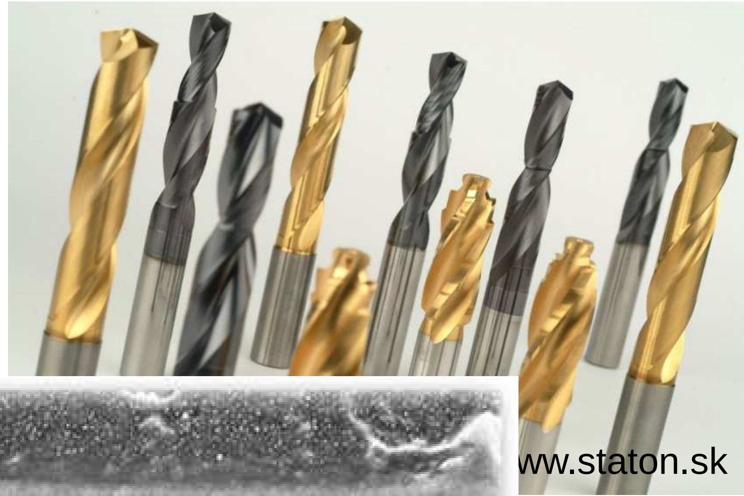
Norbert Schell
High Energy Materials Science Beamline
Helmholtz-Zentrum Geesthacht

LINKÖPINGS UNIVERSITET

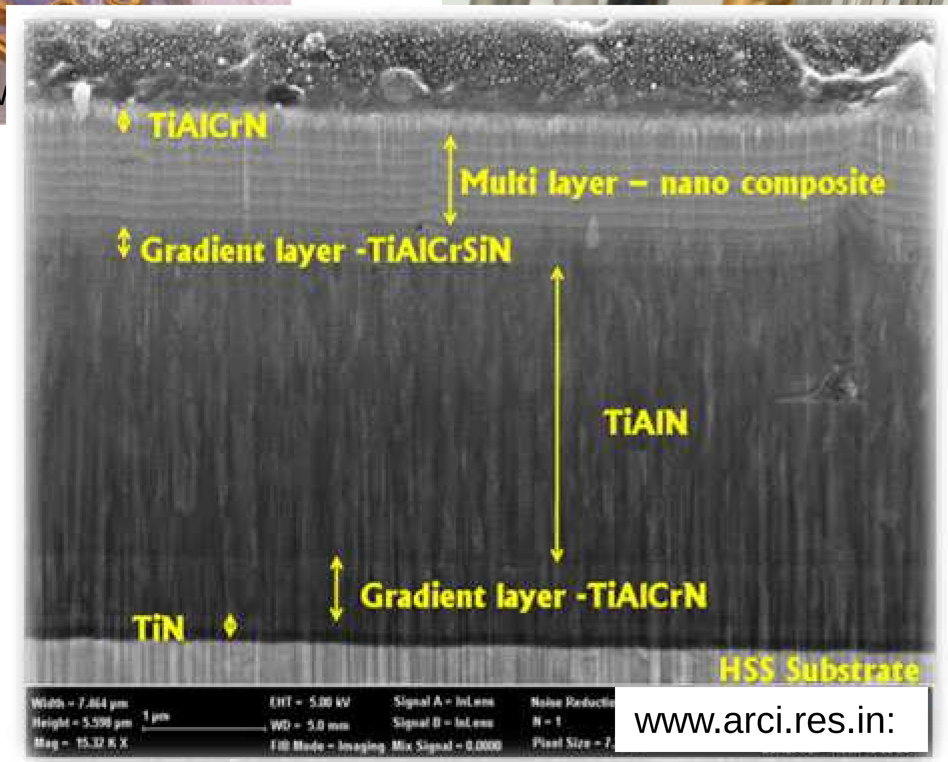
Helmholtz-Zentrum Geesthacht
Zentrum für Material- und Küstenforschung

Coated cutting tools
Important for Sweden & Germany!

Cutting inserts



and drills



LINKÖPING UNIVERSITY

Coated cutting tools
Important for Sweden & Germany!

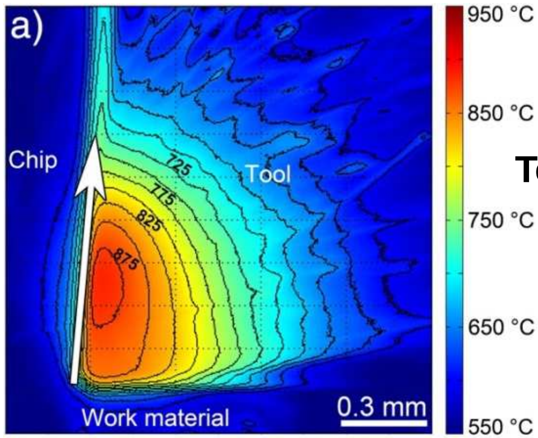
Cutting inserts

Mills and drills

High speed dry cutting

Higher productivity
No cooling liquids

Hotter tools!



Temperature distribution on cutting tool insert during cutting

Prof. Jens Birch, Thin Film Physics division
Linköping University

The "X-Cut" project

"Materials Science of High Performance Cutting Tool Coatings by use of *in-situ* High Energy X-ray Scattering"

Beam Line P07 at Petra III - "High Energy Materials Science" HEMS

Materials Science of Thin Films

Cutting Tool Manufacturer

Money

Helmholtz-Zentrum Geesthacht
Zentrum für Material- und Küstenforschung

Röntgen Ångström Cluster

Prof. Jens Birch, Thin Film Physics division
Linköping University

The "X-Cut" project

In-situ high energy X-ray scattering during the Birth and Death of Cutting Tool Coatings

Beam Line P07 at Petra III - "High Energy Materials Science" HEMS

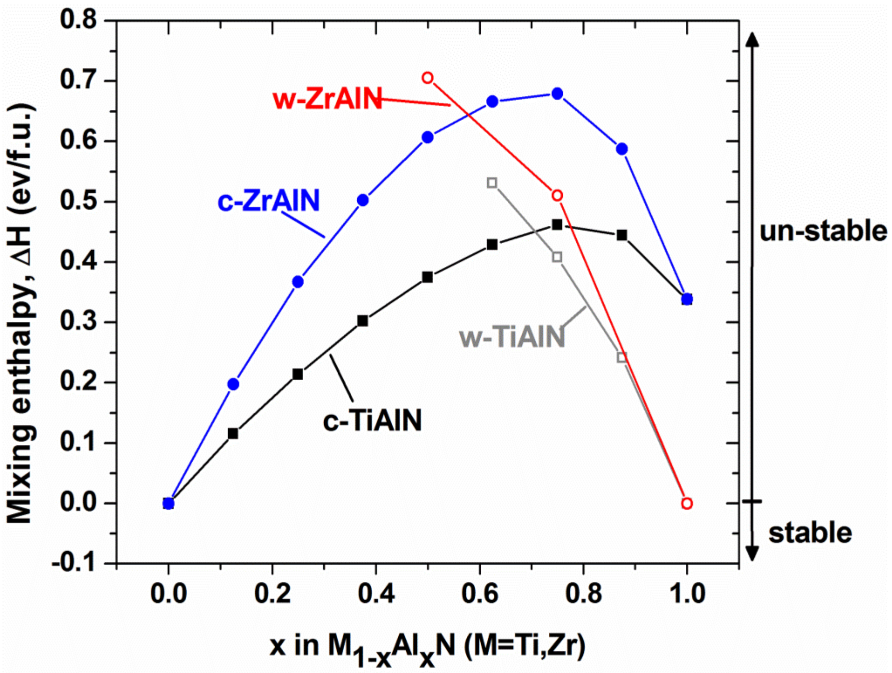
Materials Science of Thin Films — Linköping University

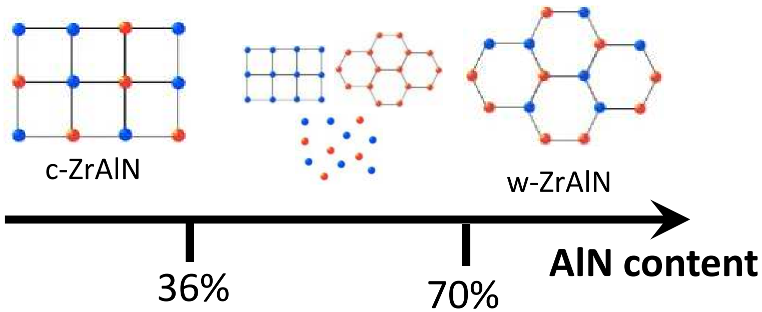
Helmholtz-Zentrum Geesthacht — Zentrum für Material- und Küstenforschung

Cutting Tool Manufacturer — SECO

Money — Röntgen Ångström Cluster

Stability of metastable hard coatings





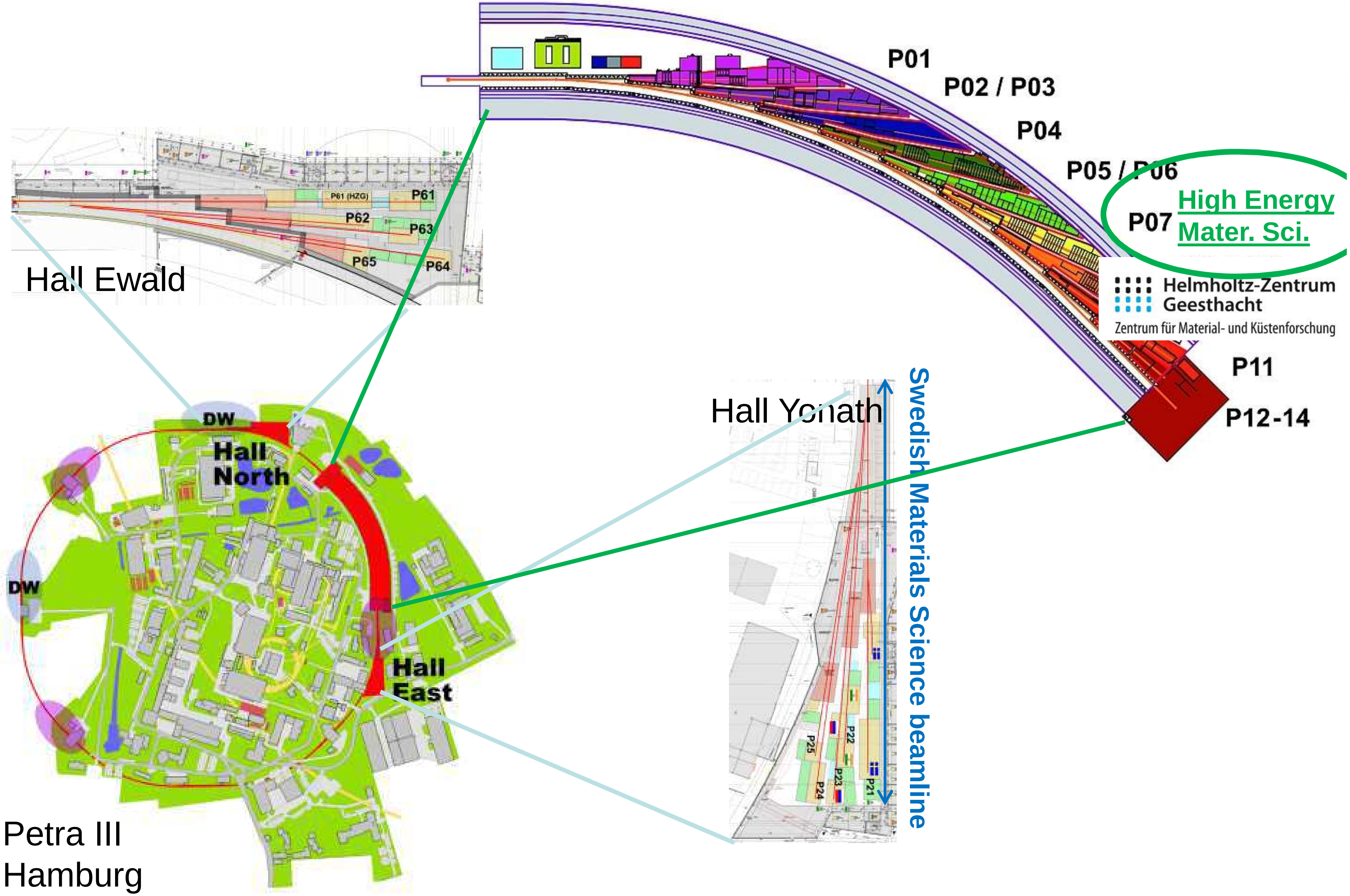
Future's workhorse hard coatings
ZrAlN?
TiNbAlN?
CrTiAlN?
...
Formation?
Phase transitions?
Coating stability?
Substrate interactions?

Alling *et al.*, Appl. Phys. Lett. **95** (2009) 181906.
L. Rogström *et al.*, J. Vac. Sci. Technol. A 30 (2012) 031504.

