Dilatometer for neutron diffraction in-situ investigations

Victoria Kononikhina, Andreas Stark, Peter Staron

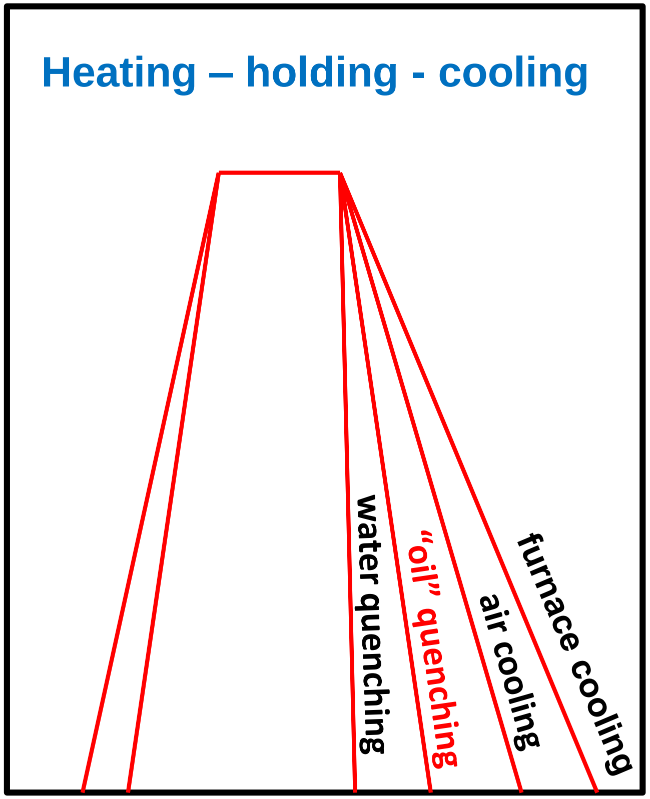
Institute of Materials Research, Helmholtz-Zentrum Geesthacht, Germany

14th May 2018: PNPI-TUM CREMLIN Workshop Peterhof, St. Petersburg

Helmholtz-Zentrum Geesthacht
Centre for Materials and Coastal Research

Motivation: in-situ industrial heat-treatment

Helmholtz-Zentrum Geesthacht
Centre for Materials and Coastal Research



The development of suitable hot-forming processes is an important step towards the serial production .

Several **microstructure parameters change** during processing – *in-situ* investigations are necessary.

Currently existed sample enviroments are restricted by cooling with a furnace.

Solution: Use dilatometer in-situ in a neutron beam!

DIL 805A/D at High-energy XRD at HZG beamline at DESY, Germany

Helmholtz-Zentrum Geesthacht
Centre for Materials and Coastal Research

- sample size: Ø 4–5 mm; *l* = 10 mm
- inductive heating (up to 1500 °C)

Wide heating range: up 4000°/s (for hollow samples)

Cooling rates up to quenching samples:

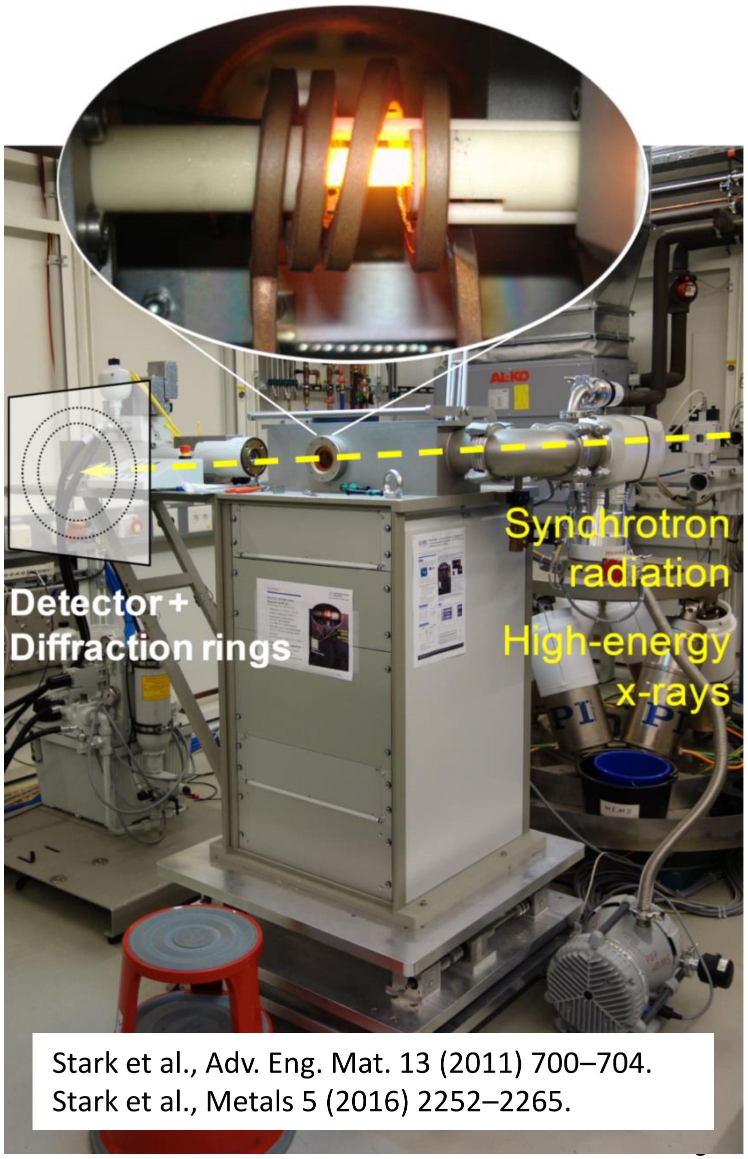
- quenching by blowing with gas
  (Ar, up to 100 °C·s^{-1} "Oil" quenching)

- deformation (compression, tension)
  with 0.01–125 mm·s^{-1} and max. 25 kN

Precise temperature control:

Resolution: $\Delta l = 0.05 \mu m,\ \Delta T = 0{,}05°C,$
$\Delta t = 0{,}0005 s$

- photon energy: 100 keV (λ = 0.124 Å).
- beam cross section: < 1 mm × 1 mm.
- 2D-detectors with frame rates up to 10 Hz.



Stark et al., Adv. Eng. Mat. 13 (2011) 700–704.
Stark et al., Metals 5 (2016) 2252–2265.

The dilatometer is an universal equipment for *in-situ* thermo-mechanical experiments

Helmholtz-Zentrum Geesthacht
Centre for Materials and Coastal Research

Alloy systems studied since 2009:

- γ-TiAl based alloys
- Mg alloys
- Co-based superalloys
  Co-Re-based alloys
- NiTi shape memory alloys
- Steel (e.g. TRIP steel)
- Ti- or Al- based alloys

ELSEVIER
Acta Materialia
Volume 94, 1 August 2015, Pages 78–86

Plasticity analysis by synchrotron radiation in a $Mg_{97}Y_2Zn_1$ alloy with bimodal grain structure and containing LPSO phase

G. Garces[a], D.G. Morris[a], M.A. Muñoz-Morris[a], P. Perez[a], D. Tolnai[b], C. Mendis[b], A. Stark[b], H.K. Lim[c], S. Kim[c], N. Schell[d], P. Adeva[a]

ELSEVIER
Journal of Alloys and Compounds
Volume 632, 25 May 2015, Pages 110–115

The effect of tungsten content on the properties of $L1_2$-hardened Co–Al–W alloys

Florian Pyczak[a], Alexander Bauer[b], Mathias Göken[b], Uwe Lorenz[a], Steffen Neumeier[b], Michael Oehring[a], Jonathan Paul[a], Norbert Schell[a], Andreas Schreyer[a], Andreas Stark[a], Felix Symanzik[c]

ELSEVIER
Acta Materialia
Volume 80, November 2014, Pages 118–131

In situ synchrotron X-ray diffraction and dilatometric study of austenite formation in a multi-component steel: Influence of initial microstructure and heating rate

V.A. Esin[a, b], B. Denand[a, c], Qu. Le Bihan[a, c], M. Dehmas[a, c], J. Teixeira[a, c], G. Geandier[a, c], S. Denis[a, c], T. Sourmail[d], E. Aeby-Gautier[a, c]

ELSEVIER
Intermetallics
Volume 50, July 2014, Pages 94–107

Mechanical and functional behavior of a Ni-rich $Ni_{50.3}Ti_{29.7}Hf_{20}$ high temperature shape memory alloy

O. Benafan[a], A. Garg[b, a], R.D. Noebe[a], G.S. Bigelow[a], S.A. Padula II[a], D.J. Gaydosh[c, a], N. Schell[d], J.H. Mabe[e], R. Vaidyanathan[f]

Metals **2015**, *5*(4), 2252-2265; doi:10.3390/met5042252
Feature Paper | Open Access

Article

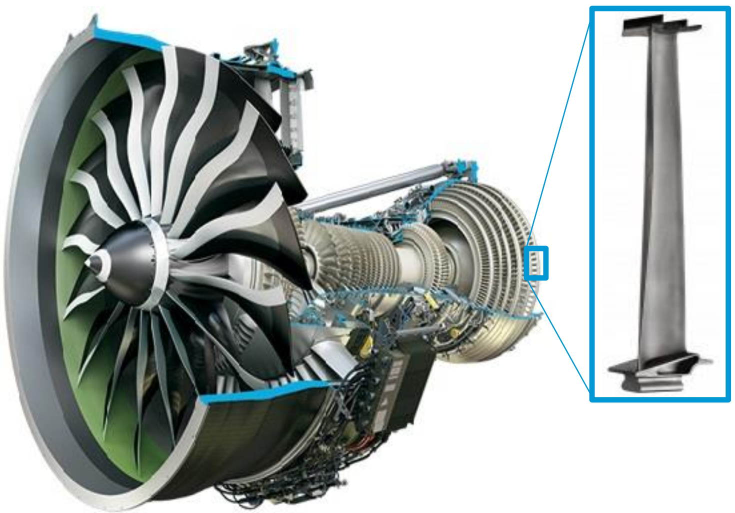
***In Situ* High-Energy X-ray Diffraction during Hot-Forming of a Multiphase TiAl Alloy**

Andreas Stark [1,*], Marcus Rackel [1], Aristide Tchouaha Tankoua [2], Michael Oehring [1], Norbert Schell [1], Lars Lottermoser [1], Andreas Schreyer [1] and Florian Pyczak [1]

Initial slide produced by P. Staron

γ-TiAl based alloys: lightweight structural materials for high-temperature applications

- low density, ~4 g/cm^3 (50% density of Ni-based superalloys)
- high specific strength up to 750 °C (~200 °C higher than Ti-based alloys)



TiAl low pressure turbine blades, last stages of the GEnx engine

http://www.airbus.com

http://www.airbus.com

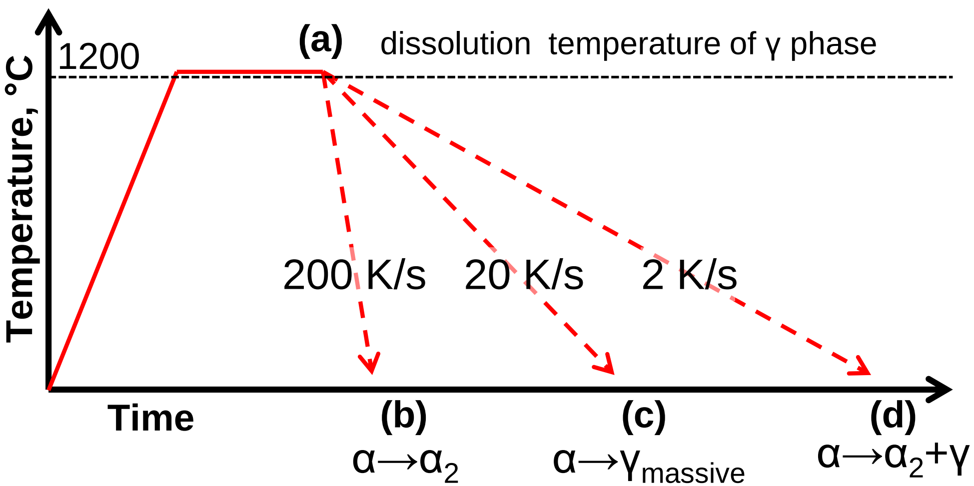
- **the blade is a forged product**
- **establishment of "forging window" for new alloys required**

Continuous Cooling Transformation (CCT)

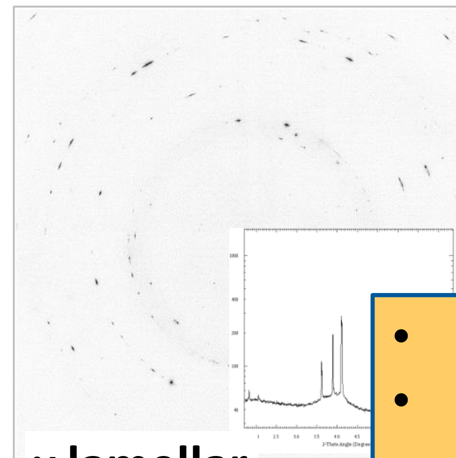
Helmholtz-Zentrum Geesthacht
Centre for Materials and Coastal Research

- **fast and controlled cooling**

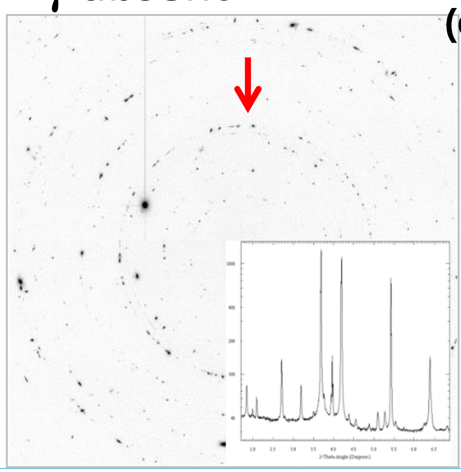
Ti-45Al-4Nb-4Ta (at. %)