LINKÖPING UNIVERSITY

More info lina.rogstrom@liu.se

High Energy Materials Science Beamline at Petra III



Advantages of High Energy X-rays for Materials Science

- **High penetration depth**
  - ✓ Non-destructively bulk properties measurable
  - ✓ Deeply buried structures accessible
  - ✓ Easy to design special-purpose in-situ equipment (growth, catalysis, HP, strain, …)
- **Large Ewald's Sphere in scattering experiments**
  - ✓ Small Bragg angles (5° to 15°) → complete diffraction rings @ area detectors
  - ✓ Wide-angle- and Small-angle X-ray scattering (WAXS & SAXS) simultaneously
  - ✓ Large range of lattice planes can be probed simultaneously
- **Focussing to spot sizes in nm range possible**
  - ✓ High spatial resolution
  - ✓ Combination of high penetration depth and high flux
  - ✓ Very short data acquisition times possible
- **High intensity**
  - ✓ Time-resolved data → 2D area detector read out at 0.1-100 Hz
  - ✓ Non-destructive observation of dynamic processes

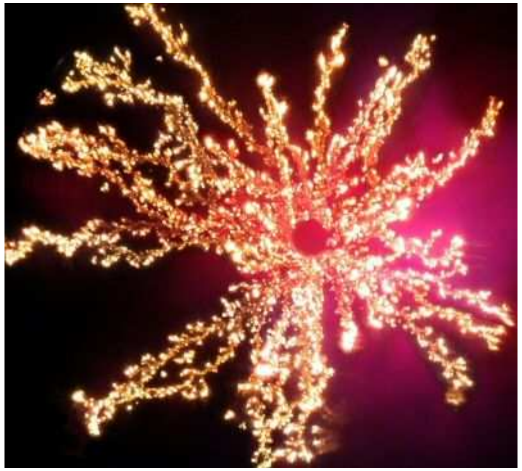
Birth of the coatings

- HT Chemical Vapour Deposition - CVD

- Physical Vapour Deposition – PVD

- Cathodic Arc Deposition

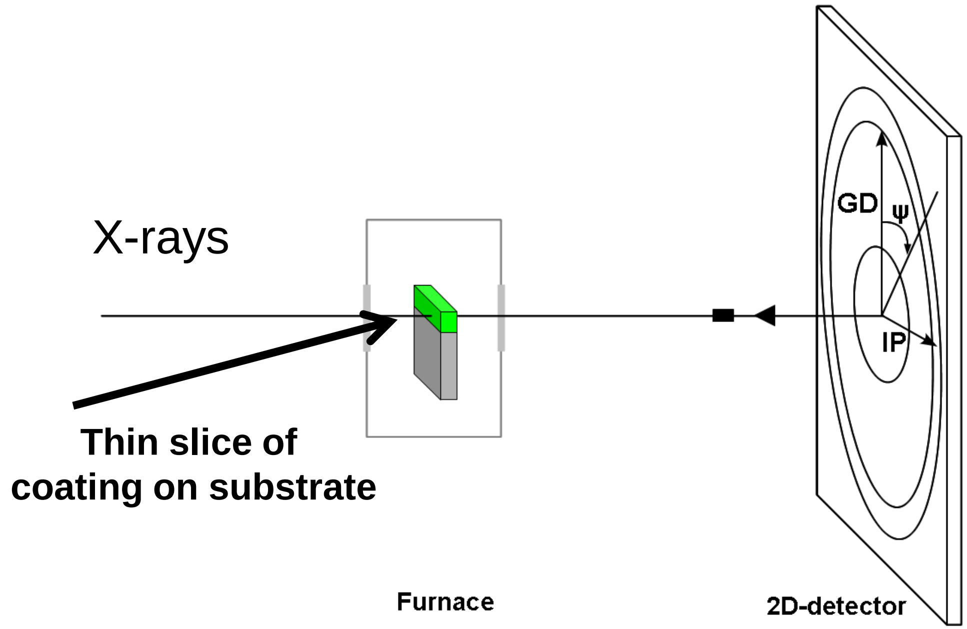
- Magnetron Sputter Deposition

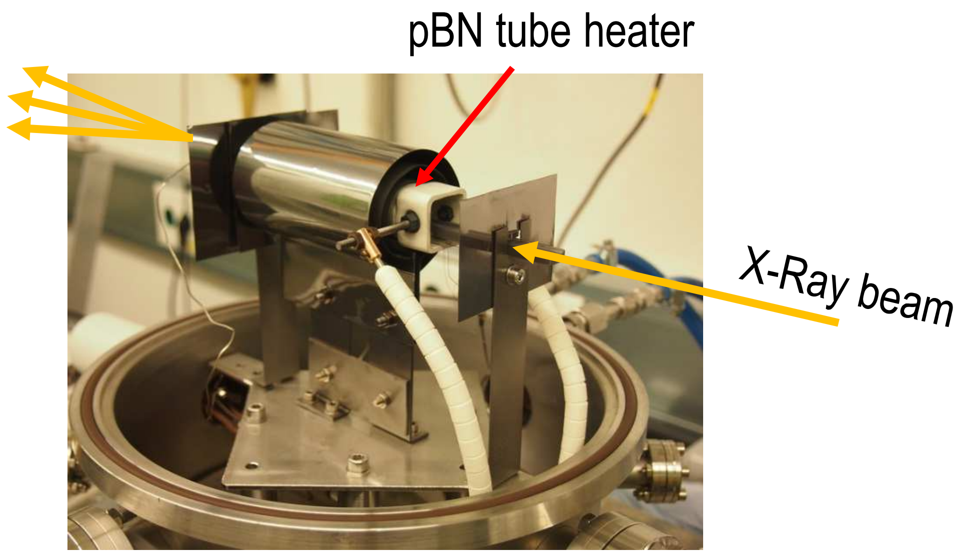


High vacuum furnace for *in situ* high energy x-ray scattering during film formation/decomposition

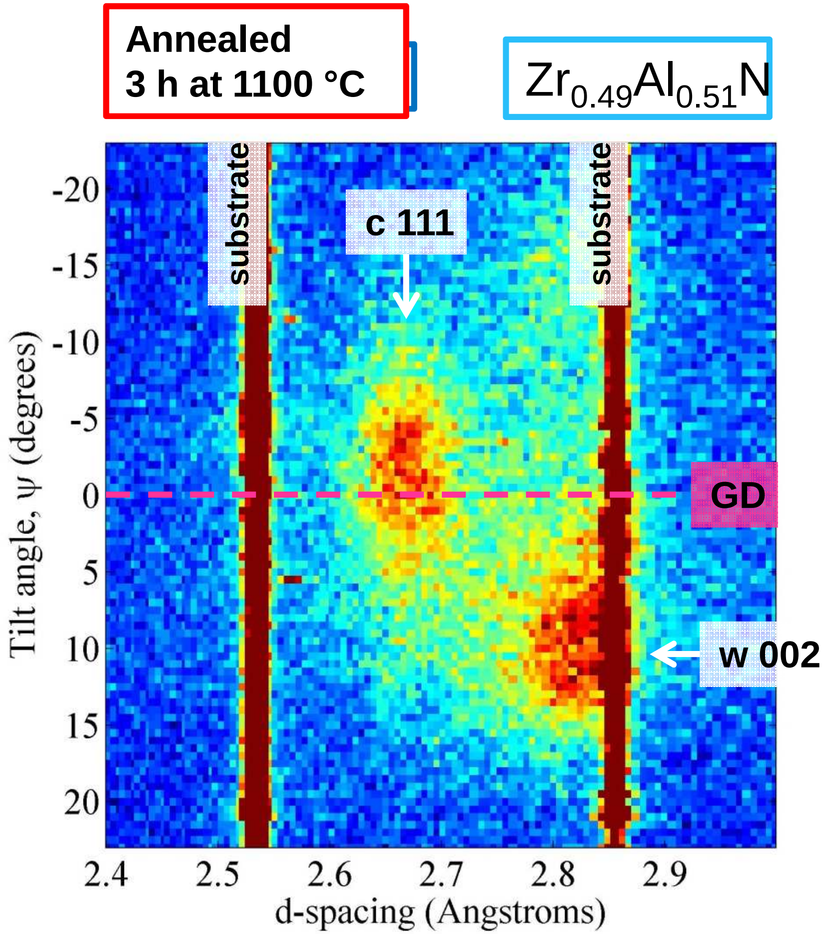
Capabilities:

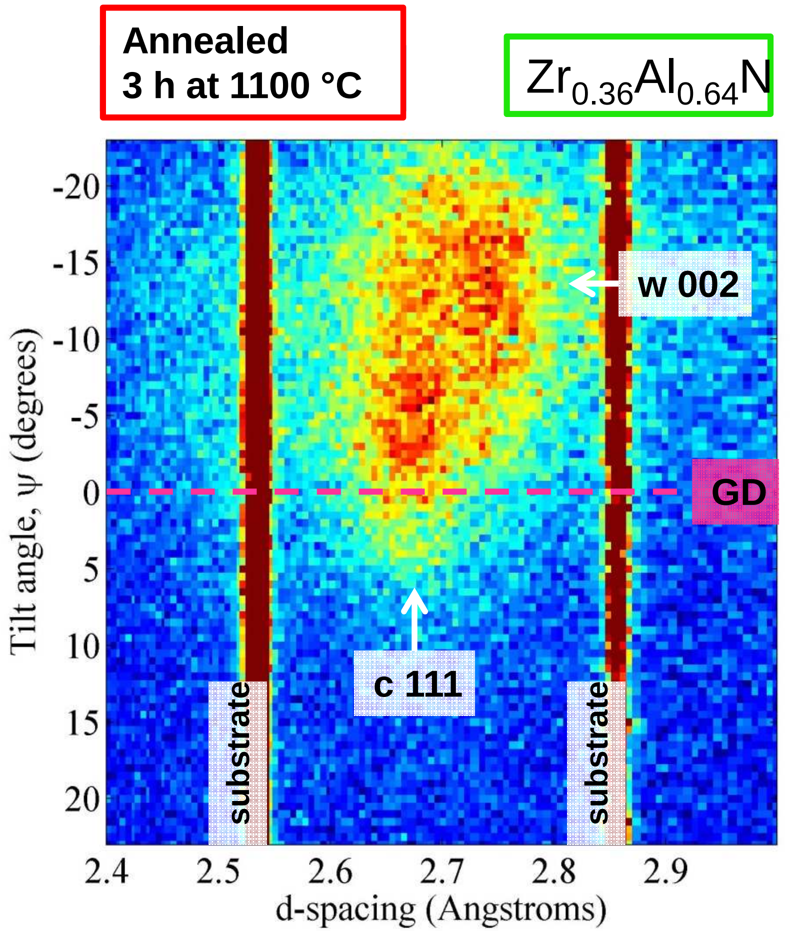
- >1100 °C
- Large uniform heating zone
- Inert athmosphere
- Simple sample alignment
- Off-beamline capability
- Portable





In situ WAXS during annealing of w-ZrAlN coatings (X-ray energy 78 keV → λ=0.158 Å)



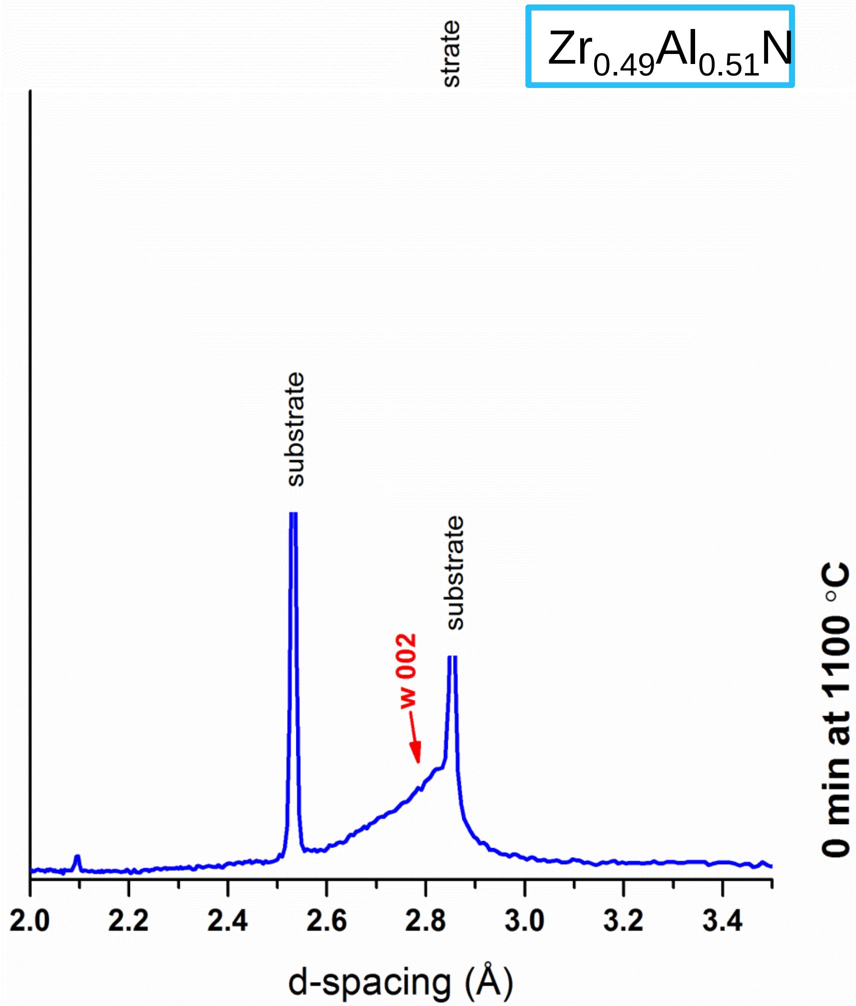


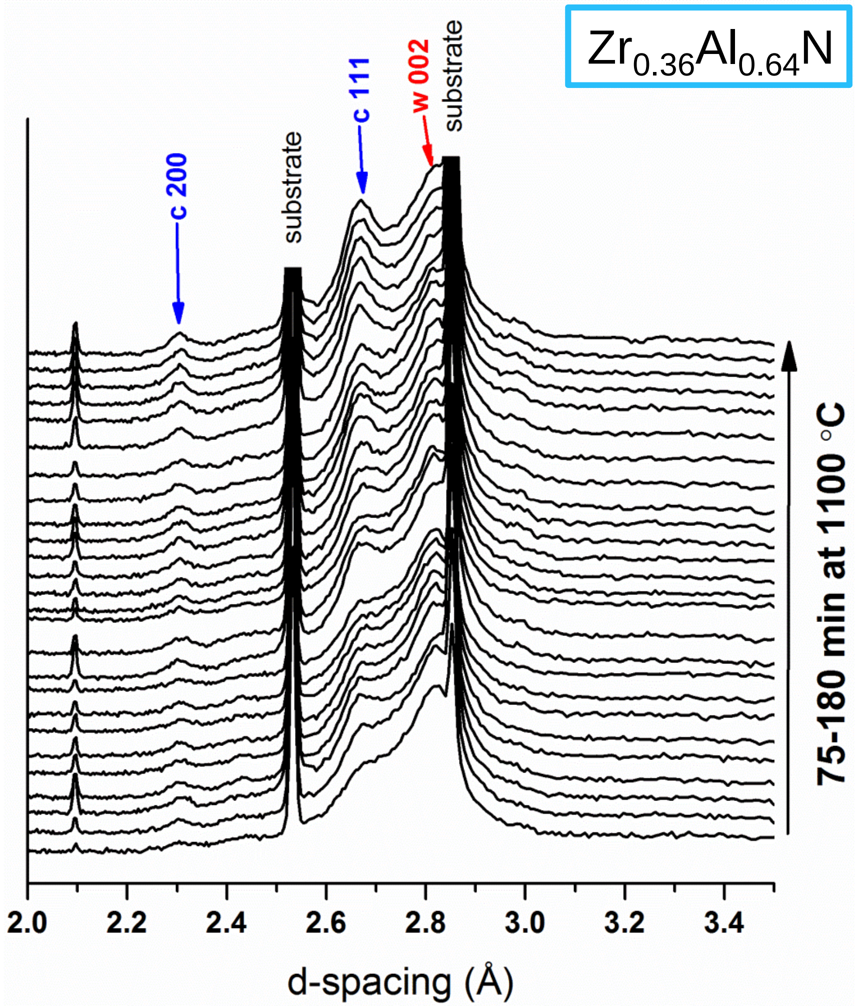
w-ZrAlN phase grows with the 002 direction tilted 10-15 degrees to growth direction.

c-ZrN forms with the 111 direction tilted ~10° to the wurtzite 002 direction.

L. Rogström, N. Ghafoor, J. Schroeder, N. Schell, J. Birch, M. Ahlgren, and M. Odén, J. Appl. Phys., **118**, 035309 (2015)

In situ WAXS → ZrAlN phase evolution during annealing

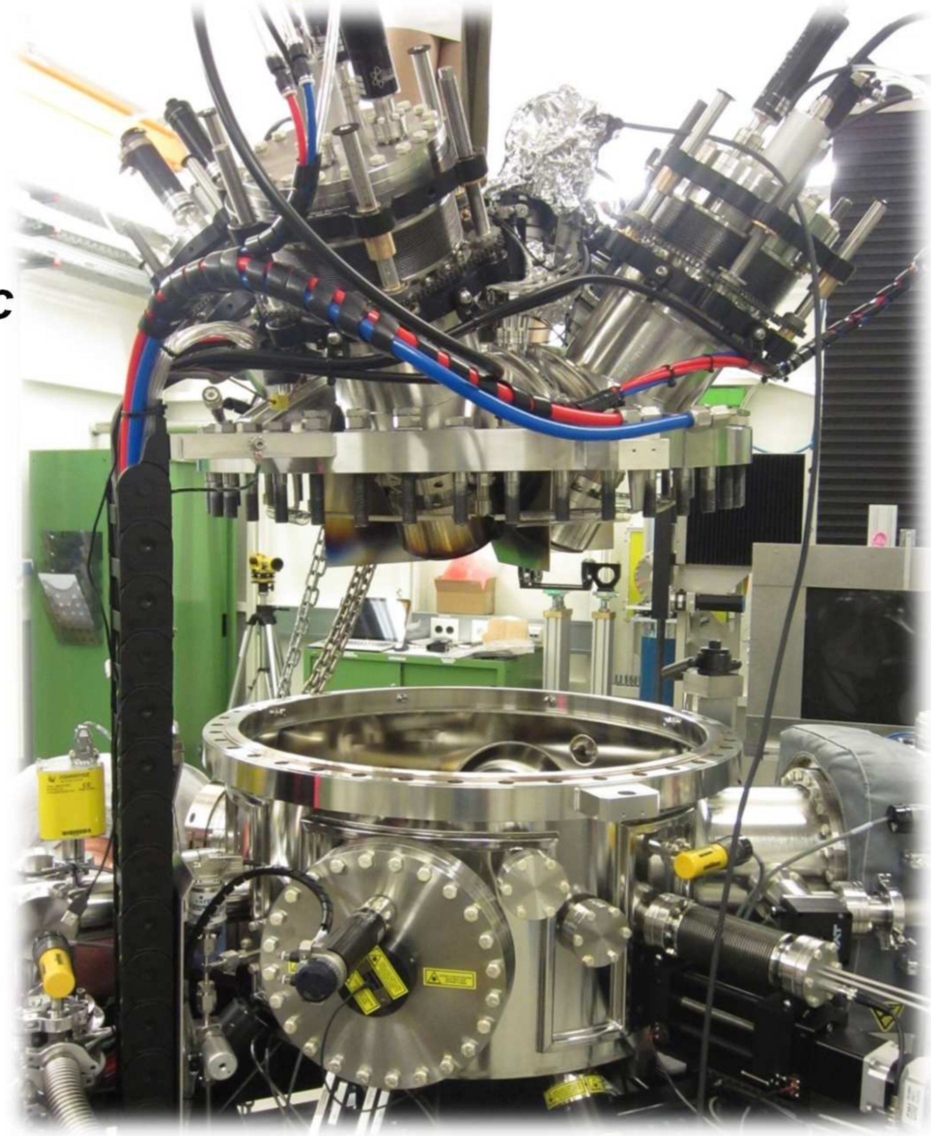




- Asymmetric broadening → w preciptates → wurtzite ZrAlN undergo spinodal decomp.
- c-ZrN nucleate and grow as soon as 1100 °C is reached.
- c-ZrN continue to grow at the expense of w-ZrAlN → **"over-ageing"**

L. Rogström, …J. Birch, et al. J. Appl. Phys., **118**, 035309 (2015)

UHV **magnetron sputtering** and **cathodic arc** deposition system for ***In situ*** **high energy x-ray scattering** during **industrially-relevant thin film growth**



<u>Capabilities</u>:

- ***Film growth under real industrial conditions:***
  - Sources: Four 75 mm ∅ ***magnetrons***
    Three 63 mm ∅ ***cathodic arc***
  - High substrate temperature T_s>1300
  - High power → high deposition rate
- ***Materials research quality films***
  - Ultra High Vacuum
  - Epitaxial growth
  - Auxiliary *in situ* tools
- ***Effective use of beamtime***
  - Minimal time for sample change
  - Minimal need of sample alignment
  - Full remote control
- ***Off-beamline capability***
- ***Portable under vacuum***

LINKÖPING UNIVERSITY

J. L. Schroeder, …,and J. Birch, Rev. Sci. Instrum. ***86****, 095113 (2015)*

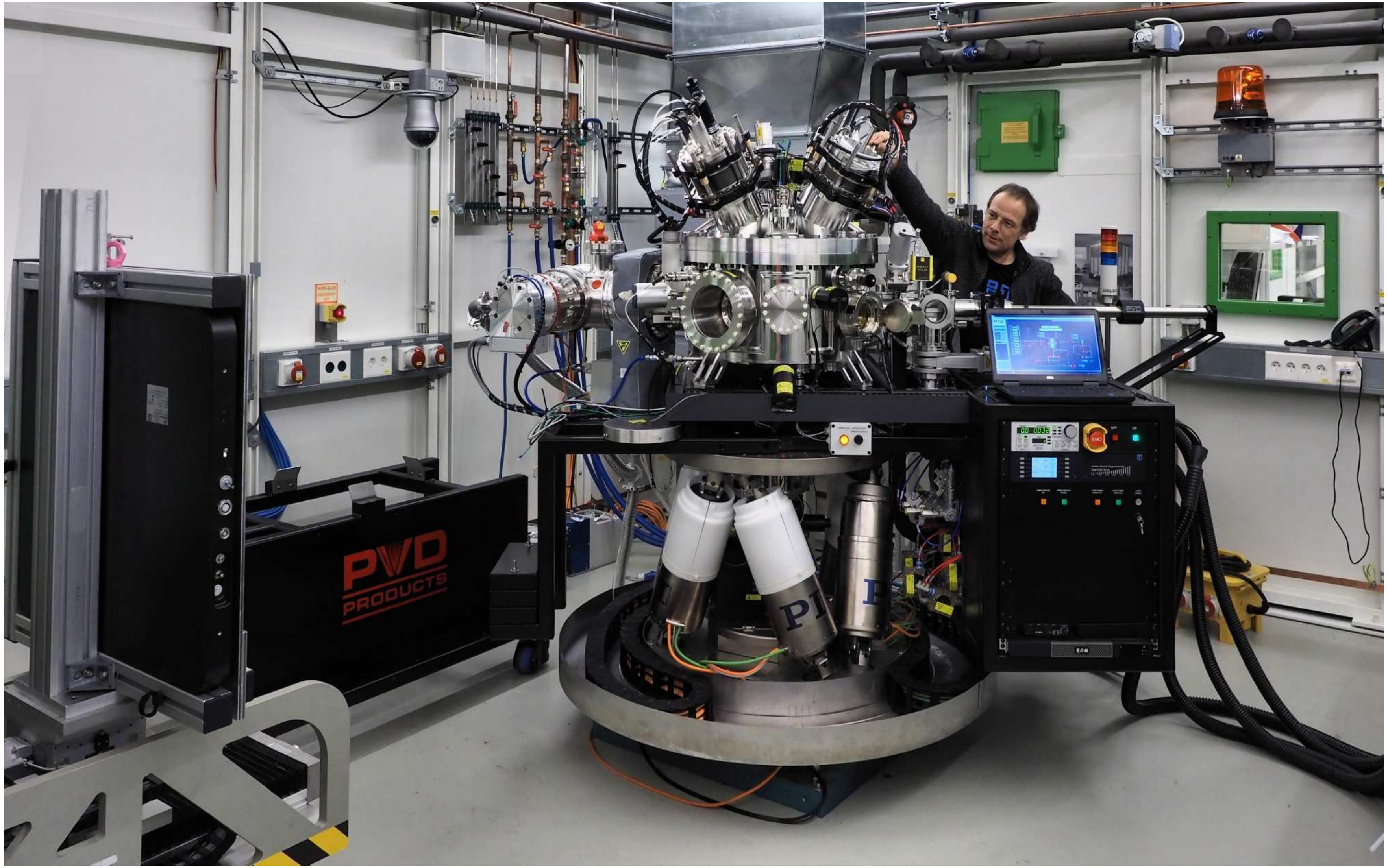
In situ UHV deposition chamber in the High Energy Materials Science beamline (HEMS)

Easy transport from off-beamline laboratory, sealed for maintained vacuum up to 2 hrs.

LINKÖPING UNIVERSITY

J. L. Schroeder, …,and J. Birch, Rev. Sci. Instrum. ***86****, 095113 (2015)*

In situ UHV deposition chamber in the High Energy Materials Science beamline (HEMS)

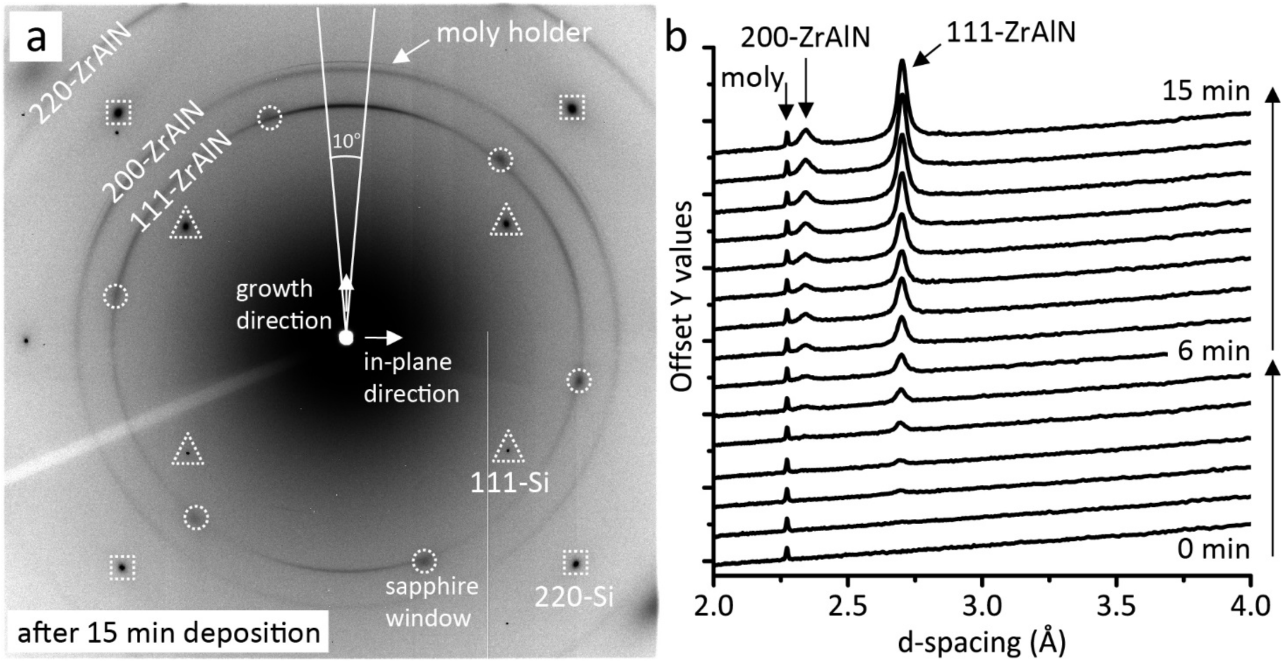


LINKÖPING UNIVERSITY

J. L. Schroeder, …,and J. Birch, Rev. Sci. Instrum. ***86****, 095113 (2015)*

Example of *in situ* XRD during growth

$Zr_{0.75}Al_{0.25}N$ reactive magnetron sputter deposition on Si (001)



300 nm

No coating

T_g = 700 °C
Growth rate = 20 nm/min
Working gas: 5 mTorr (10% N_2/90% Ar)

Time-resolved growth experiments

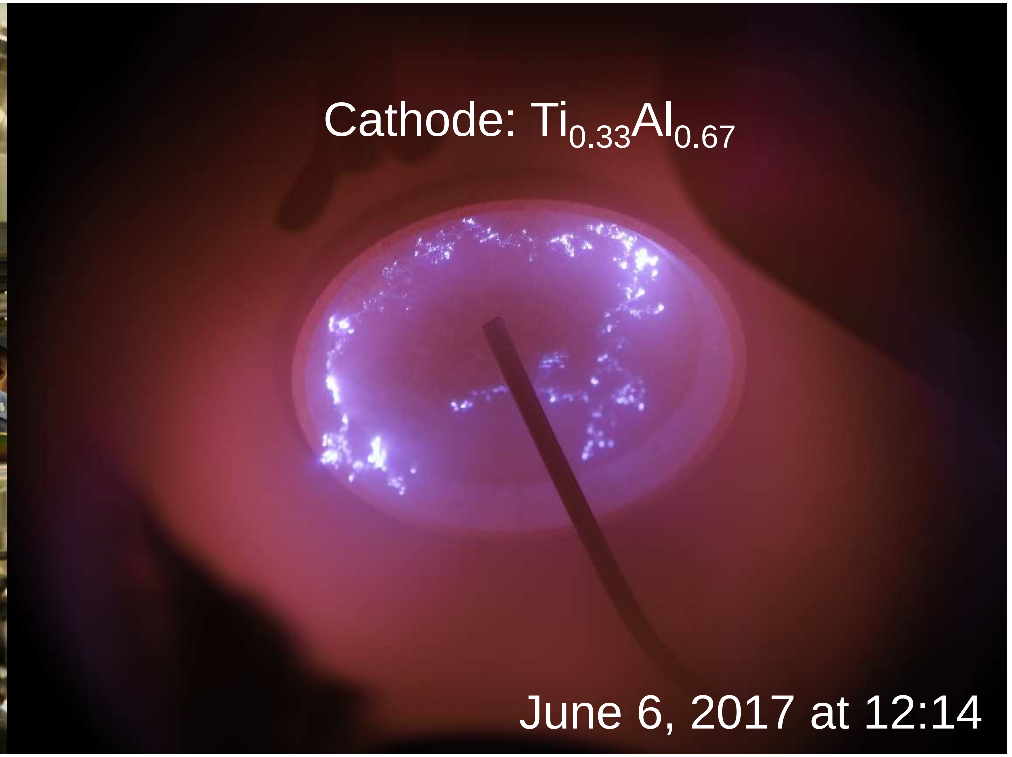
- Industrial growth rates
- Phase evolution
- Stress & strain evolution
- Nucleation & growth?