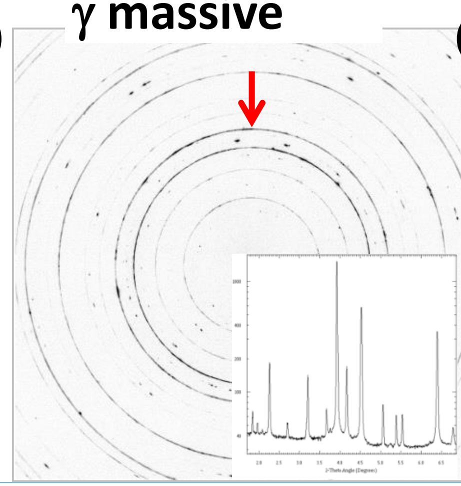
(a)



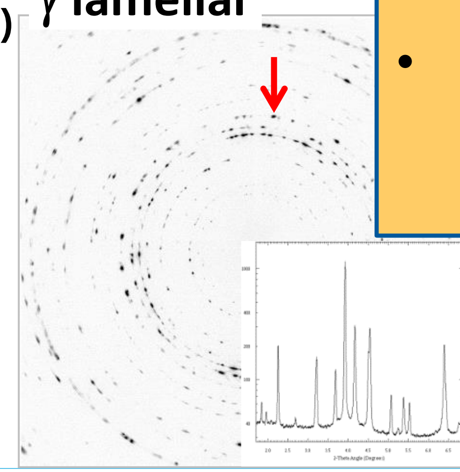
(b) γ absent



(c) γ massive



(d) γ lamellar



- phase content
- microstructure

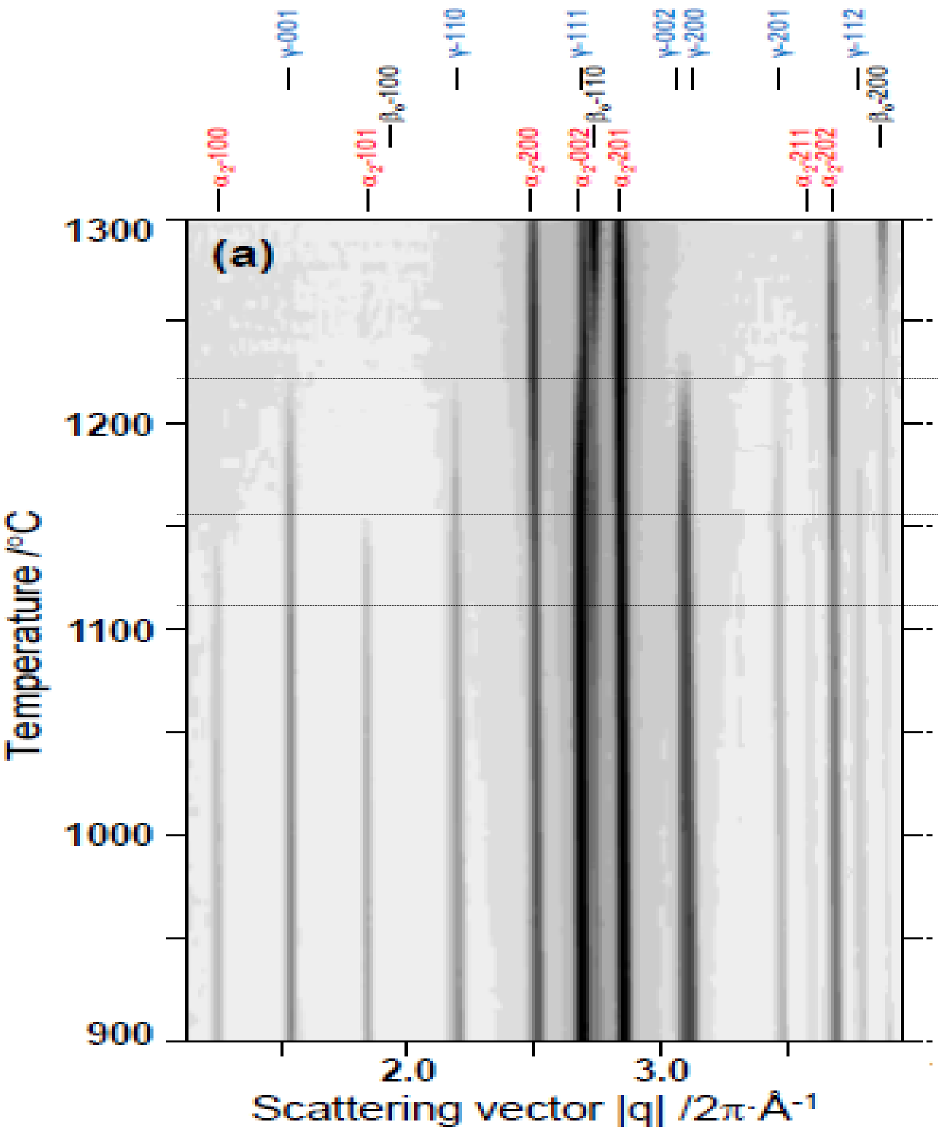
⇩

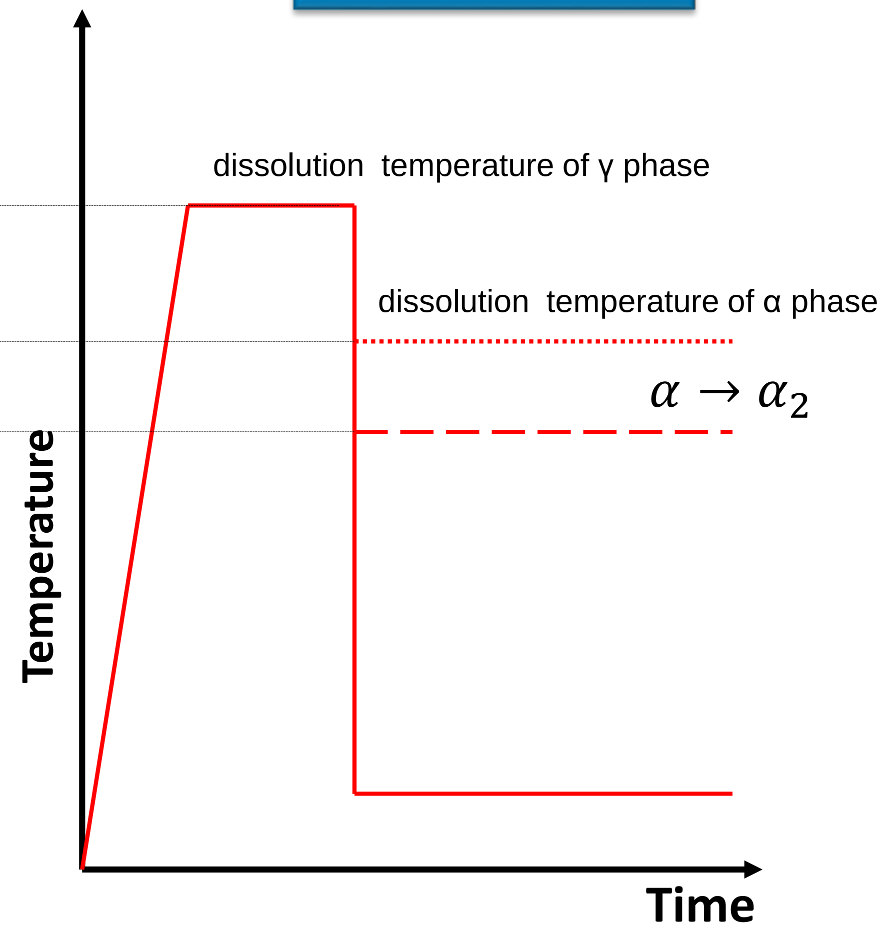
- mechanical properties predictions

Modeling of the Time Temperature Transformation (TTT) diagramms for TiAl

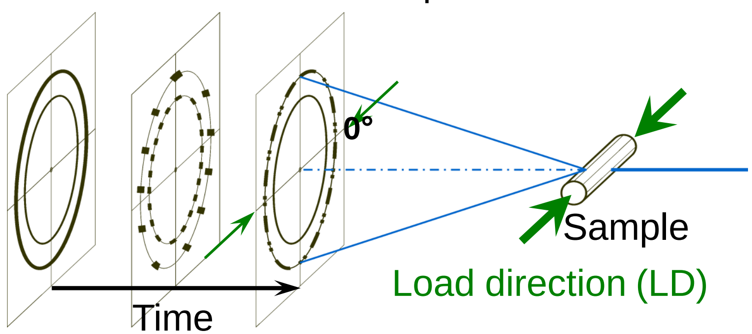
Helmholtz-Zentrum Geesthacht
Centre for Materials and Coastal Research

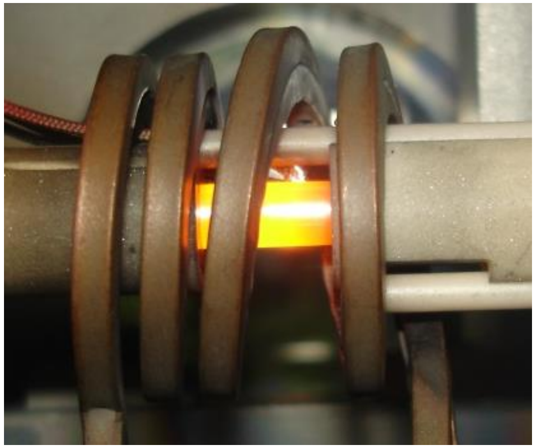
Ti-42Al-8.5Nb (at. %)



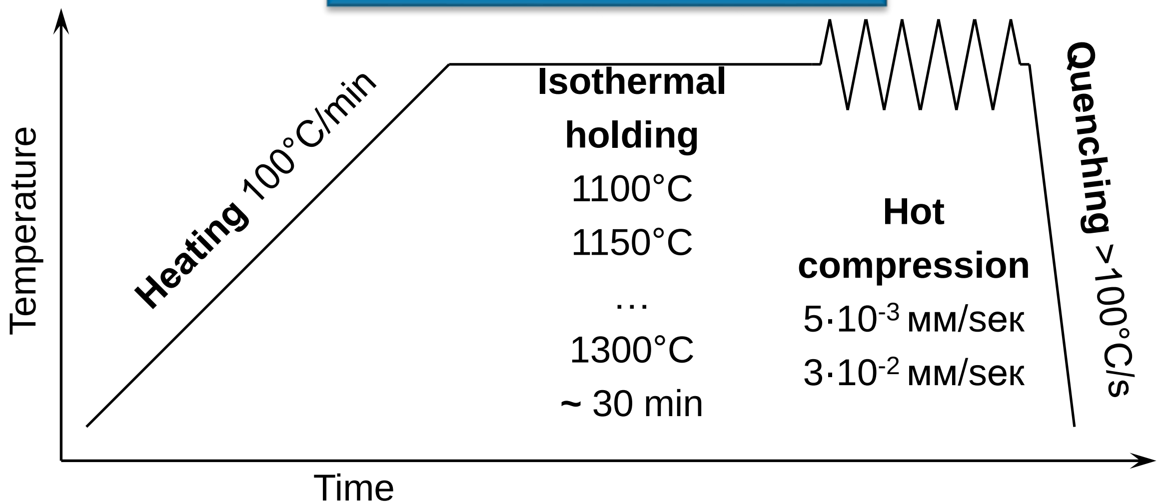


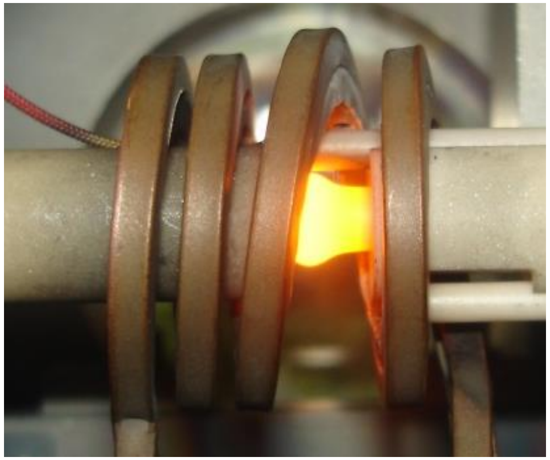
Hot-compression test





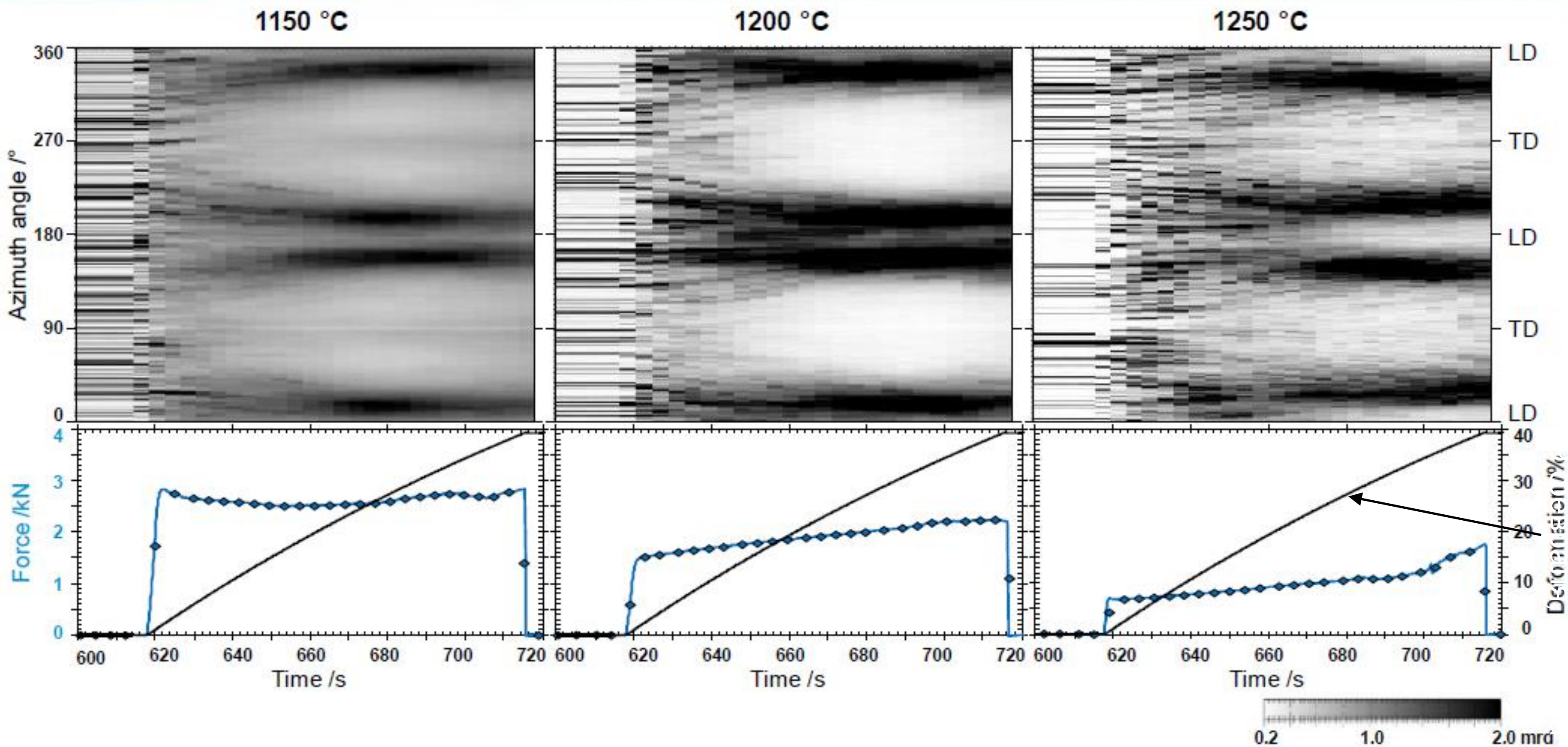
Ti-42Al-8.5Nb (at. %)





In-situ hight-energy X-ray diffraction during hot-forming of a multiphase TiAl alloy. A. Stark, M. Rackel at al. *Metals* 2015, 5

Texture changes during hot-forming process

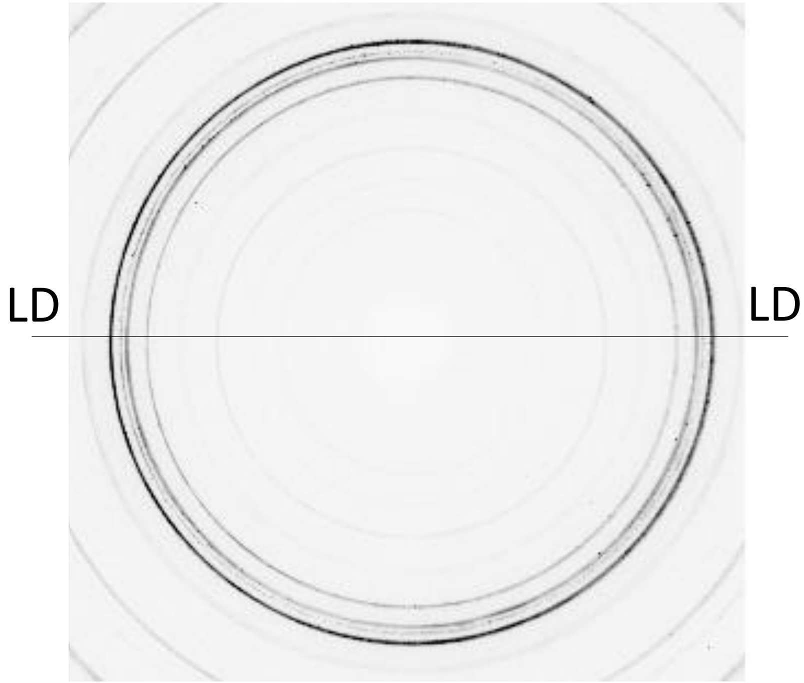


Intensity distribution over azimuth angle as a function of time for the alpha 002 reflection during hot-forming process at different temperatures with deformation rate of 0.005 mm/s.

Texture evolution during hot compression

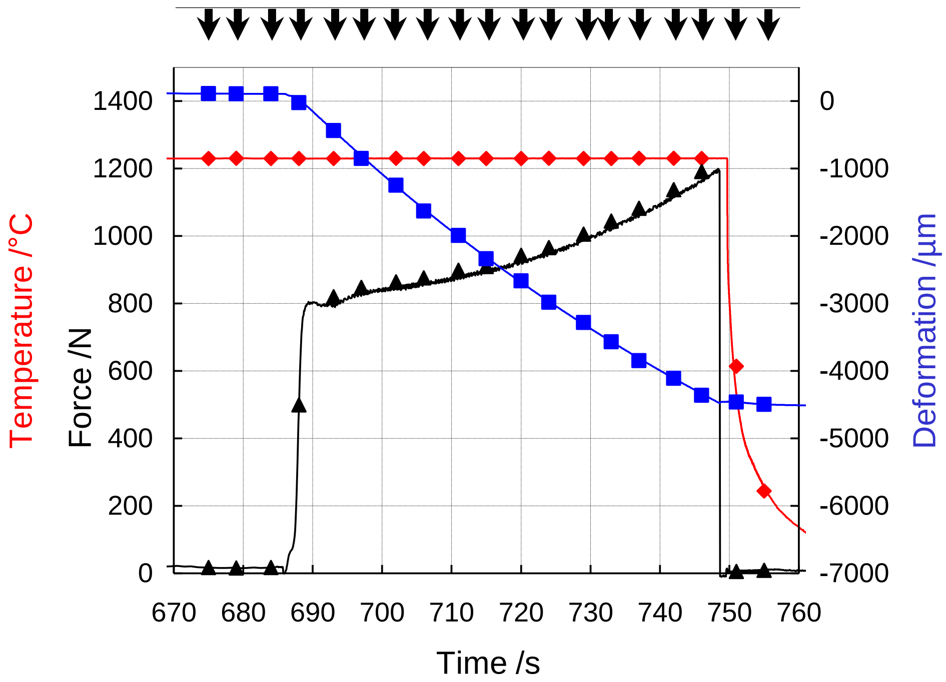
Helmholtz-Zentrum Geesthacht
Centre for Materials and Coastal Research

Slow ($1\cdot10^{-2}$ s^{-1}) deformation @ 1230 °C of Ti-43Al-4Nb-1Mo-0.1B (in at.%)



Debye-Scherrer diffraction rings

Phase fractions: 89 vol.% α 11 vol.% β

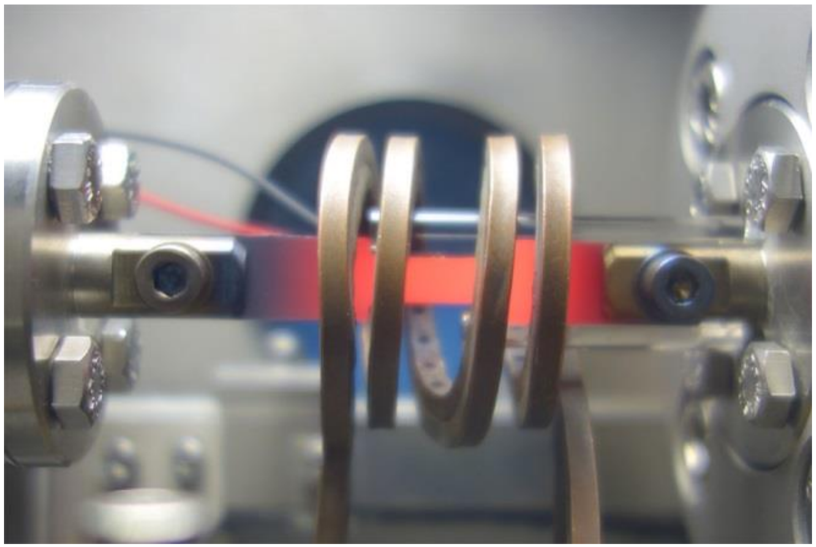


Processing parameters

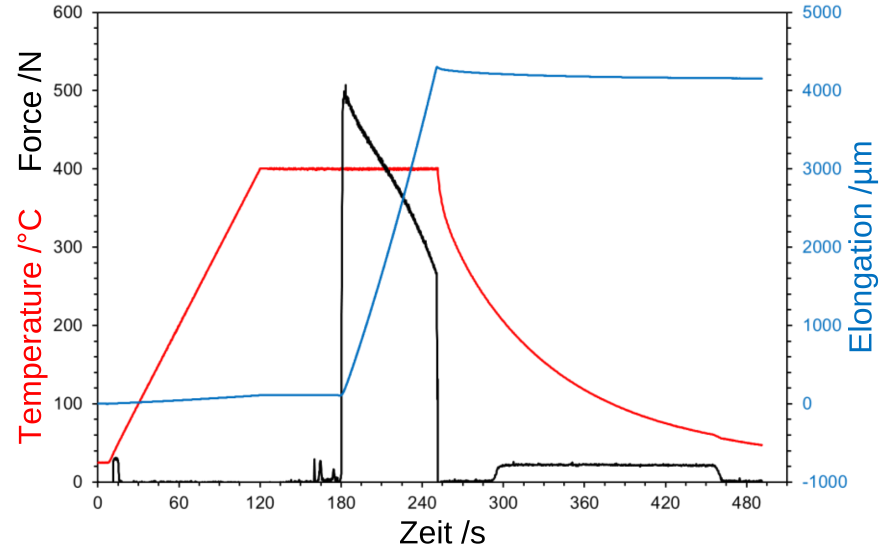
Tensile test

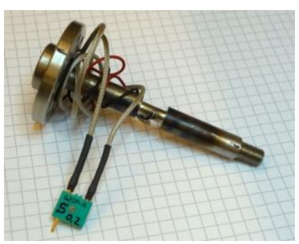
Helmholtz-Zentrum Geesthacht
Centre for Materials and Coastal Research

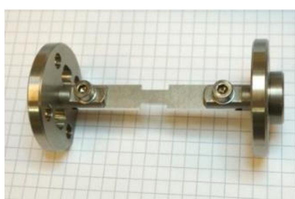
Sheet steel / 900°C

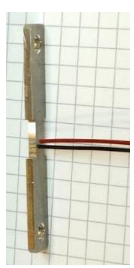


Al-alloys / 400°C



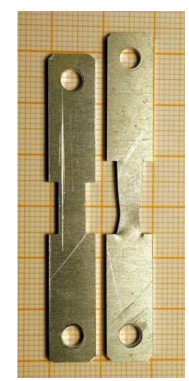






Round tensile specimens / **flat** tensile specimens (sheet metal)

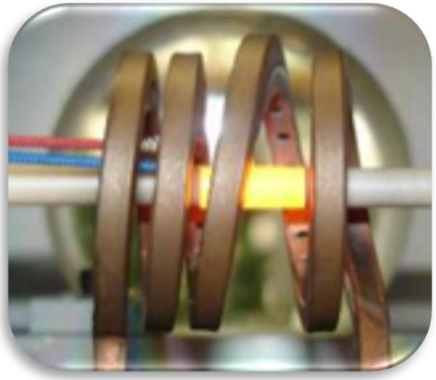
- Forming path: - Pull: up to 9 mm - Pull/ pressure ± 5 mm
- Maximum force: 10 kN
- Strain rates: 0.001 to 2.0 s-1



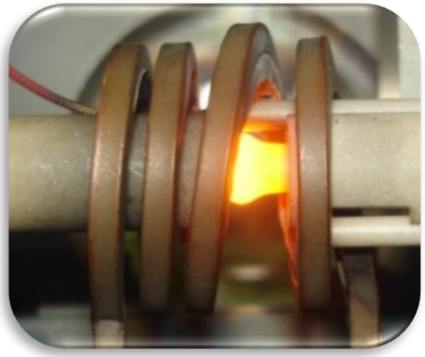
Possible experiments with dilatometer at high temperatures:

Helmholtz-Zentrum Geesthacht
Centre for Materials and Coastal Research

- Quenching



- Compression



- Tension

Neutron Instruments at FRM II

The dilatometer is going to be combined with two neutron Instruments: STRESS-SPEC and SANS-1