LINKÖPING UNIVERSITY

J. L. Schroeder, …,and J. Birch, Rev. Sci. Instrum. ***86****, 095113 (2015)*

Smoking Hot!

$Ti_{0.33}Al_{0.67}N$ cathodic arc deposition with *in situ* High Energy XRD



Mounting dedicated chamber lid with arc source

X-Cut design

UHV

Running UHV arc source in the beamline

$I_{cathode}$ = 90 A

Gas: 34 mTorr N_2

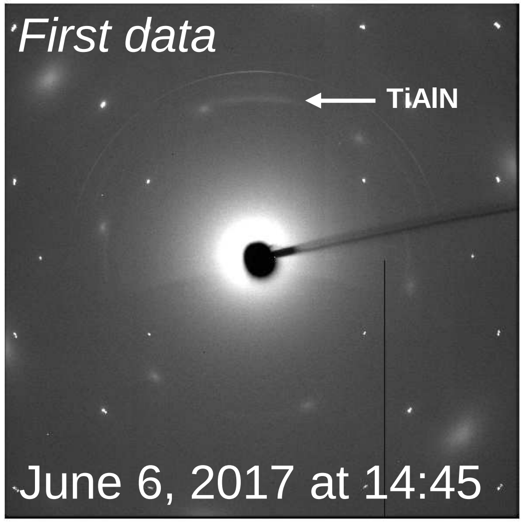
Bias = - 30 V

Temperature ≈ 400 °C

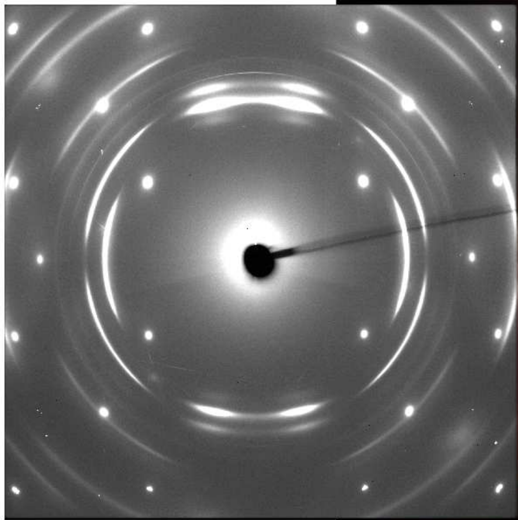
Smoking Hot!

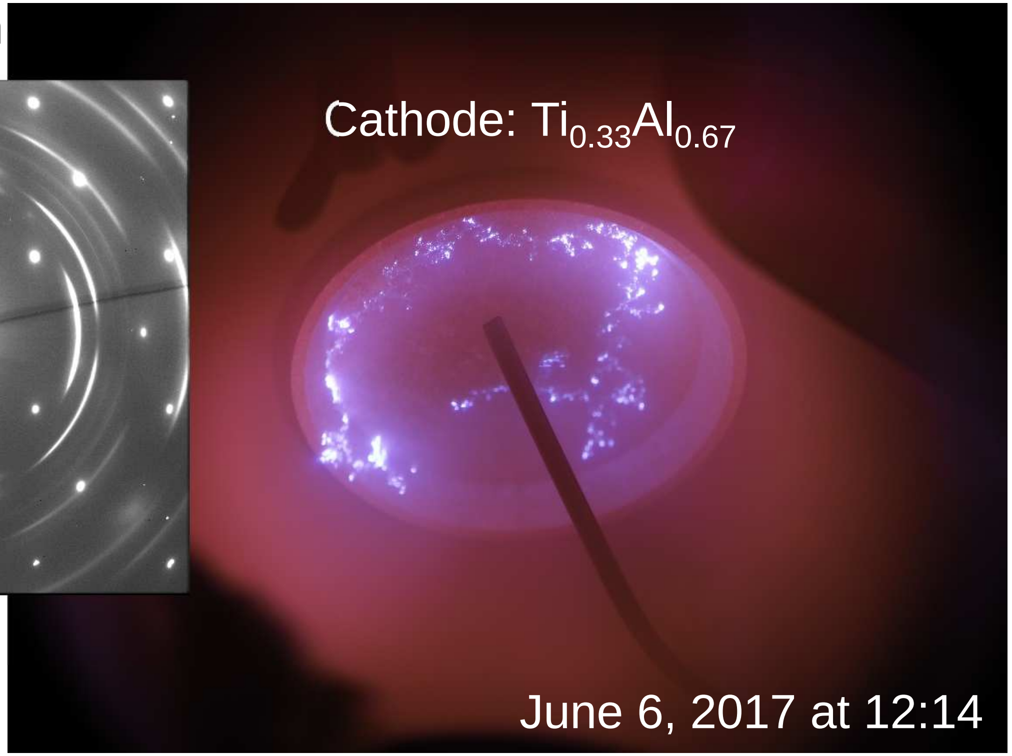
$Ti_{0.33}Al_{0.67}N$ cathodic arc deposition with *in situ* High Energy XRD

Dep. time = 30 s



Dep. time10 min





Exposure times = 6 x 1 s
Energy = 79.8 keV

In situ* WAXS of $Ti_{0.33}Al_{0.67}$N coating *during cathodic arc deposition

Never-before conducted experiment!

Running UHV arc source in the beamline

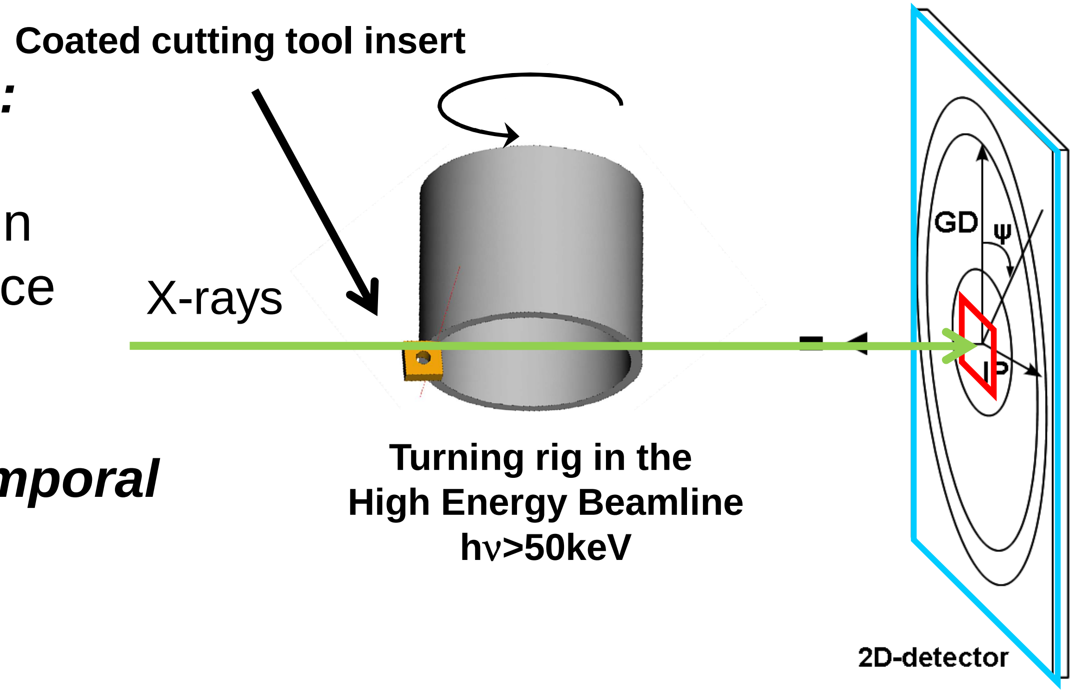
- $I_{cathode}$ = 90 A
- Gas: 34 mTorr N_2
- Bias = - 30 V
- Temperature ≈ 400 °C

Death of thin films…

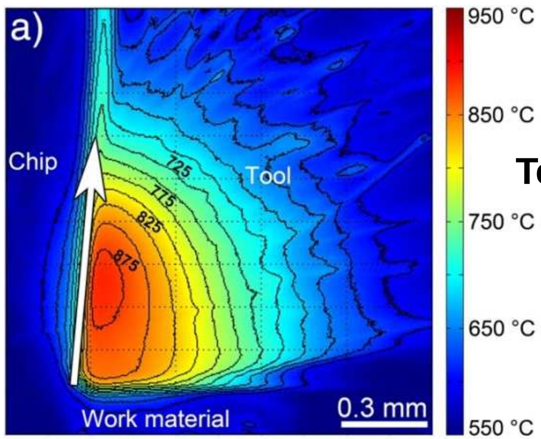
Video courtesy of SECO Tools AB

In operando high energy X-ray scattering during high-speed dry cutting

- ***Industrial cutting conditions:***
  - Vertical lathe
  - High speed cutting >200 m/min >750 rpm, 100 mm ∅ workpiece
  - Standard tool holder



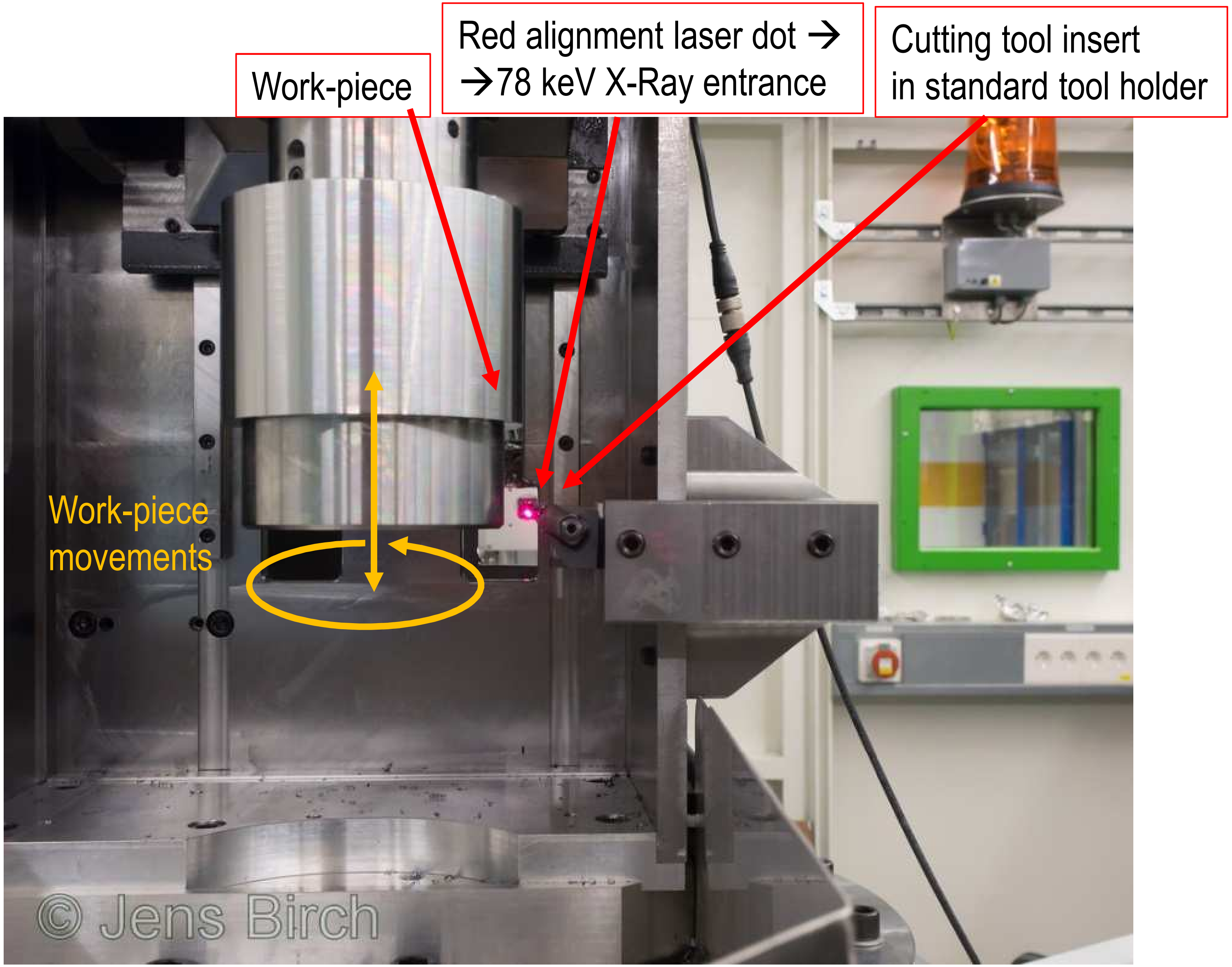
- ***High d-spacing, spatial & temporal resolution***
  - µm tool stability *during* cutting
  - precision beam access
  - Area detector compatible