3.5 Å – 30 Å **SANS-1**

STRESS-SPEC **1 Å – 2.4 Å**

(TUM, HZG) A. Heinemann

Neutrons
vs
Synchrotron
=
Different contrast

(TUM, HZG) Weimin Gan

Neutron Dilatometer at FRM II

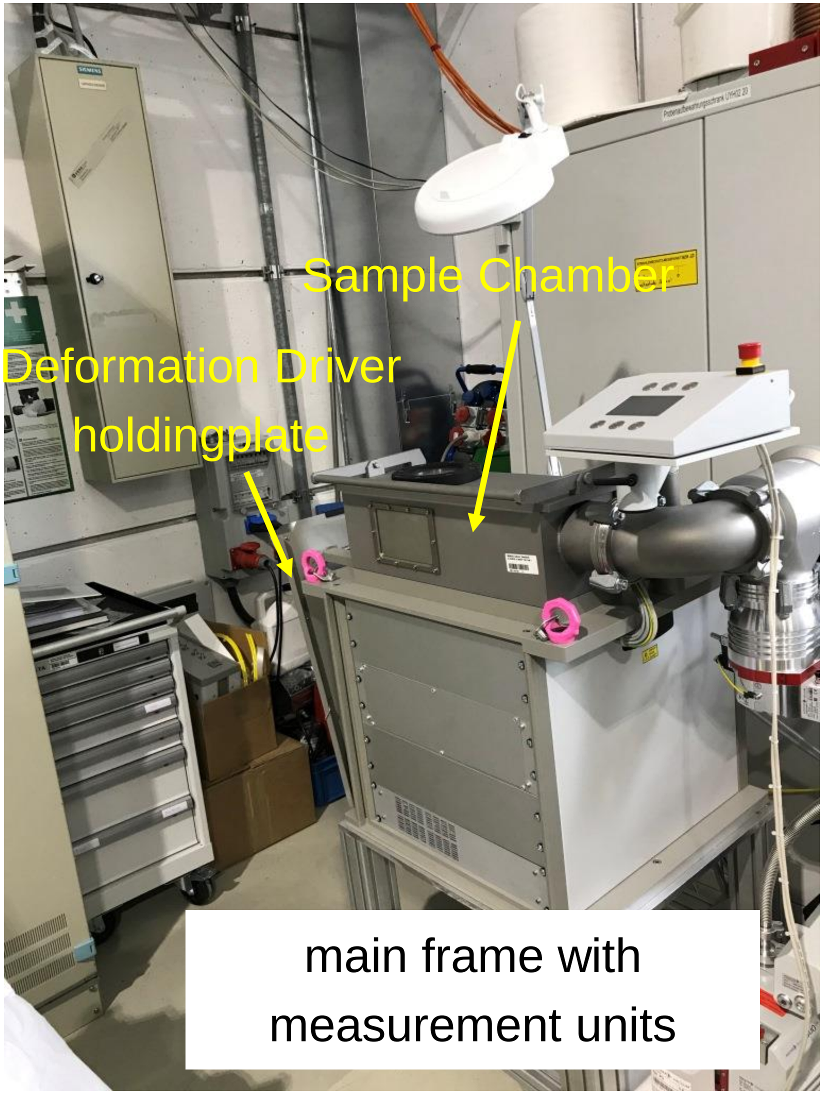


Foto from Weimin Gan

Difference between synchrotron and neutron dilatometers:

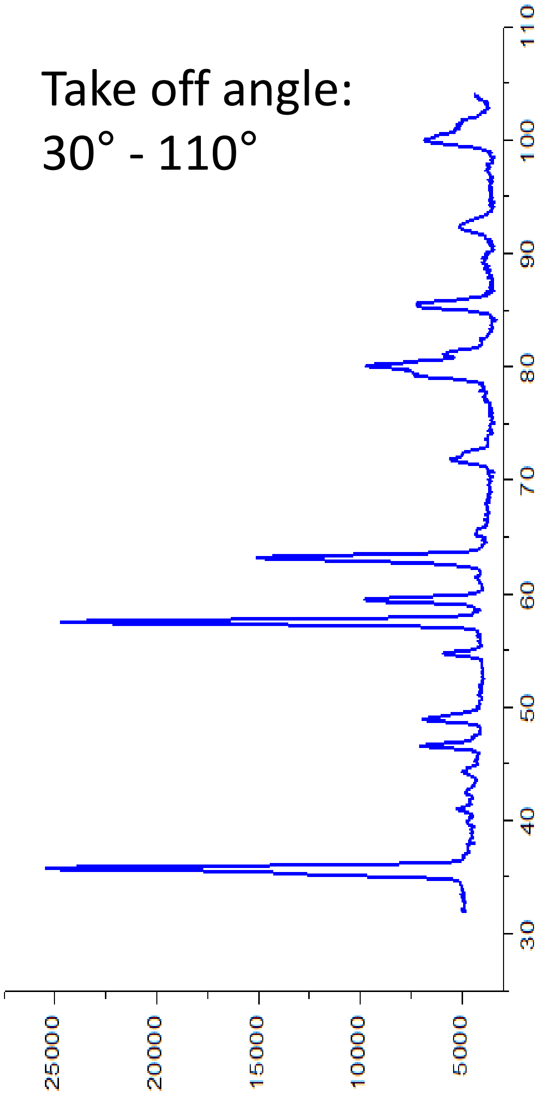
- Windows shape and material
- Coil shape
- Blowing gas mechanism

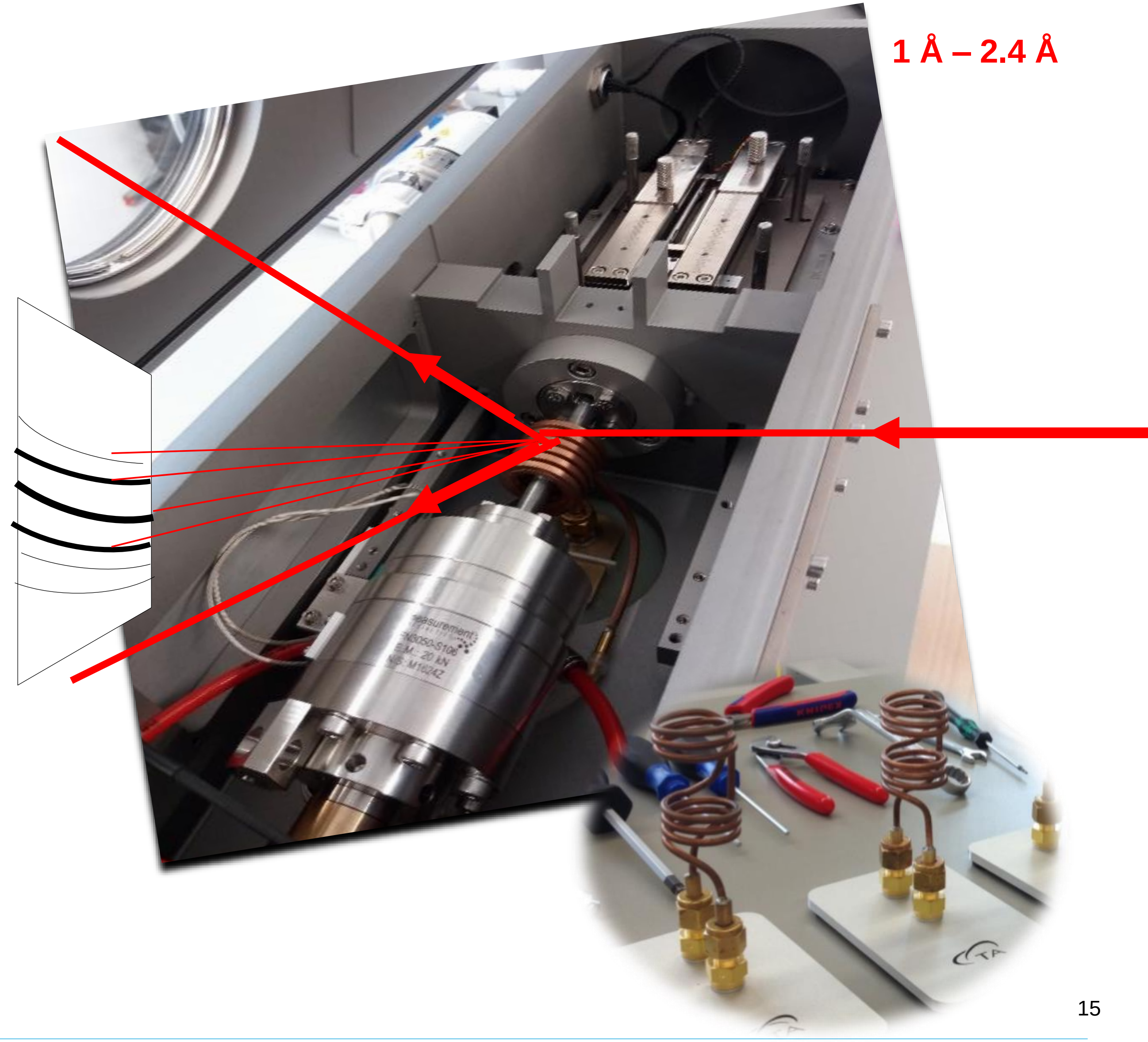
- Hight of the main frame with measurement units

Dilatometer at STRESS-SPEC

Helmholtz-Zentrum Geesthacht
Centre for Materials and Coastal Research

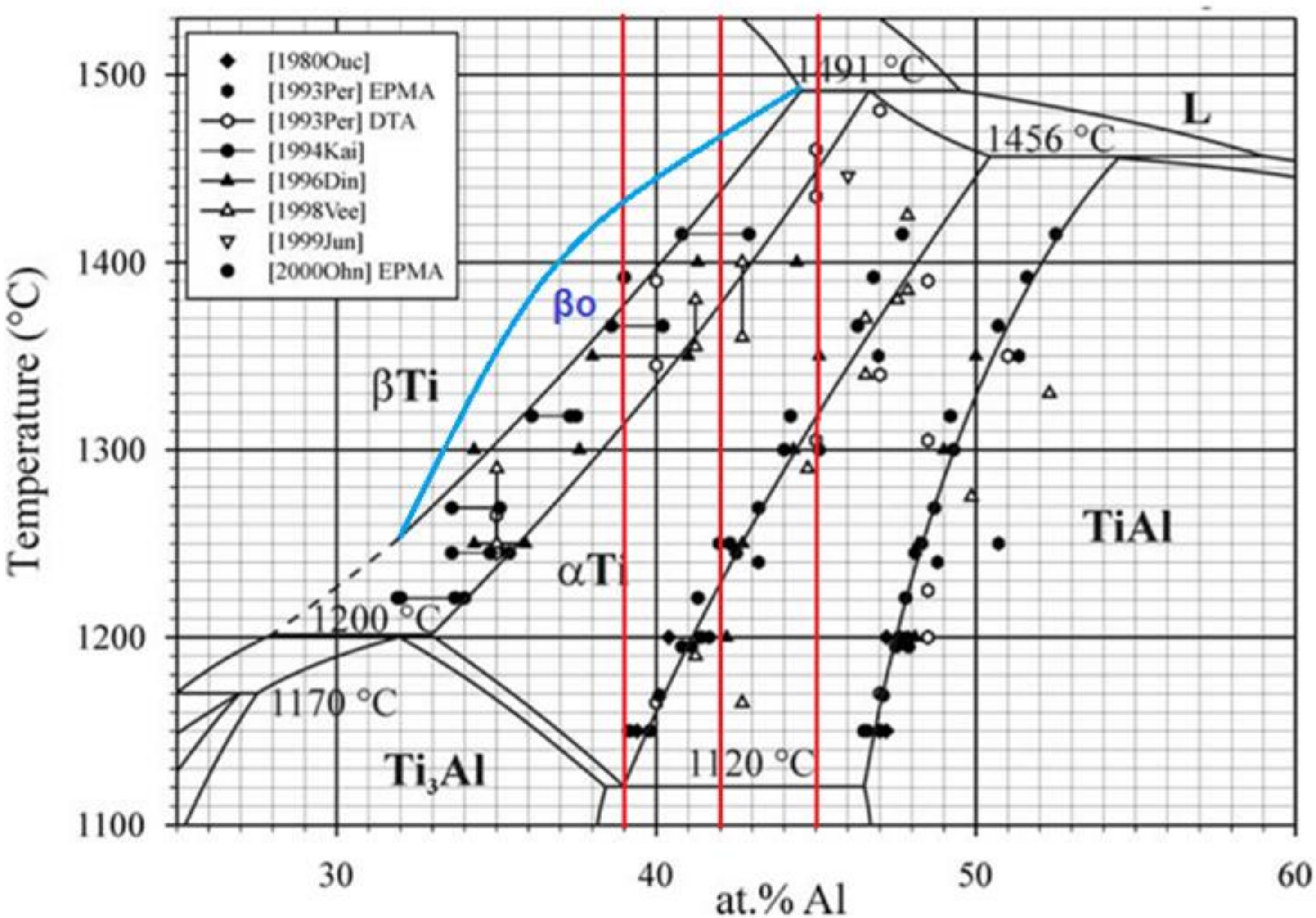
Take off angle:
30° - 110°



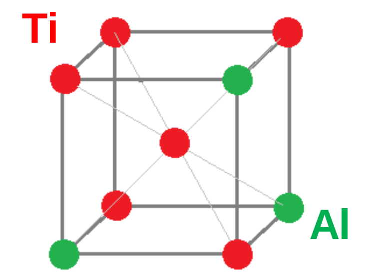


Neutron applications: Order–disorder transformations in TiAl

Helmholtz-Zentrum Geesthacht
Centre for Materials and Coastal Research

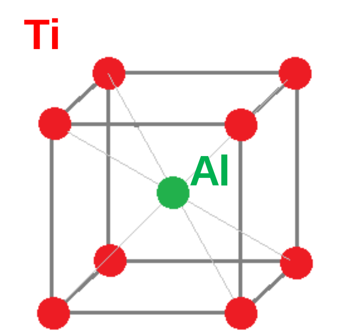


Binary TiAl phase diagramm. Julius C. Schuster and Martin Palm, Journal of Phase Equilibria and Diffusion, Vol. 27 No. 3 (2006).



Disordered Beta TiAl
Ti and Al have random distribution at atom positions

or

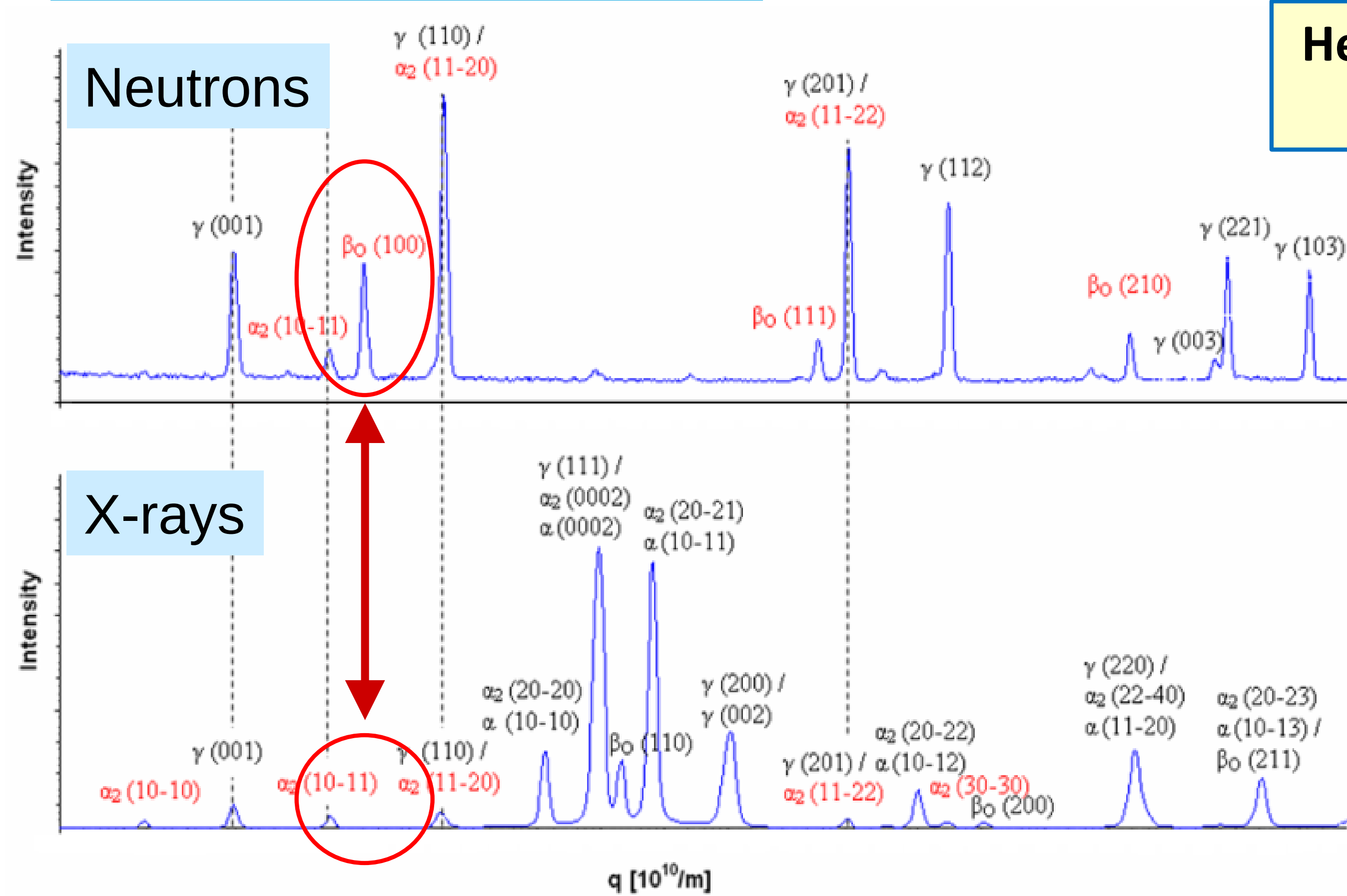


Ordered Beta TiAl
Positions of Ti and Al are determined

Neutron applications: TiAl alloys

Helmholtz-Zentrum Geesthacht
Centre for Materials and Coastal Research

Helmut Clemens, MU Leoben



SPODI@FRM II
λ = 1.55 Å

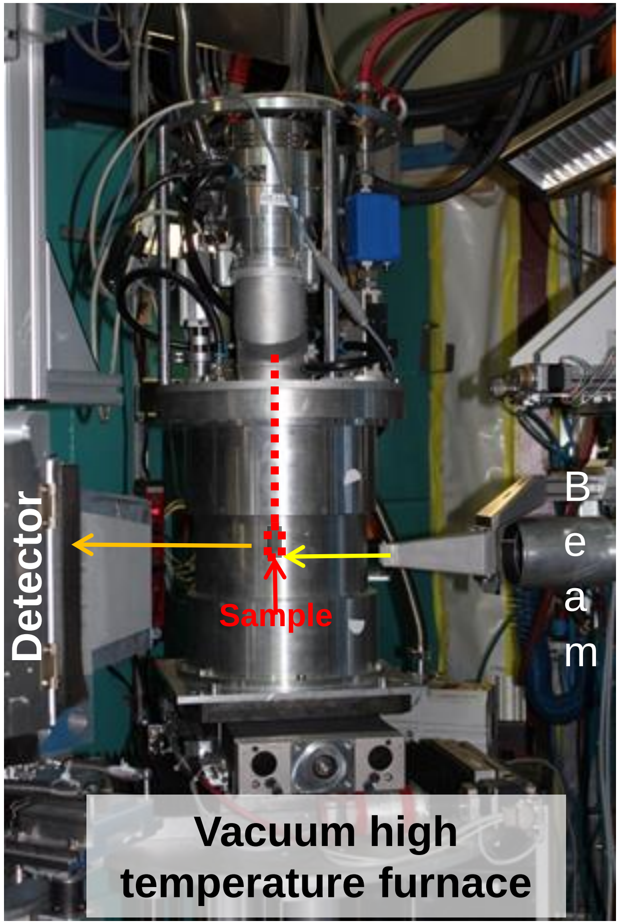
ID15@ESRF
E = 87 keV

… only neutrons allow the study of order–disorder transformations in TiAl alloys, e.g. of the β/β_o-phases. •$b_{Ti} = -3,370\ fm$; $b_{Al} = 3,449\ fm$

L. Drössler, Diploma Thesis, MU Leoben (2008).

Initial slide produced by P. Staron

Neutron applications: Order–disorder transformations

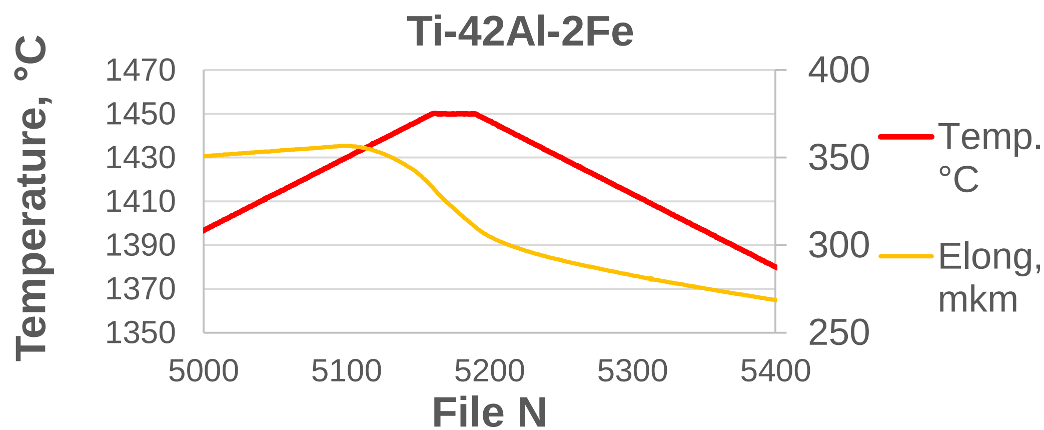


STRESS-SPEC@MLZ

Temperature range:
1100 – 1450 °C

Heating ramp:
20 K/min

12h per sample:
8 h Measurements + 2 h heating and 2 h of cooling

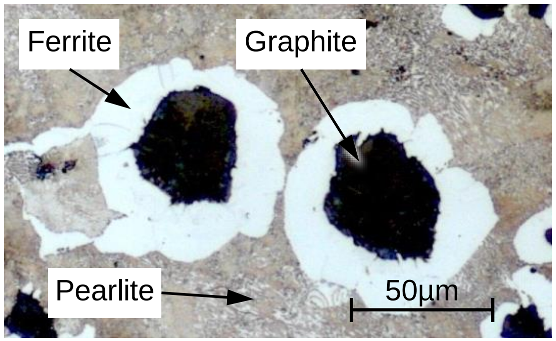


Dilatometer:
→ Faster and controlled heating and cooling for full temperature range of interest
→ Dilatometer signal can detect sample melting

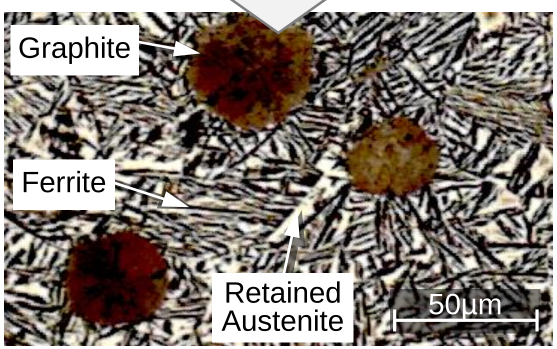
Neutron applications: Study of Austempered Ductile Iron (ADI)

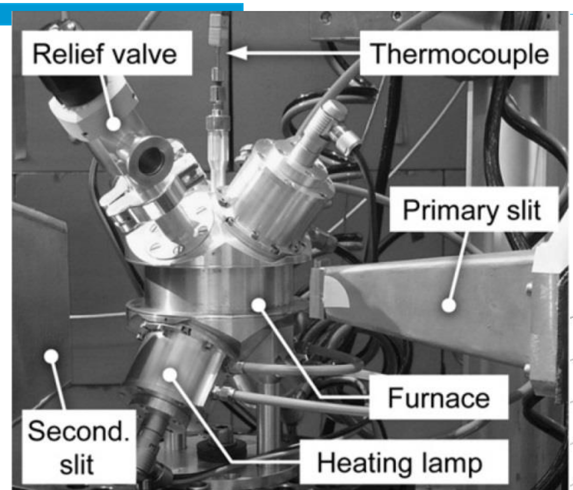
Helmholtz-Zentrum Geesthacht
Centre for Materials and Coastal Research

Michael Hofmann, MLZ/TUM

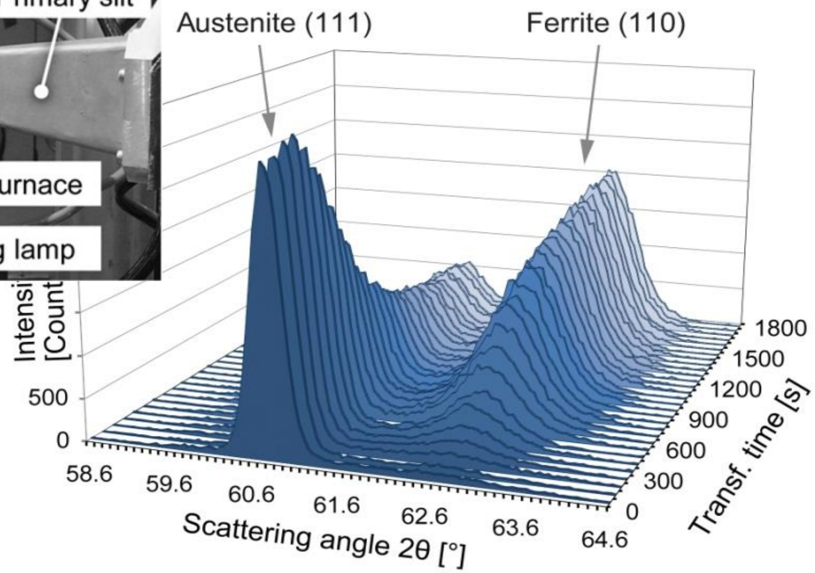


Heat Treatment





The mirror furnace at FRM-II



Diffraction yields:

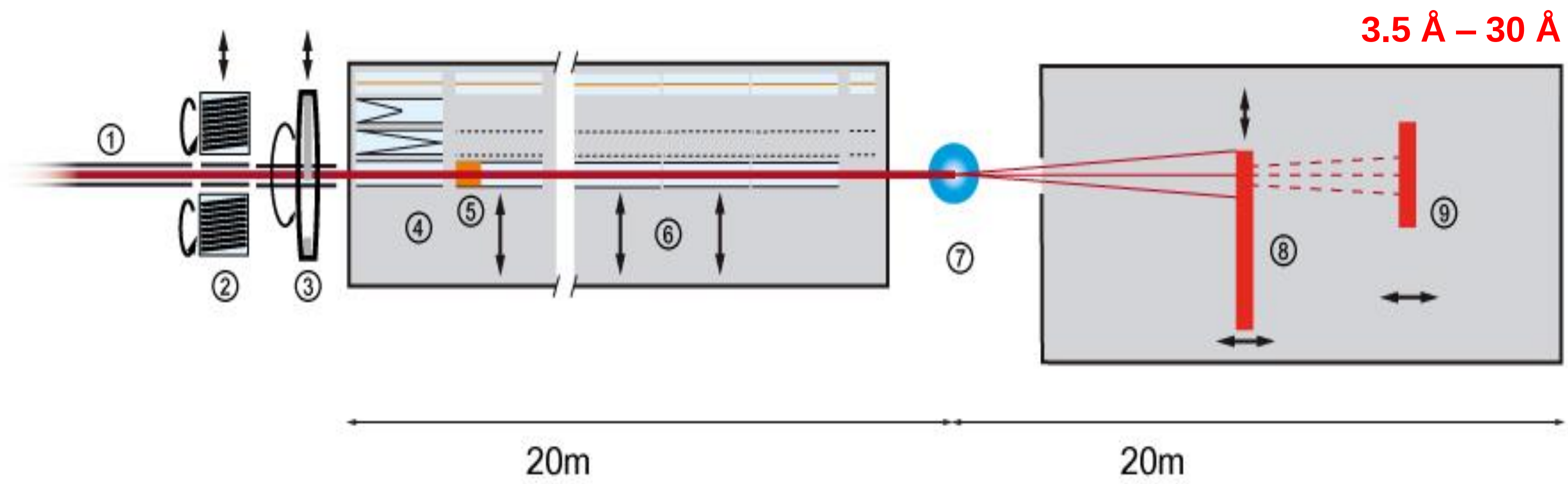
- phase fractions during tempering
- carbon content (aust. latt. param).
- morphology via peak width