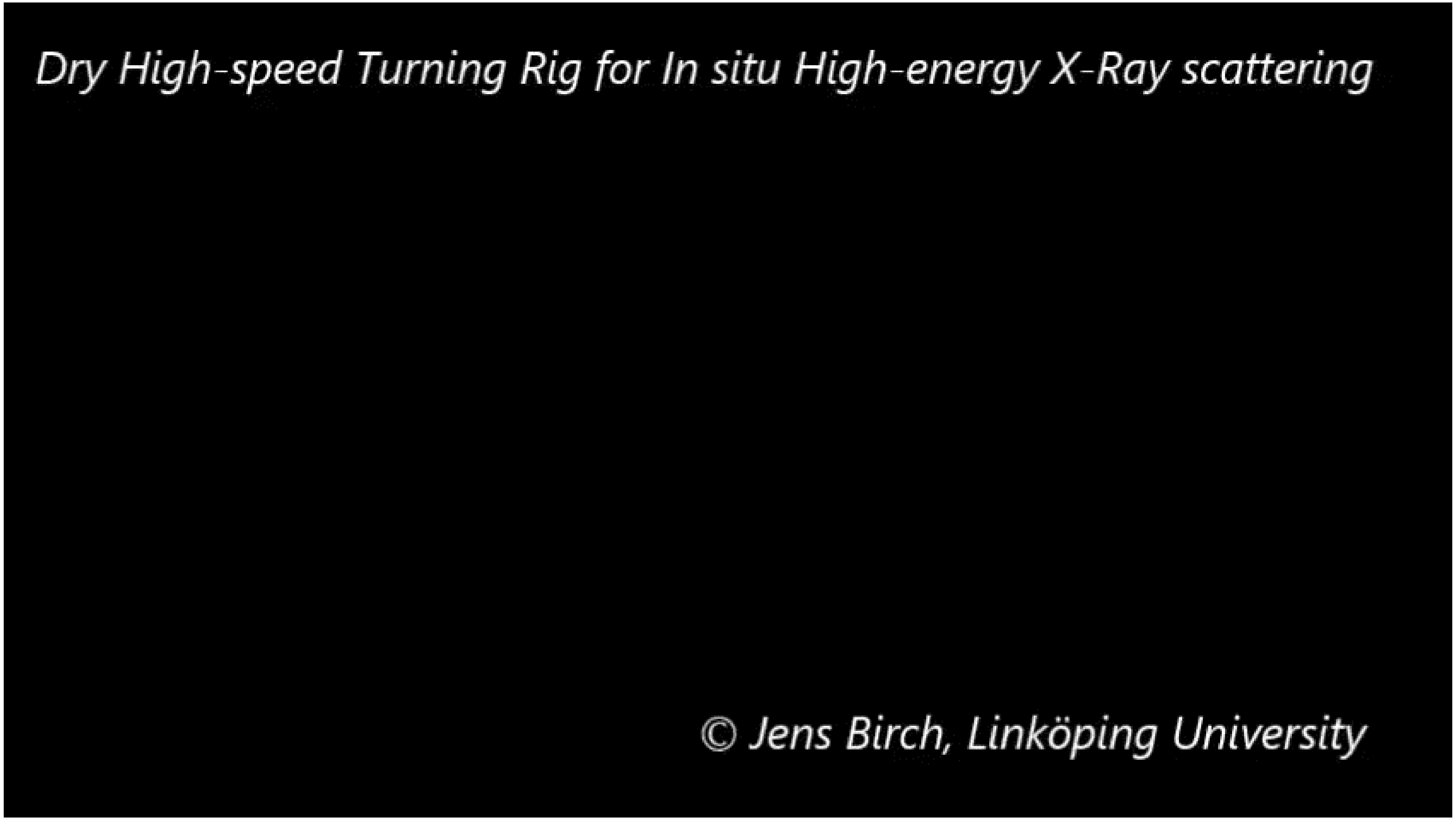
Temperature distribution on cutting tool insert during cutting

- ***Efficient use of beamtime***
  - Full remote control
  - portable
  - fast setup

Turning operation at the beamline



Turning operation at the beamline



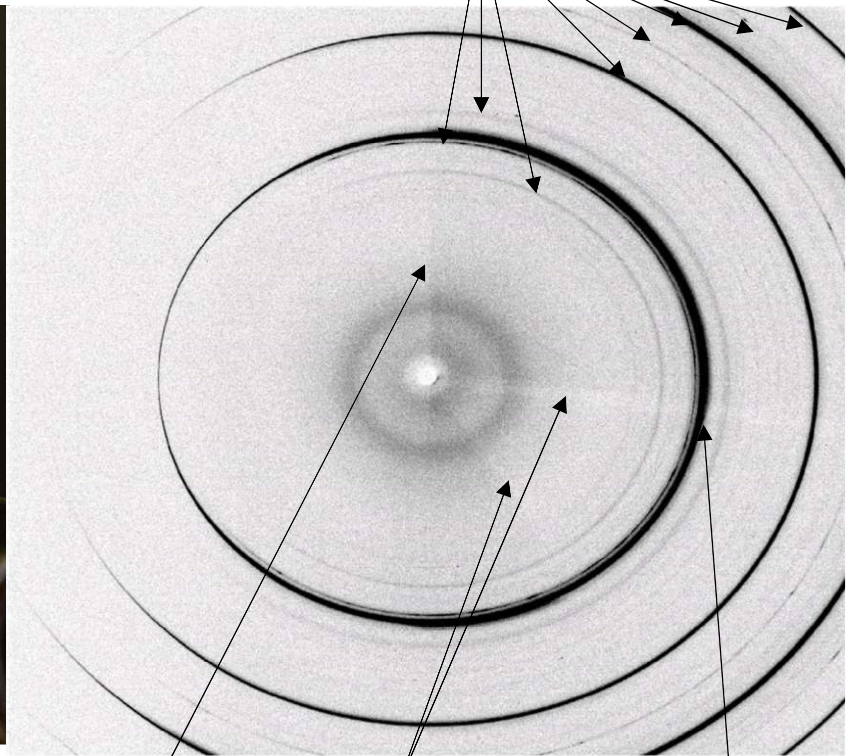
LINKÖPING UNIVERSITY

SECO

Dry High-speed Turning – *In operando* XRD

Dry High-speed Turning – Detailed view

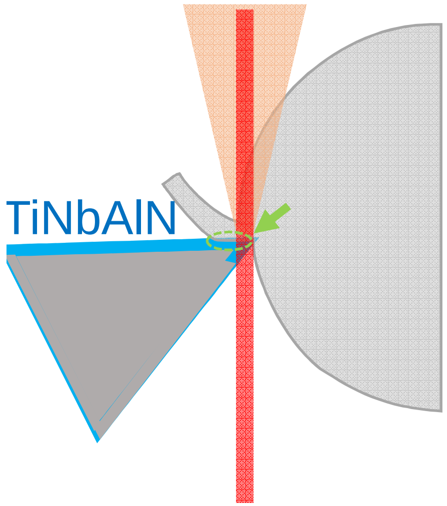
Diffraction by the tool substrate and coating



X-Rays shadowed by work piece

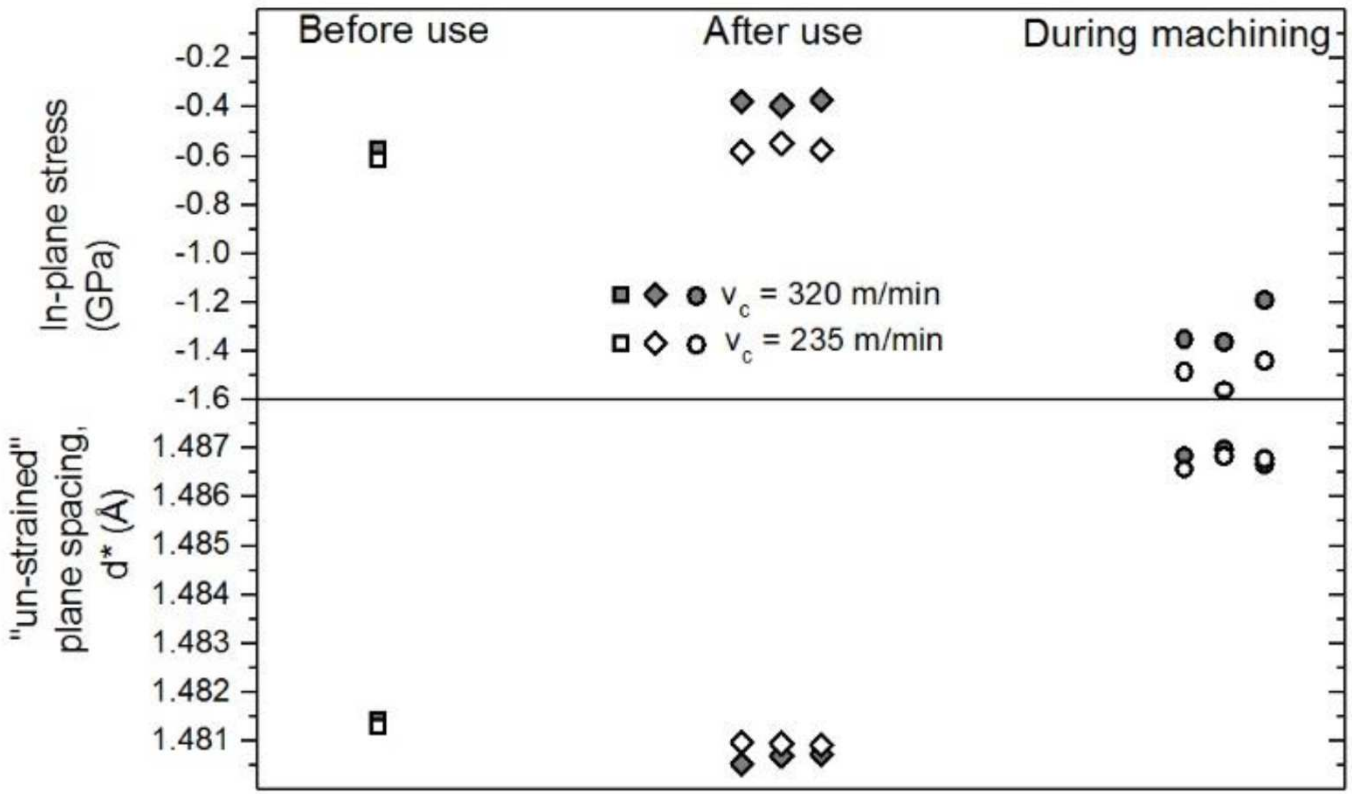
X-Rays shadowed by tool

Diffraction by the chips



$Ti_{0.22}Nb_{0.36}Al_{0.42}N$ coated onto WC-Co

Stress on tool edge - different cutting speeds



Lowest stresses during highest cutting speeds! → Paradox?

- Higher speed → Larger relaxation of intrinsic stresses
- Lower speed → Higher stress during machining (less relaxation)

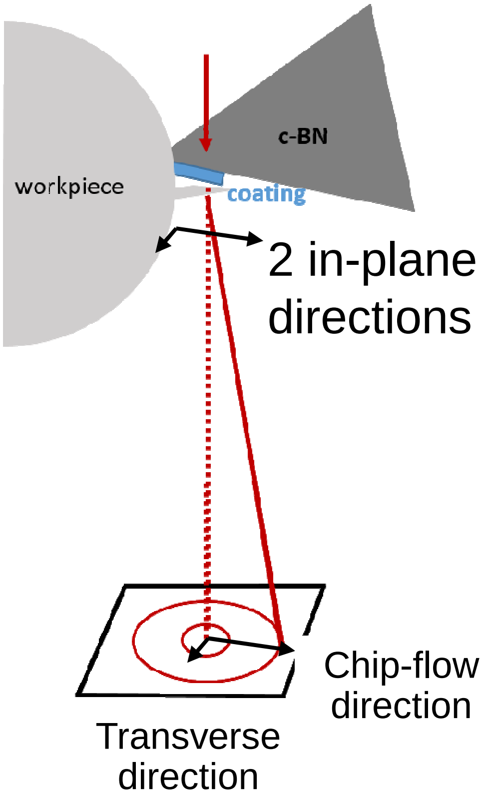
LINKÖPING UNIVERSITY

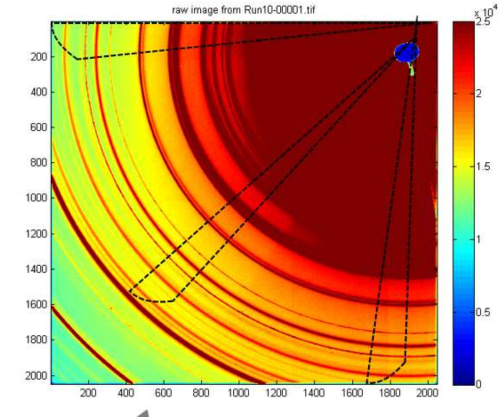
***Preliminary results by* Lina Rogström, Jens Birch et al.**

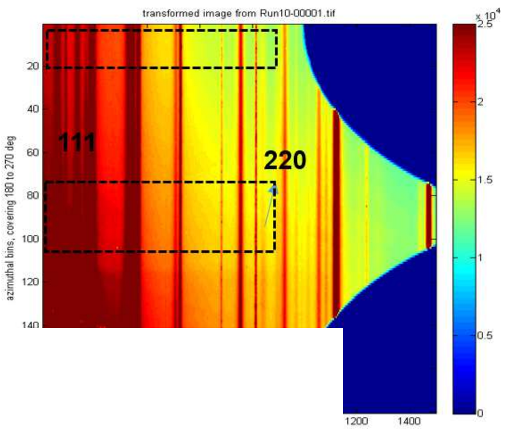
SECO

$Ti_{0.22}Nb_{0.36}Al_{0.42}N$ / c-BN, focussed beam

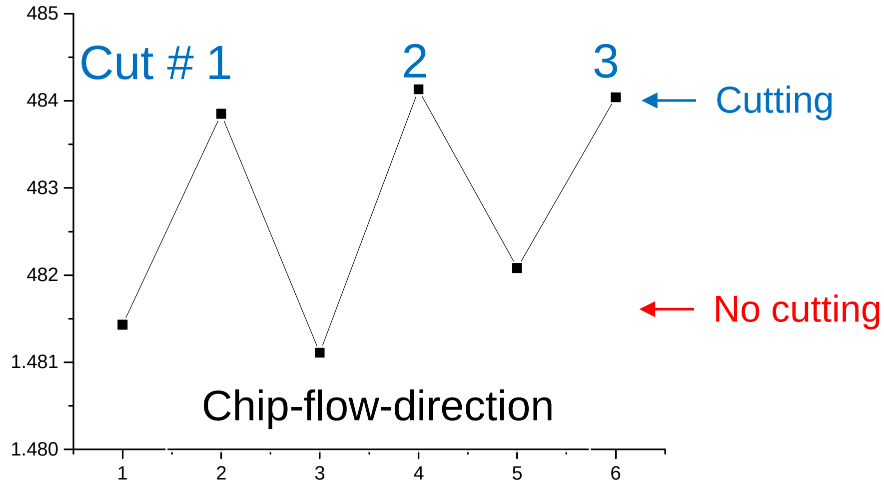
Preliminary results by Lina Rogström, Jens Birch et al.