Dilatometer:
→ Faster and controlled cooling to processing temperature possible
→ Dilatometer signal

L. Meier, M. Hofmann, P. Saal, W. Volk, H. Hoffmann, Materials Characterization 85 (2013) 124–133.

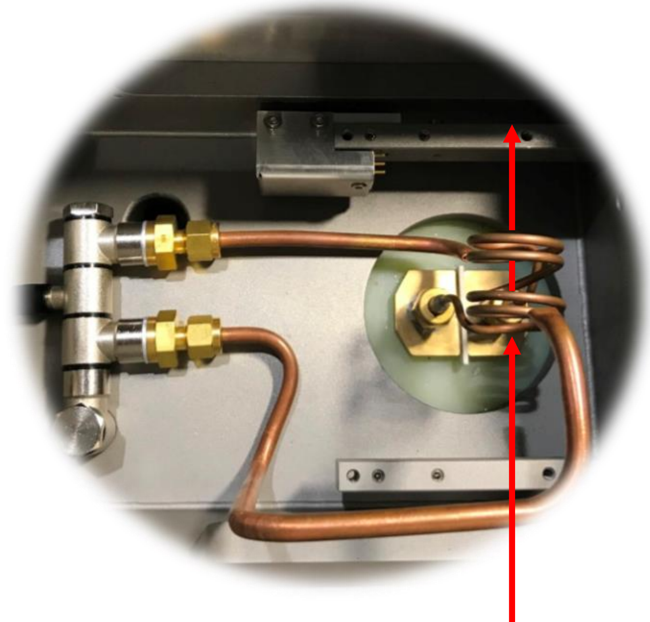
Dilatometer at SANS-1

Helmholtz-Zentrum Geesthacht
Centre for Materials and Coastal Research



•Precipitates and segregation in alloys

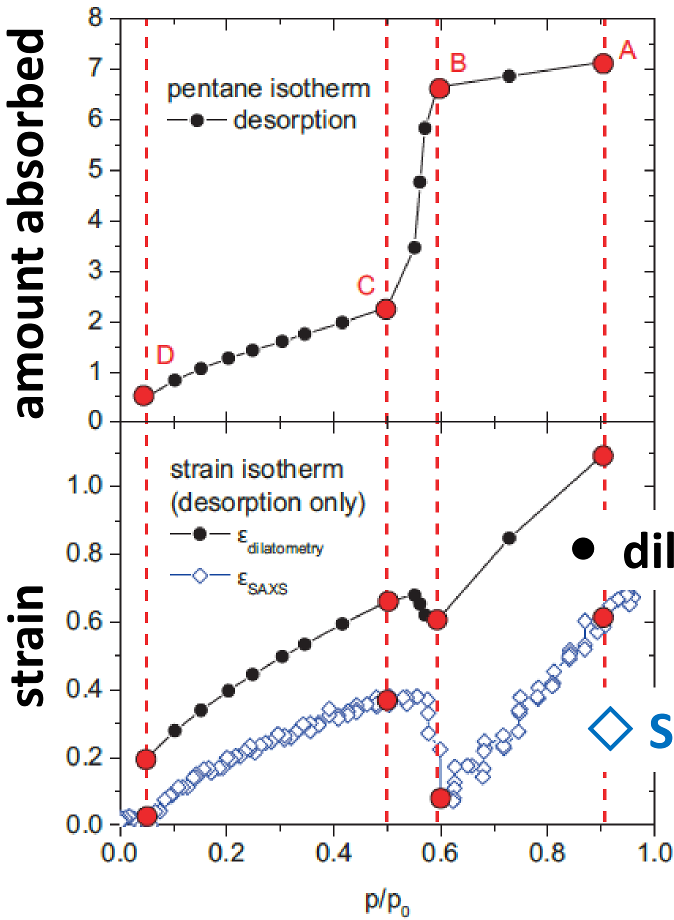
•Defects in materials



Study of Sorption in monoliths with hierarchical pore structure

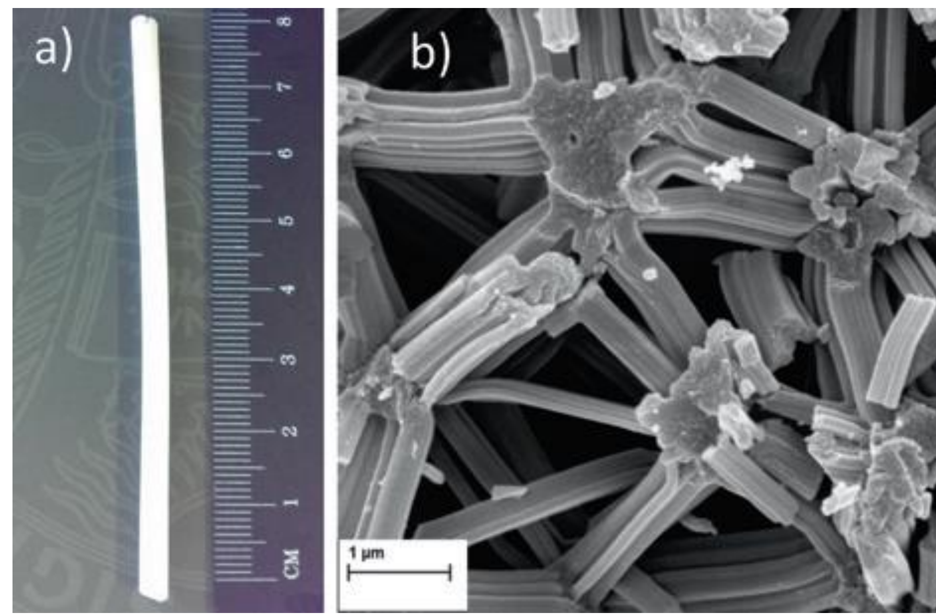
Helmholtz-Zentrum Geesthacht
Centre for Materials and Coastal Research

Oskar Paris, MU Leoben



● **dilatometry**

◇ **SAXS**



→ Potential for simultaneous in-situ SANS and dilatometry

*C. Balzer, R. Morak, M. Erko, C. Triantafillidis, N. Hüsing, G. Reichenauer, O. Paris, *Relationship between pore structure and sorption-induced deformation in hierarchical silica-based monoliths*, Z. Phys. Chem. (2015) 1–21; DOI 10.1515/zpch-2014-0542.

Summary:

Helmholtz-Zentrum Geesthacht
Centre for Materials and Coastal Research

- The dilatometer is a unique equipment that allows performing in-situ diffraction measurements under heat treatment and **deformation** conditions up to 1500°C

- The dilatometer enables not only fast and controlled heating, but also fast and **controlled cooling or quenching**!

- **Thus, the use of a dilatometer is of high importance for improvement of industrial materials processing technologies**

Acknowledgements

Helmholtz-Zentrum Geesthacht
Centre for Materials and Coastal Research

For materials of dilatometer use:

Dr. Markus Rackel (HZG)

Dr. Weimin Gan (HZG)

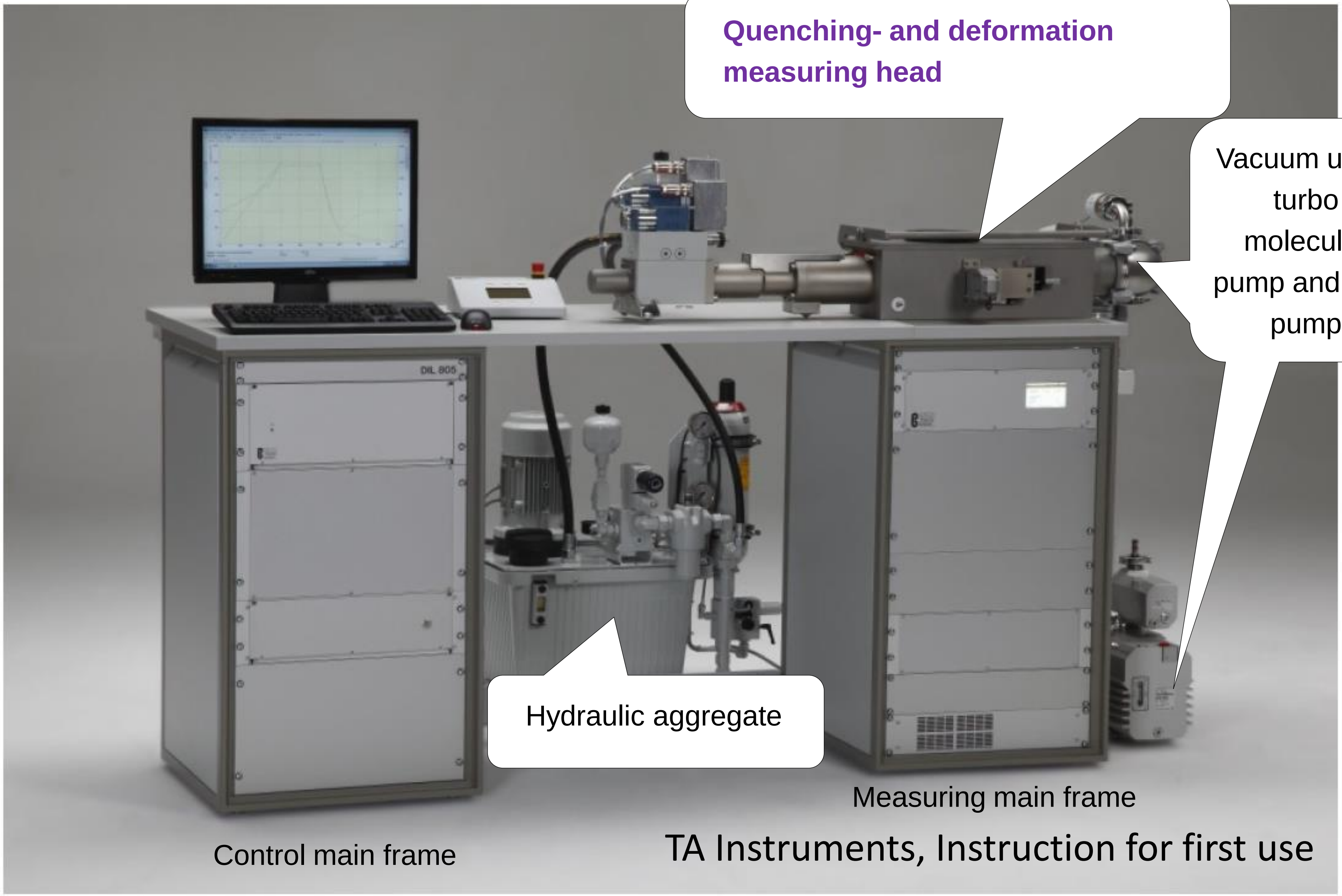
Dr. Michael Hofmann

Petra Erdely, Dr. Helmut Clemens (MU Leoben)

Thank you for your attention!

Dilatometer – an equipment for *reproduction* heat treatment conditions

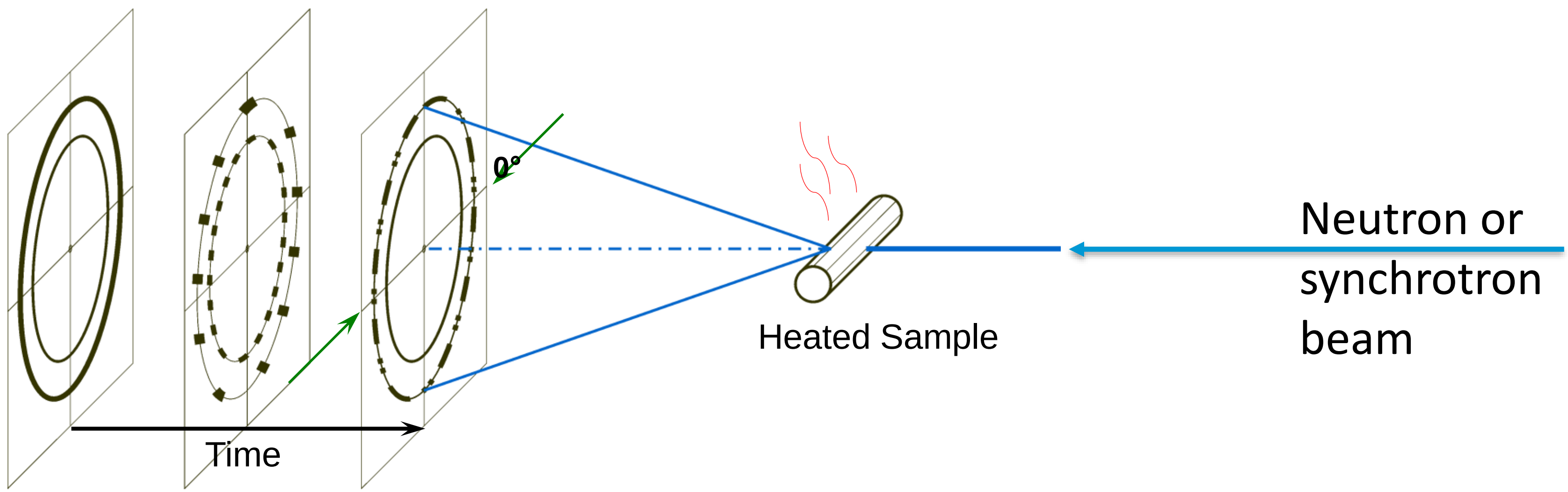
Helmholtz-Zentrum Geesthacht
Centre for Materials and Coastal Research



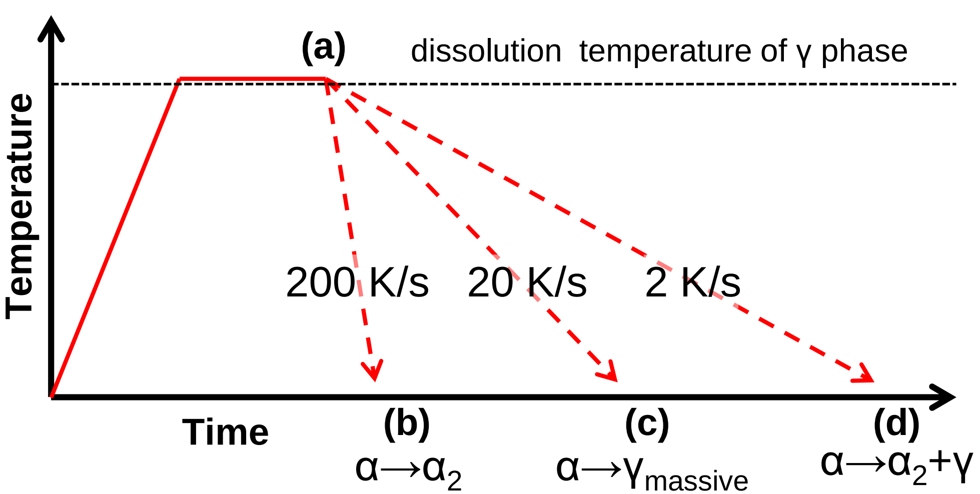
TA Instruments, Instruction for first use

In-situ phase analysis for TTT or CCT diagramms

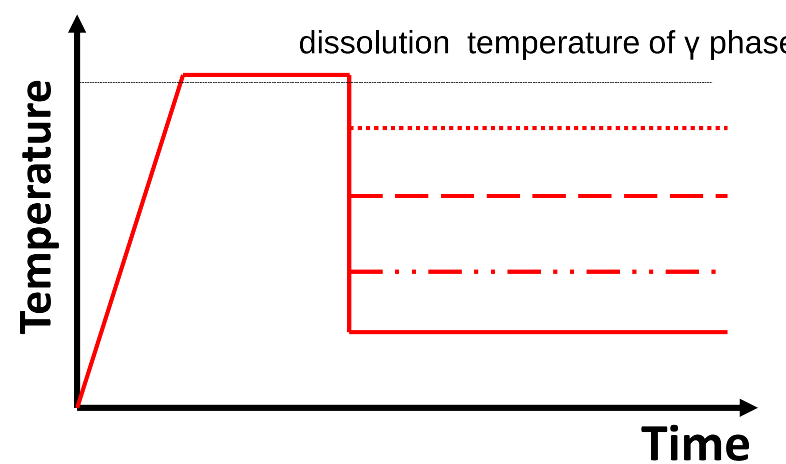
Helmholtz-Zentrum Geesthacht
Centre for Materials and Coastal Research



Continuous Cooling Transformation (CCT)



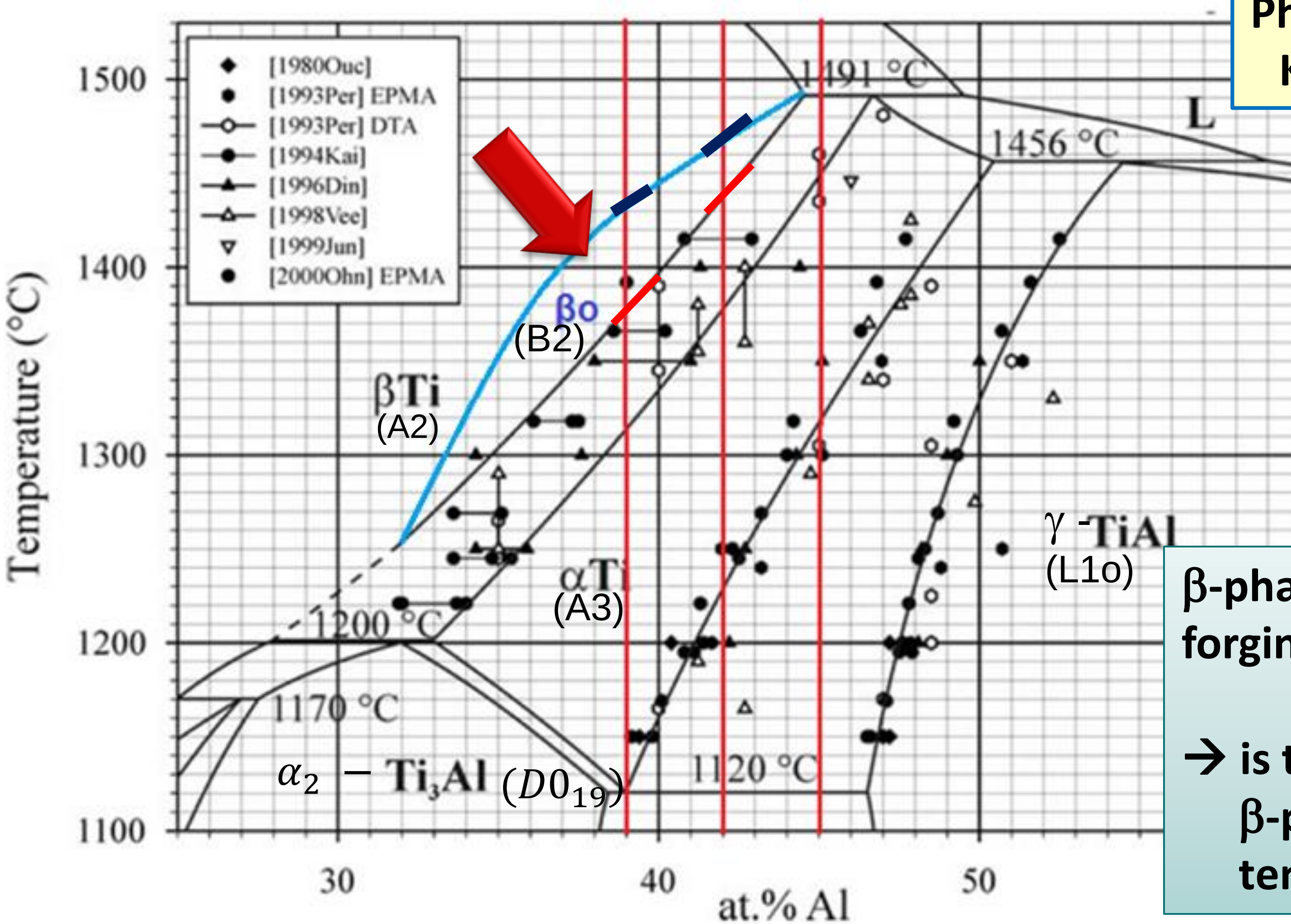
Time Temperature Transformation (TTT)



Neutron applications: Order–disorder transformations

Helmholtz-Zentrum Geesthacht
Centre for Materials and Coastal Research

PhD work of Viktoria Kononikhina, HZG



Ti-42Al
+
β-stabilizing elements:
Fe, Mo, Cr, Nb, Ta

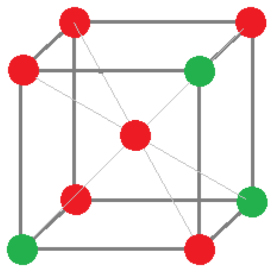
β-phase important for forging.

→ is there ordered β-phase at high temperatures?

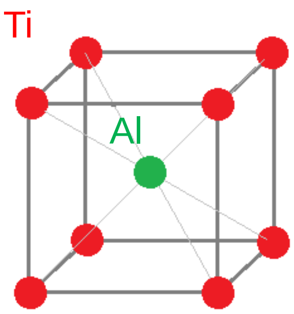
Binary TiAl phase diagramm. Julius C. Schuster and Martin Palm, Journal of Phase Equilibria and Diffusion, Vol. 27 No. 3 (2006).

Ordering and disordering and a Structure factor

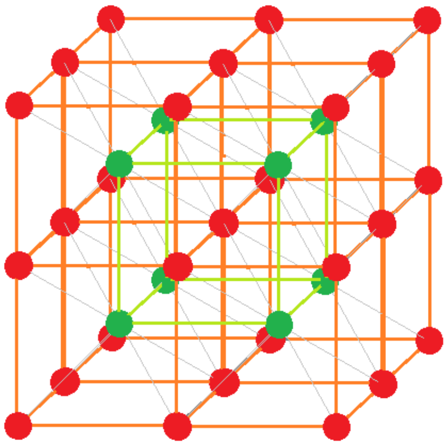
Helmholtz-Zentrum Geesthacht
Centre for Materials and Coastal Research



Disordered Beta TiAl
Ti and Al have random distribution at atom positions



Ordered Beta TiAl
Positions of Ti and Al are determined



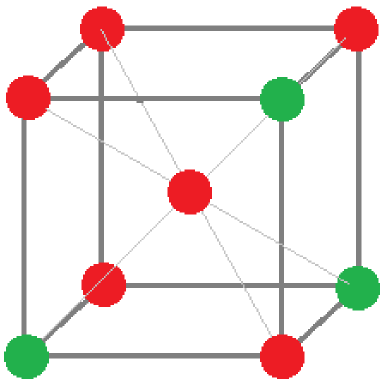
Every kind of atom has Simple Cubic Structure

Influence of the crystall symmetry to a diffracton pattern :

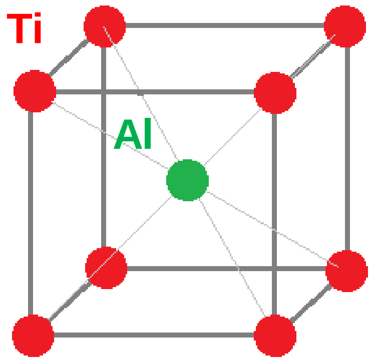
Forbiddened reflections of the BCC structure: **h+k+l odd** : **100; 111**; … are allowed in Simple Cubic lattice and called **Superstructural**

Reflections, allowed for both crystal structures called **Fundamental: 200; 101…**

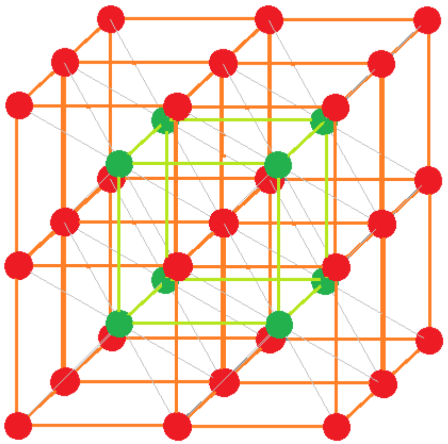
Ordering and disordering and a Structure factor



Disordered Beta TiAl
Ti and Al have random distribution at atom positions



Ordered Beta TiAl
Positions of Ti and Al are determined



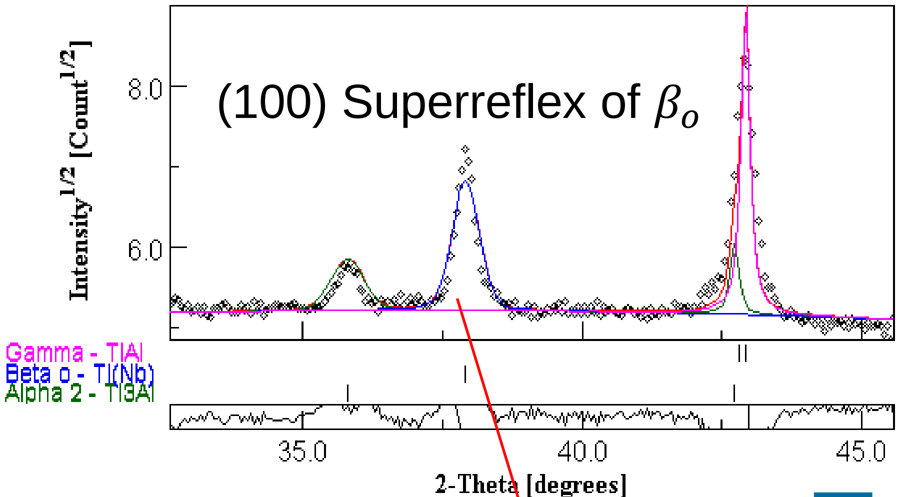
Every kind of atom has Simple Cubic Structure

<u>Influence of the crystall symmetry to a diffracton pattern :</u>

Forbiddened reflections of the BCC structure: h+k+l odd : 100; 111; … are allowed in Simple Cubic lattice and called Superstructural