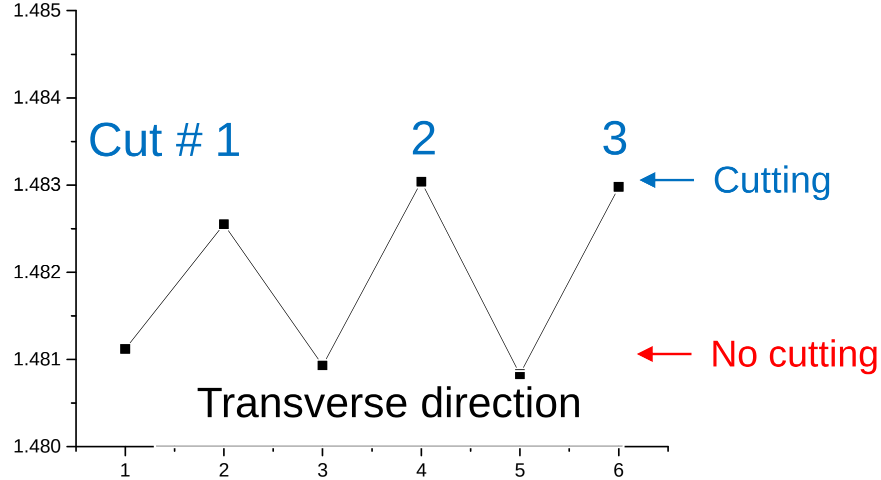






Beam size = 30 x 5 µm²
Exposure time = 60 x 1 s (one exposure/length)
Rotation speed = 750 rpm





- ***In-plane directional measurements***
- ***~10 µm resolution***

- Lattice expansion higher in chip-flow direction
  → not the usual biaxial state of stress
- Anisotropic plastic deformation of grains (Peak widths)

LINKÖPING UNIVERSITY
SECO

”A Lathe Like No Others”

EDGE Magazine 1, p.32-35 (2017)
https://www.secotools.com/#article/67498

PHOTO: PER GROTH

Seco's Janne Eriksson and Mats Johansson Jõesaar – Unusual inspiration has led to brilliant development.

"Seco is the first metalworking company in the world that's doing it, and we now have a unique head start."

4 questions for

Jens Birch
Professor of Thin Film Physics at Linköping University in Sweden

SECO TOOLS AND LINKÖPING UNIVERSITY have worked together since 2010. PhD students have worked real-time with X-ray-technology studies of Seco's coatings.

Why does your institution work together with industry?

"Simply put, we are streamlining material process in order to better understand materials. It's important that our research is close to reality, and so it's only natural to pick up issues and problems from industry. As we produce the answers required, new knowledge often arises, which generates new questions and new ideas for new materials. It's a really fun way to work."

What challenges are you taking on with Seco Tools?

"We have worked together for a long time, among other things on real-time studies of the effects of pressure, temperature and other variables that affect the characteristics of the layers when they are used. We look at a few layers at a time to better understand each layer's characteristics and to be able to interpret the results that we see. The intensity of Petra III's X-rays offers unique opportunities in this area in

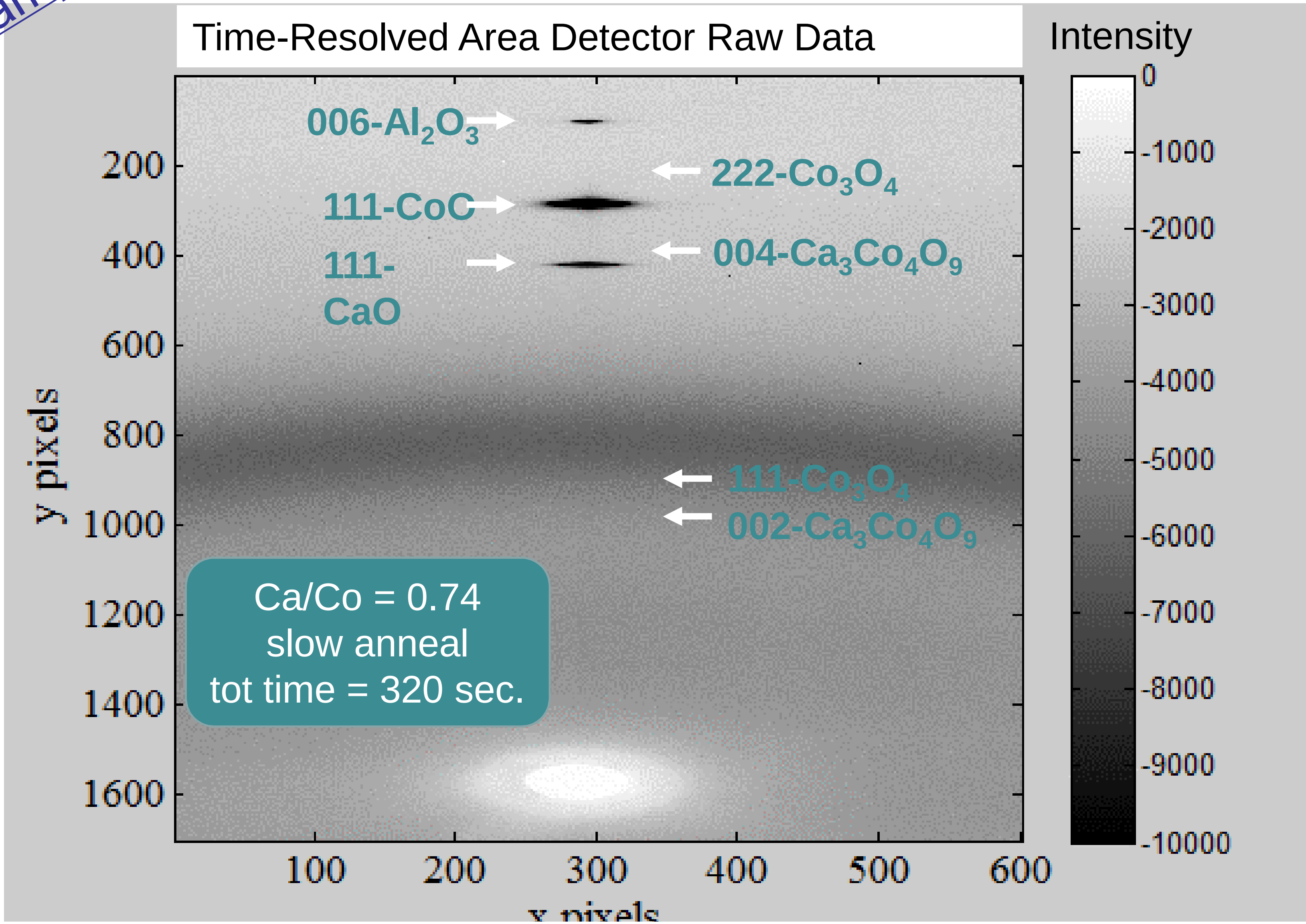
X-Cut Experiments 2012-2017 **Red=Spin-off projects**

1. Thermal stability of h-ZrAlN coatings (annealing chamber, EH3) [1]
2. Thermal expansion of TiAlN (annealing chamber, EH1) [2]
3. Growth kinetics of h-AlN in TiAlN coatings (annealing chamber, EH1, EH3) [3]
4. Thermal stability of c-TiZrAlN coatings (annealing chamber, EH1, EH3) [4]
5. Hardness in epitaxial TiAlScN alloy thin films and rocksalt TiN/(Al,Sc)N superlattices (annealing chamber EH1) [5]
6. Thermal stability of epitaxial cubic-TiN/(Al,Sc)N metal/semiconductor superlattices (annealing chamber EH1),[6]
7. Formation of Layered Cobaltate Ca3Co4O9 by Annealing [7]
8. Thermal stability of HfN/ScN, ZrN/ScN, and Hf0.5Zr0.5N/ScN metal/semiconductor superlattices [9]
9. Growth of h-AlN in c-TiCrAlN coatings (annealing chamber, EH1)
10. Effect of grain size on decomposition of TiAlN (annealing chamber, EH1)
11. Phase transformations in $Zr_{0.65}Al_{0.35}N$/TiN (annealing chamber, EH1)
12. Phase identification in MS ZrAlN thin films (EH1)
13. Structure of MS ZrAlN/ZrN multilayers (EH1)
14. Oxidation behavior of c-TiCrAlN coatings (Lamp setup, EH1)
15. Phase transformations in ‘stress-free’ TiAlN grown by HiPIMS (annealing chamber, EH3)
16. Phase transformations in amorphous TiSiBN coatings (annealing chamber, EH3)
17. Phase evolution during growth of TiN/ZrAlN multilayers (Sputtering chamber, EH3)
18. Phase evolution during growth of ScAlN films (Sputtering chamber, EH3)
19. Dynamics of nucleation and growth of InAlN nanorods (Sputtering chamber, EH3)
20. Thermal stability of $(CrAl)_2O_3$ protective coatings on superalloys
21. Ti/Ni neutron mirrors
22. Cathodic arc in situ X-ray scattering during growth

Sum X-Cut Beamtime: 95 days

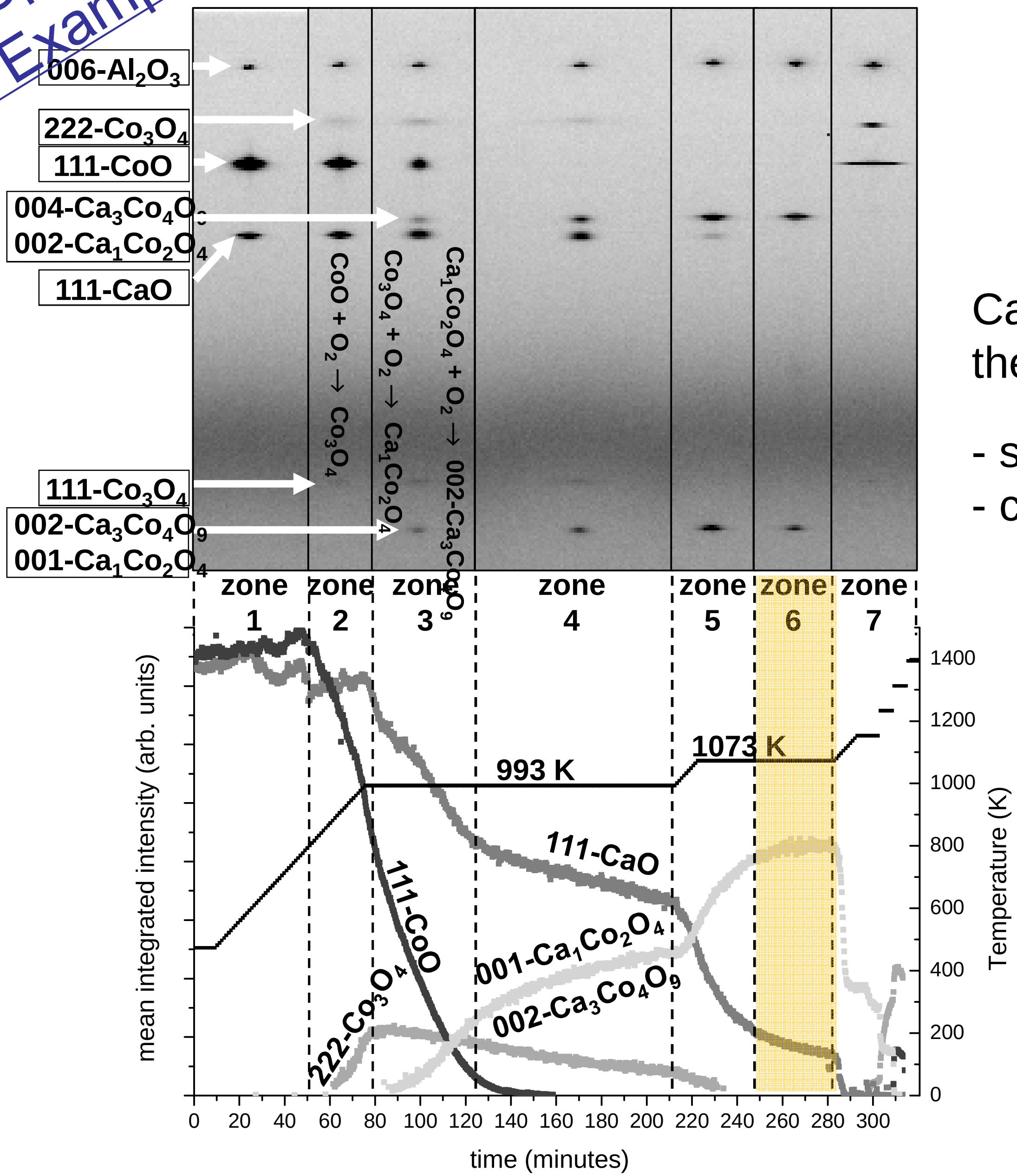
Spin-off Example

Synthesis of $Ca_3Co_4O_9$ thermoelectric coatings



B. Paul, J. L. Schroeder, S. Kerdsongpanya, N. V. Nong, N. Schell, D. Ostach, J. Lu, J. Birch, and Per Eklund, Advanced Energy Materials 1, 1400022 (2015)

Synthesis of $Ca_3Co_4O_9$ thermoelectric coatings



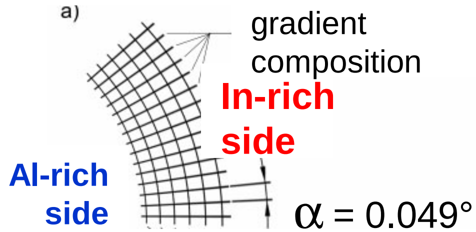
CaO and CoO transformation into the thermoelectric phase $Ca_3Co_4O_9$

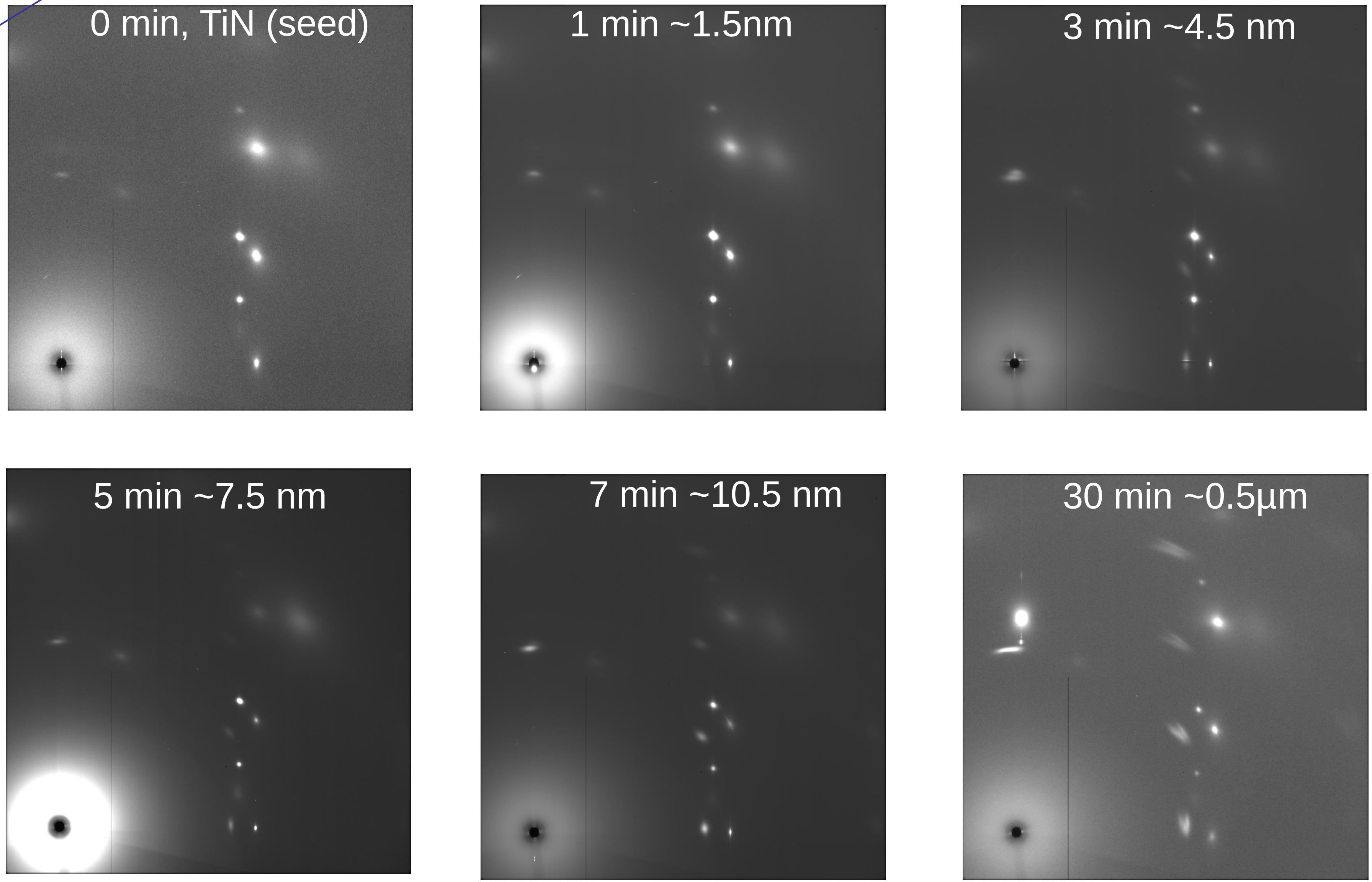
- starts at 993 K
- complete: >1 hr at 1073 K (Zone 6)

B. Paul, J. L. Schroeder, S. Kerdsongpanya, N. V. Nong, N. Schell, D. Ostach, J. Lu, J. Birch, and Per Eklund Advanced Energy Materials 1, 1400022 (2015)

Spin-off Example

In situ nucleation and growth of curved $In_xAl_{1-x}N$ nano-rods





Preliminary results by Ching-Lien Hsiao, Alexandra Serban, and Jens Birch

Summary of the RÅC X-Cut project

- **High-energy Synchrotron makes possible *in situ* time-resolved WAXS** (and SAXS) **for …**
  - nucleation and growth
  - solid state reactions
  - nano-structure formation
  - degradation of tool coatings at real cutting conditions
  - study tool coatings

    … tool substrates

    … phase evolution in the chips
  - …**during industrial synthesis and use (cutting)**

LINKÖPING UNIVERSITY

SECO

Helmholtz-Zentrum Geesthacht

Zentrum für Material- und Küstenforschung

Röntgen Ångström Cluster

- **Brought industry to the synchrotron**
- **Generated *a lot* of research & industrial collaboration proposals**
- **Generated *a lot* of spin-off projects**

The ”X-Cut” team

LINKÖPING UNIVERSITY

Thin Film Physics Division:

Jens Birch (project leader)
Lars Hultman
Jeremy Schroeder
Muhammad Junaid
Naureen Ghafoor
Martin Magnusson
Ching-LienHsiao
Alexandra Serban
Erik Ekström

Nanostructured Materials Div.:

Magnus Odén
Lina Rogström
Y. H. Chen

Helmholtz-Zentrum Geesthacht
Zentrum für Material- und Küstenforschung

Beamline P07 Petra III

Norbert Schell
Daniel Ostach
Andreas Schreyer
Martin Müller

SECO

Cutting Instrument

Mats Johansson
Rachid M'Saoubi
Mikael Fallqvist
Mats Carlsson
Janne Eriksson

Röntgen Ångström Cluster

for financial support

Trends and Opportunities for Advanced Materials and Techniques

Norbert Schell & Jens Birch

09 June 2017 / DESY

GEMS
German Engineering Materials Science Centre

Helmholtz-Zentrum Geesthacht
Centre for Materials and Coastal Research

National, international and industry interest for current RÅC-equipment at HEMS

Helmholtz-Zentrum Geesthacht
Centre for Materials and Coastal Research

In operando lathe:

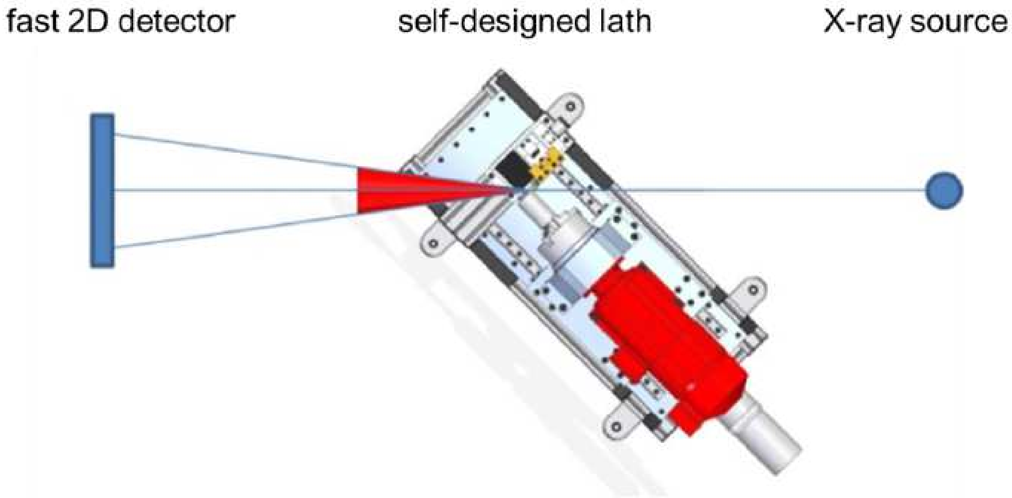
→ **Prof. Jens Gibmeier, Karlsruhe Institute of Technology**

→ **Prof. Wolfgang Hintze*, Hamburg Technical University**

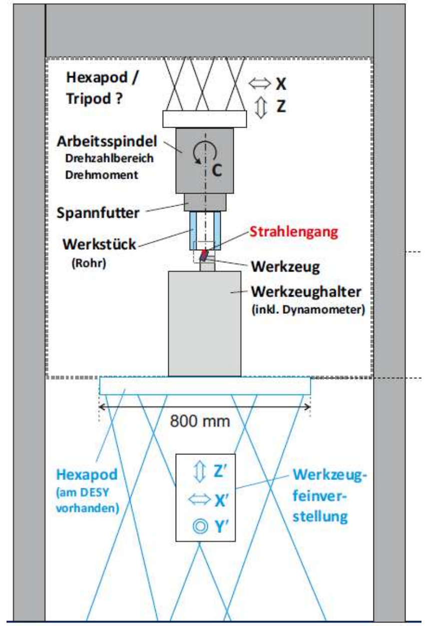
KIT Karlsruher Institut für Technologie

TUHH Hamburg University of Technology

Prof. Dr. J. Gibmeier, KIT, Karlsruhe
BUE (built-up edge) in SAE 1045 **steel** and EN AW-5754 **Al-alloys** on **WC/Co** and **Si_3N_4** substrates



Prof. Dr.-Ing. W. Hintze, TU Hamburg-Harburg,
Fa. Gühring (manufacturer of precision tools in Albstadt)
Cemented carbide (WC-Co), PCBW, mixed ceramics



National, international and industry interest for current RÅC-equipment at HEMS

***In operando* lathe:**

- → **Prof. Jens Gibmeier, Karlsruhe Institute of Technology**
- → **Prof. Wolfgang Hintze*, Hamburg Technical University**
- → **Prof. Jan-Eric Ståhl, Lund University**

- → **SANDVIK Coromant**

National, international and industry ***interest for current RÅC-equipment*** *at HEMS*

Helmholtz-Zentrum Geesthacht
Centre for Materials and Coastal Research

In operando lathe:

→ **Prof. Jens Gibmeier, Karlsruhe Institute of Technology**

→ **Prof. Wolfgang Hintze*, Hamburg Technical University**

→ **Prof. Jan-Eric Ståhl, Lund University**

→ **SANDVIK Coromant**

KIT Karlsruher Institut für Technologie

TUHH Hamburg University of Technology

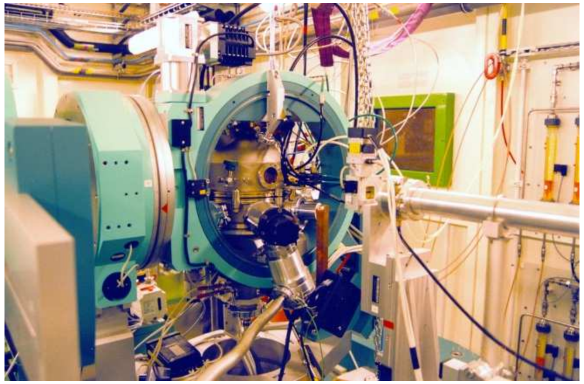
LUNDS UNIVERSITET
Lunds Tekniska Högskola

SANDVIK Coromant

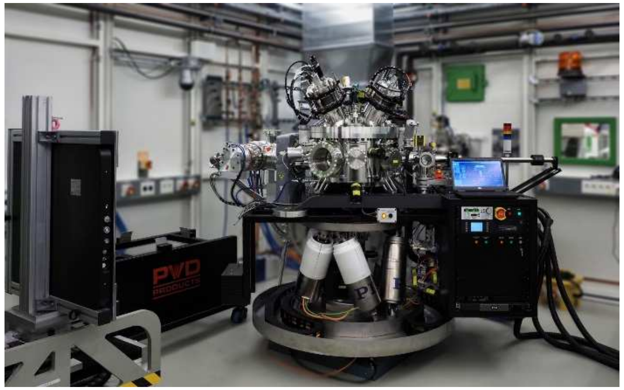
In situ sputter deposition system

→ **Dr. Rui Martins*, Lissabon NOVA University**

FCT FACULDADE DE CIÊNCIAS E TECNOLOGIA UNIVERSIDADE NOVA DE LISBOA



ROBL-CRG, ESRF
academic



HEMS, PETRA III
industry-relevant

LINKÖPING UNIVERSITY

National, international and industry interest for current RÅC-equipment at HEMS

In operando lathe:

→ Prof. Jens Gibmeier, Karlsruhe Institute of Technology

→ Prof. Wolfgang Hintze*, Hamburg Technical University

→ Prof. Jan-Eric Ståhl, Lund University

→ SANDVIK Coromant

In situ sputter deposition system

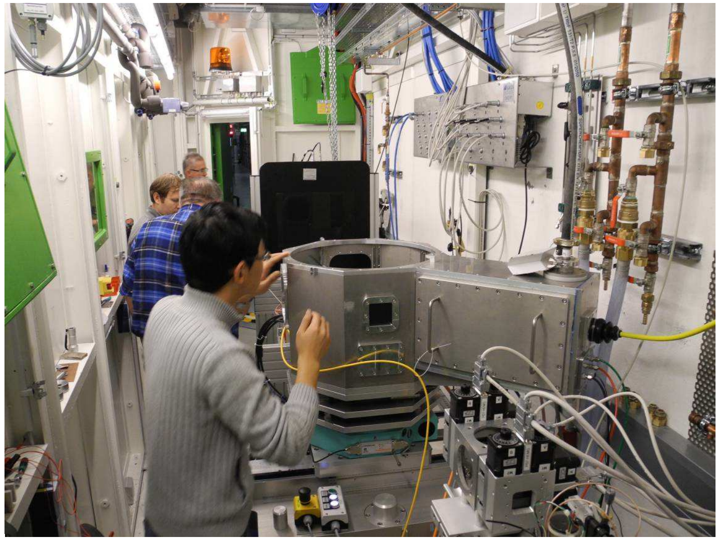
→ Dr. Rui Martins*, Lissabon NOVA University

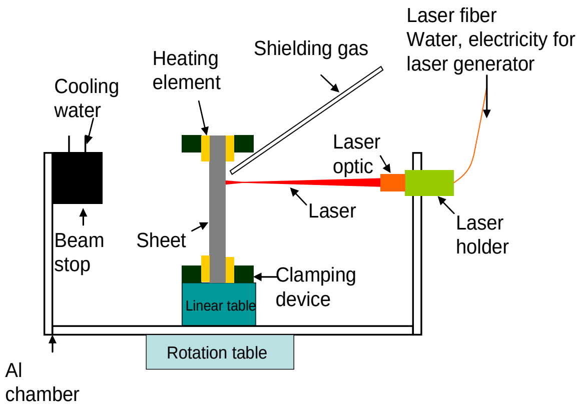
→ Dr. Erik Lindahl, Uppsala University

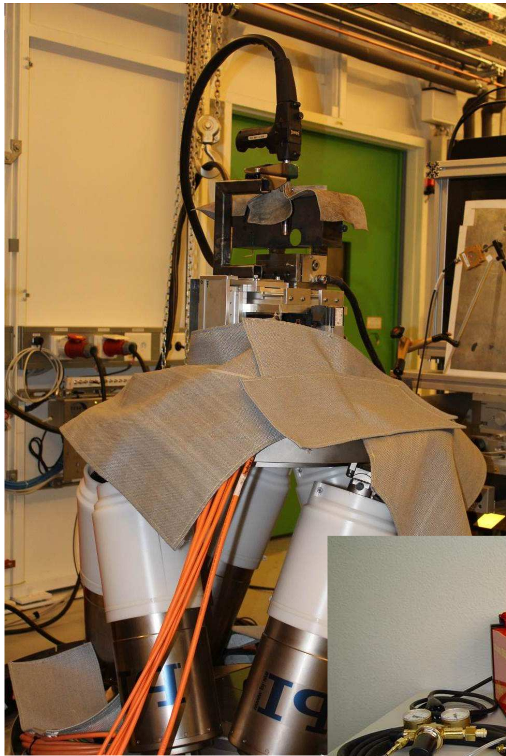
→ PLANSEE*

***Participated in joint grant proposals**

Future Trends: in-situ laser beam welding
in-situ TIG (Tungsten Inert Gas) welding







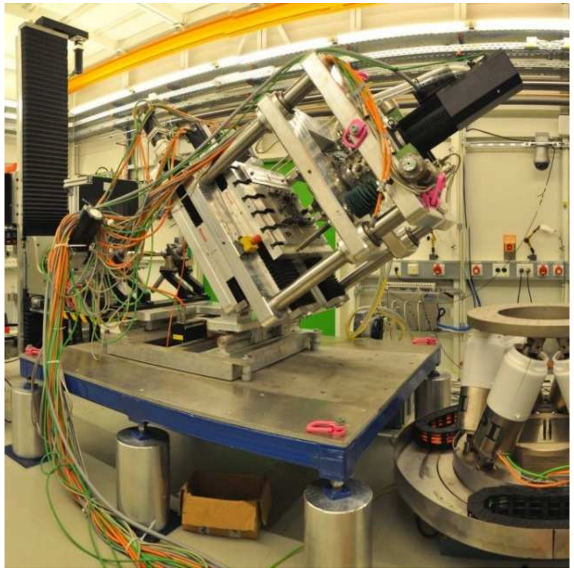
CastoTIG 1401 DC

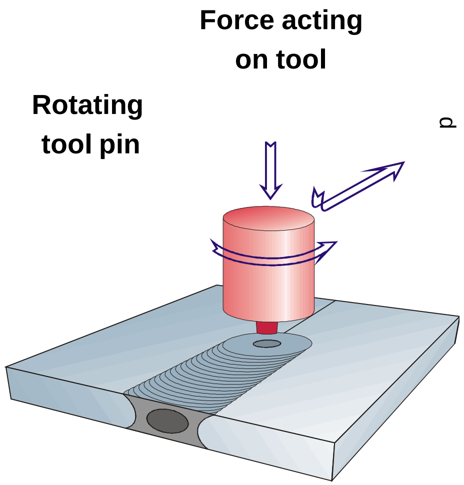
- voltage: 31.3 V
- **current (mean): 343 A (pulsed)**
- feed rate: 370 mm / min
- gas: CARGAL 1 (Ar/CO_2)
- LTT-wire-∅: 1.6 mm
- LTT: 10% Cr / 10% Ni

γ-TiAl attractive aero-space application, low density, good tensile properties at high temperature, high creep & oxidation resistance.

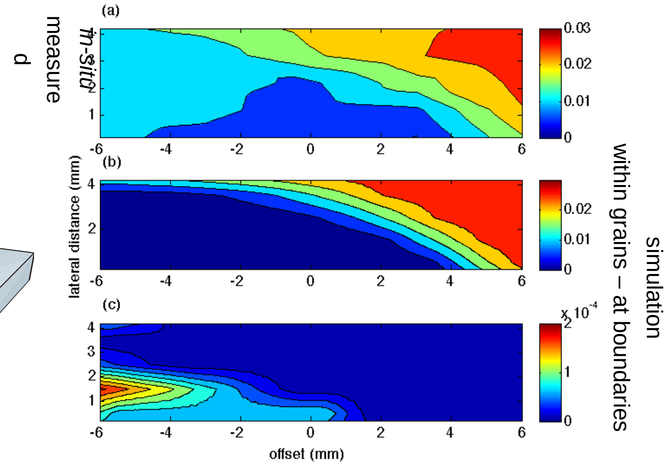
Helmholtz-Zentrum Geesthacht
Centre for Materials and Coastal Research

In-situ friction stir welding

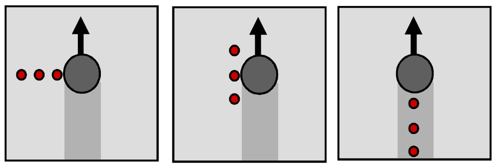




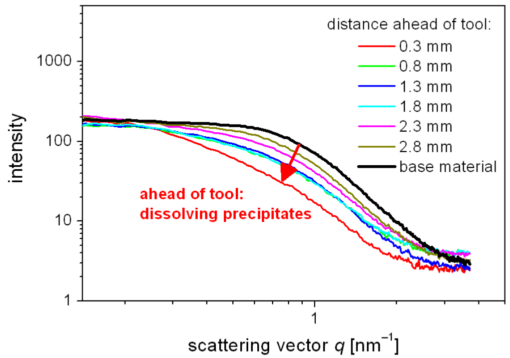
precipitate volume fraction
→ unique data for numerical models

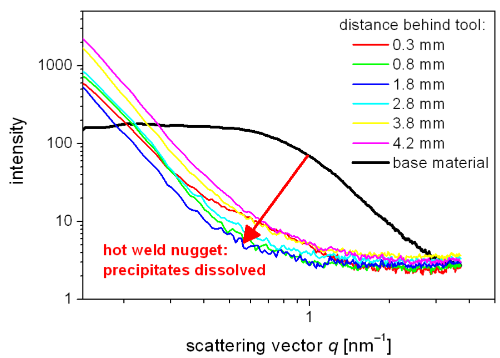


Highly versatile instrument for *in-situ* studies of:
different FSW methods, residual stress evolution, precipitation forming

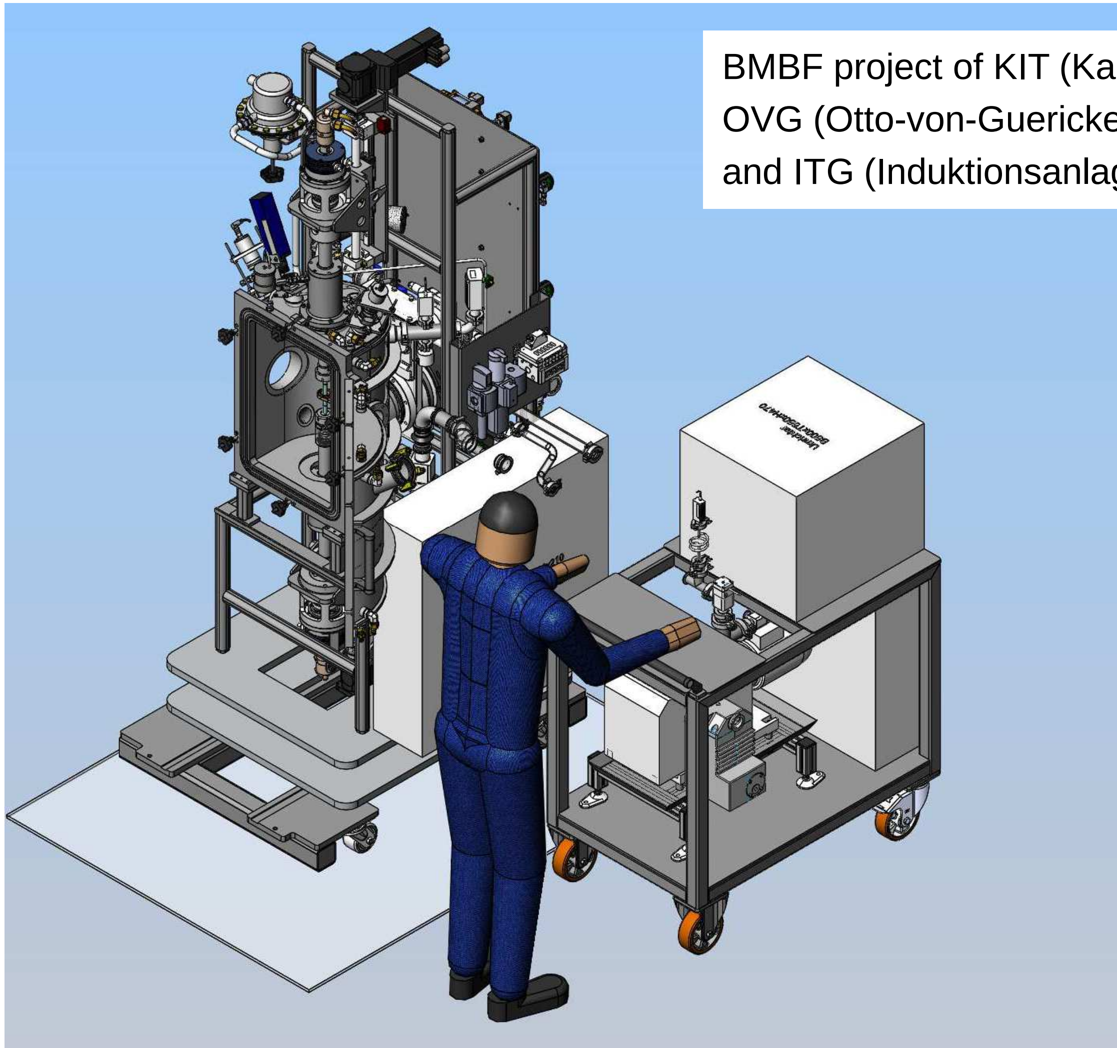


Size & density distribution of precipitates by *in-situ* SAXS





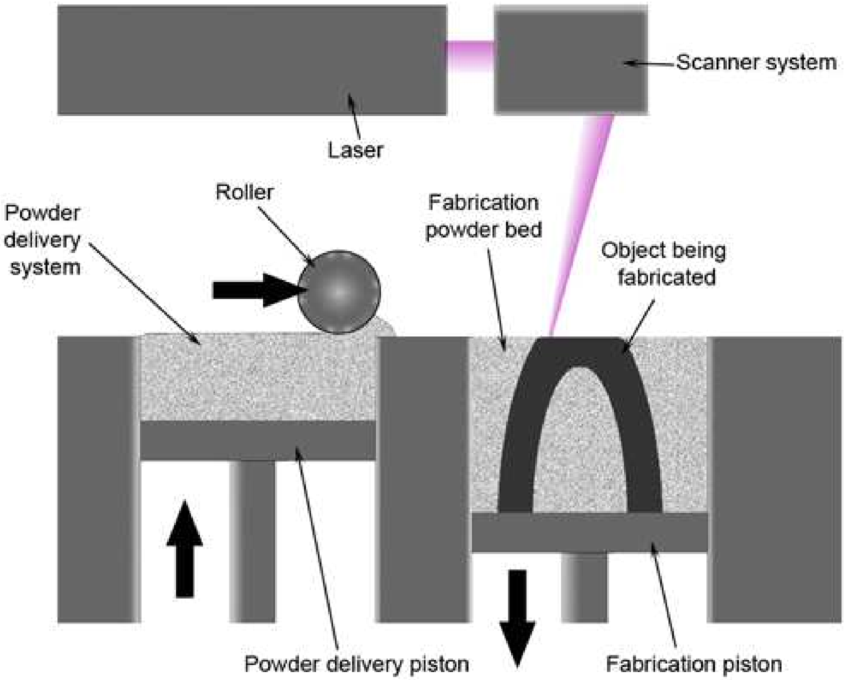
Future Trends: in-situ directional zone melting

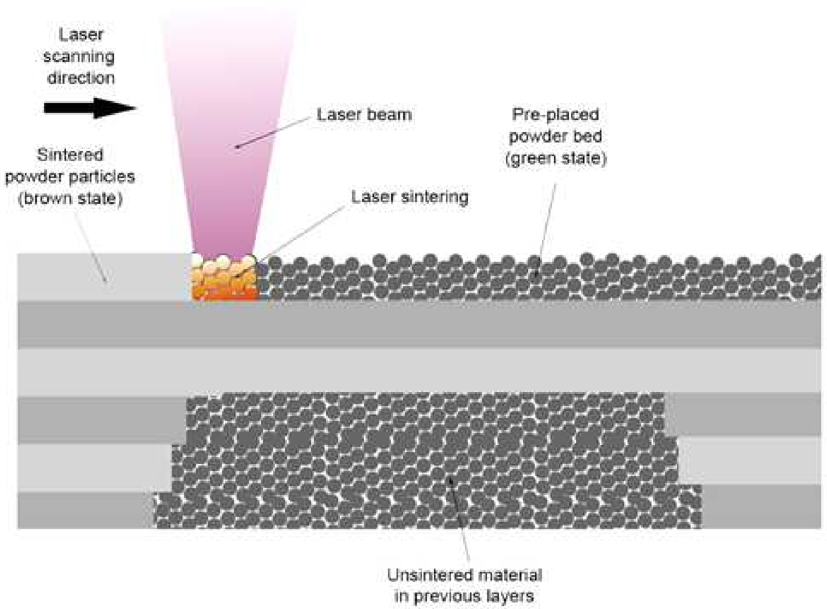


BMBF project of KIT (Karlsruher Institut für Technologie), OVG (Otto-von-Guericke-Universität Magdeburg), HZG and ITG (Induktionsanlagen GmbH).

Insights to the solidification of the aligned microstructure
and
clarification of the influence of varying solidification parameters (e.g. solidification velocity, temperature gradient) on the microstructure evolution in novel materials with melting points exceeding 1000 °C, e.g. Fe-Al, Ti-Al, Nb-Si-Cr, Mo-Si-B, Mo-ZrB_2.

Future Trends: 3D Printing - SLM (Selective Laser Melting)





Conclusion

HEMS / GEMS — works hard to offer **equipment to a larger community**
offers **expertise in complex *in-situ* sample environments**
explores and is open to future ideas


Thank You
© Jens Birch