Reflections, allowed for both crystal structures called Fundamental: 200; 101…

Neutron applications: Order–disorder transformations



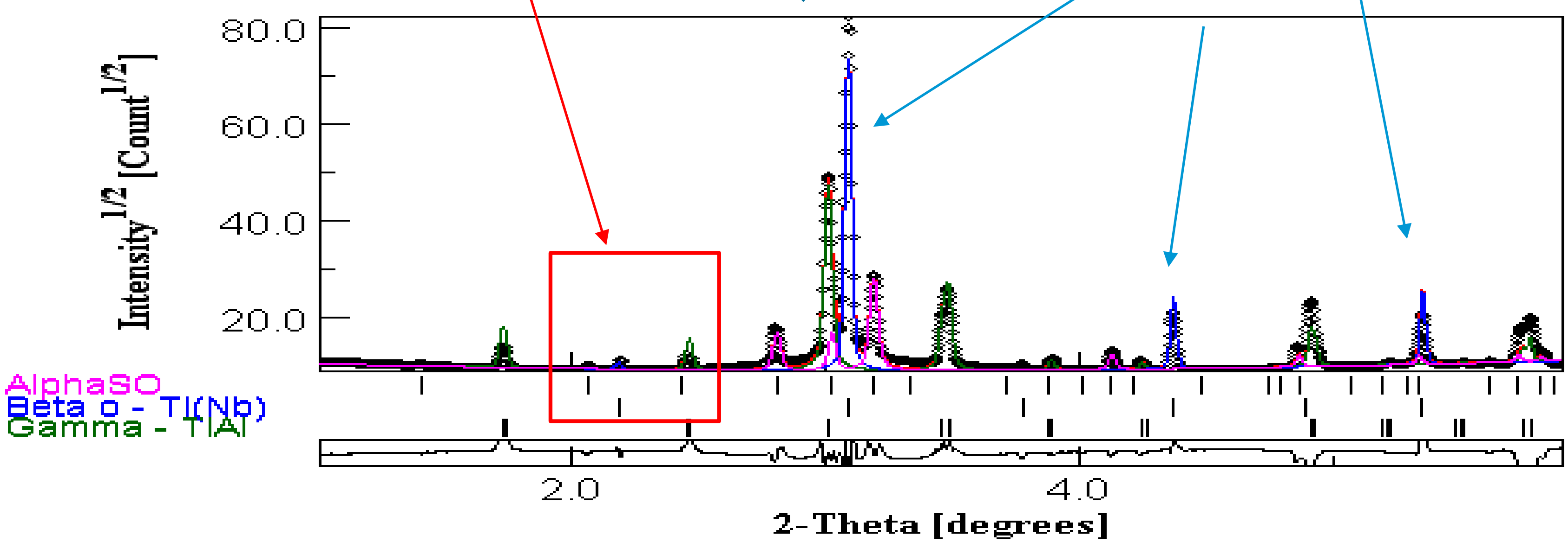
For TiAl: Best contrast of β/β_0

•$b_{Ti} = -3,370\ fm$; $b_{Al} = 3,449\ fm$

Ti-42Al-2Mo 1100°C Neutron spectrum

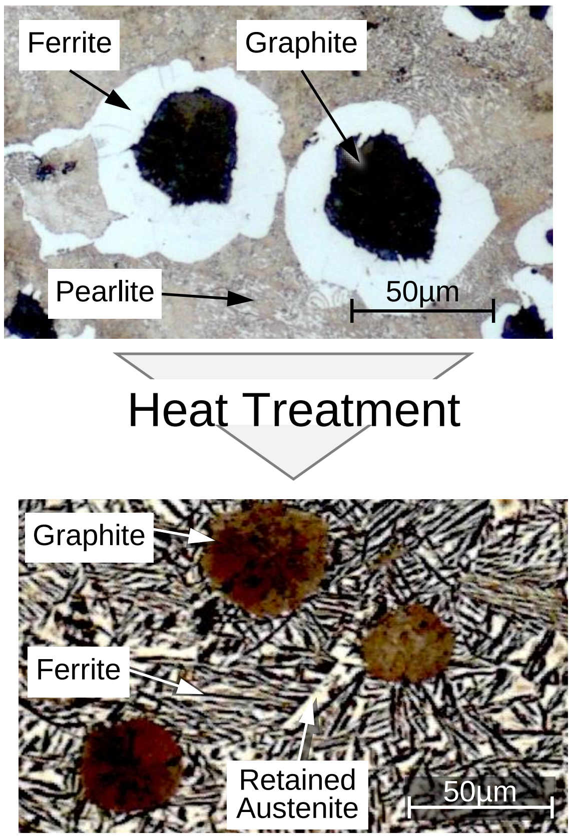
Invisible with neutrons fundamental reflexes of β_o

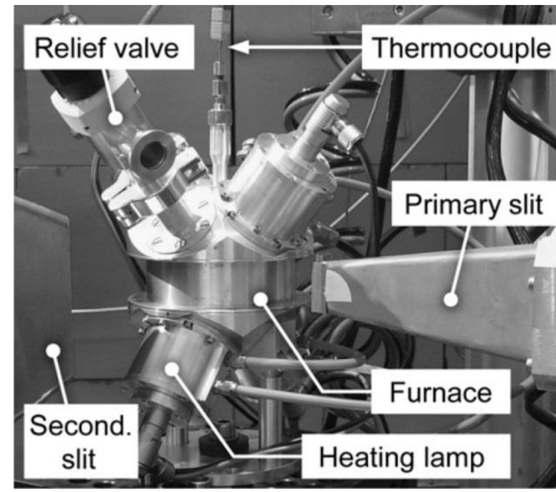
Ti-42Al-2Mo 1100°C Neutron spectrum



Michael Hofmann, MLZ/TUM

Material used in automotive and other industries, low manufacturing costs





Diffraction yields:

- phase fractions during tempering
- carbon content (aust. latt. param).
- morphology via peak width

L. Meier, M. Hofmann, P. Saal, W. Volk, H. Hoffmann, Materials Characterization 85 (2013) 124–133.

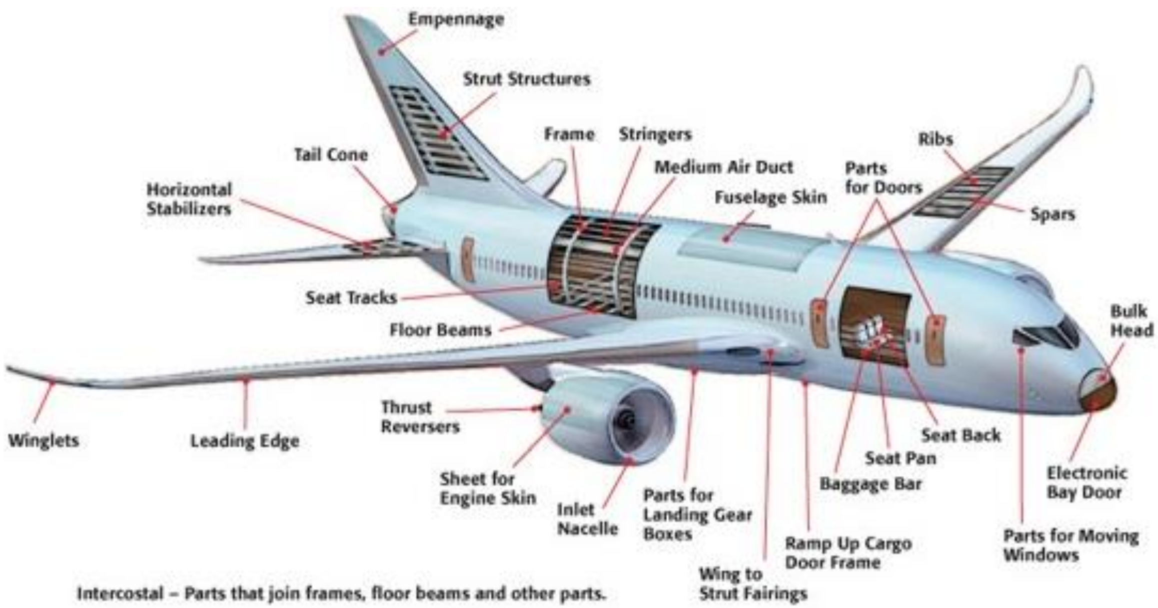
Initial slide produced by P. Staron

Summary:

Helmholtz-Zentrum Geesthacht
Centre for Materials and Coastal Research

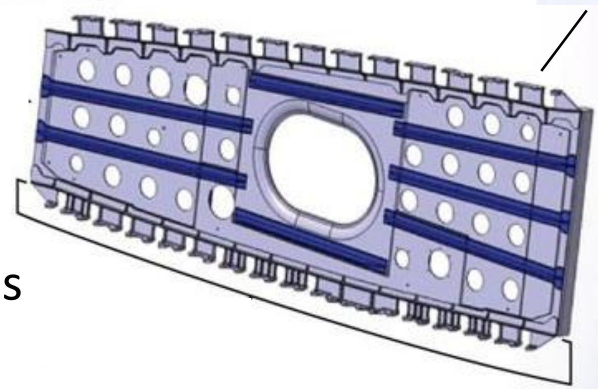
- Dilatometer is unique equipment allows to perform in-situ diffraction measurements under heat treatment and deformation conditions up to 1500°C
- Dilatometer combined with neutron instrument allows us determine microstructure and phase composition of the samples
- Currently there is the only dilatometer installed at a neutron facility
- Use of dilatometer is of high importance for improvement industrial materials processing technologies
- Dilatometer saves experimental time by faster heating and cooling. Precise processing temperature control is possible
- Dilatometer signal can detect sample melting

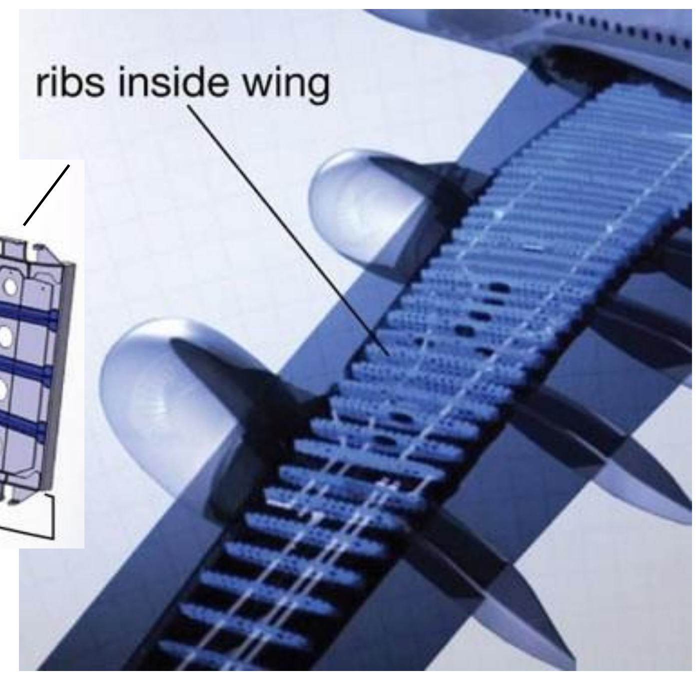
Welding temperature cycles



Various Al alloys with specific properties used for different parts of an aircraft

- strength
- fatigue resistance
- corrosion resistance
- …





Al alloy AA7449:

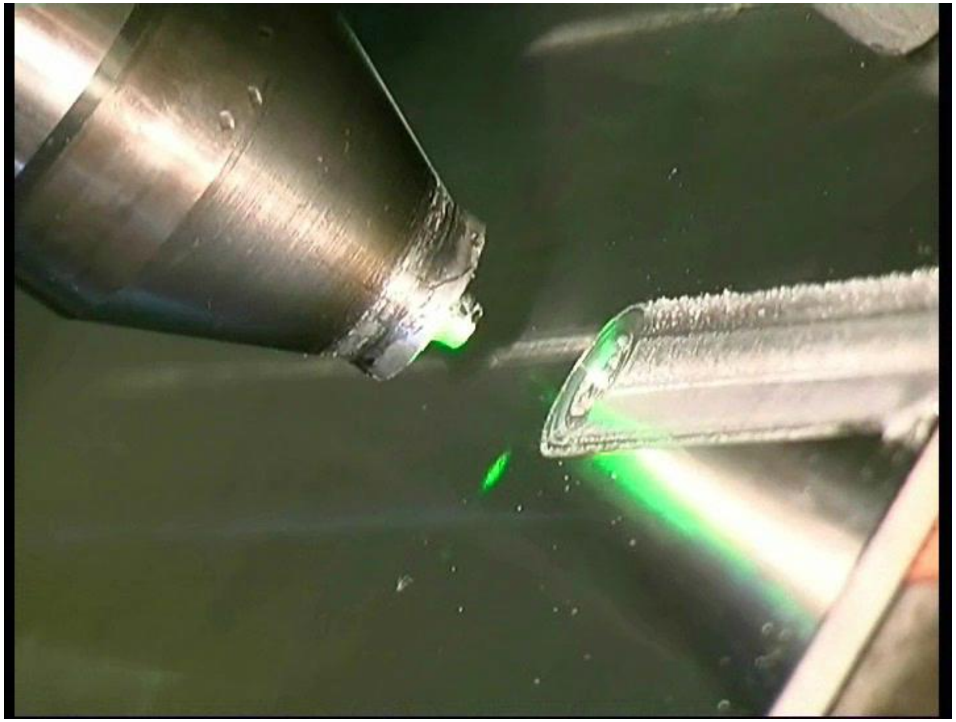
- developed for Airbus A340-500/600 wing panels
- very high strength (550 Mpa yield strength)
- used for A380 wing ribs

Source: Airbus

Initial slide produced by P. Staron

Helmholtz-Zentrum Geesthacht
Centre for Materials and Coastal Research

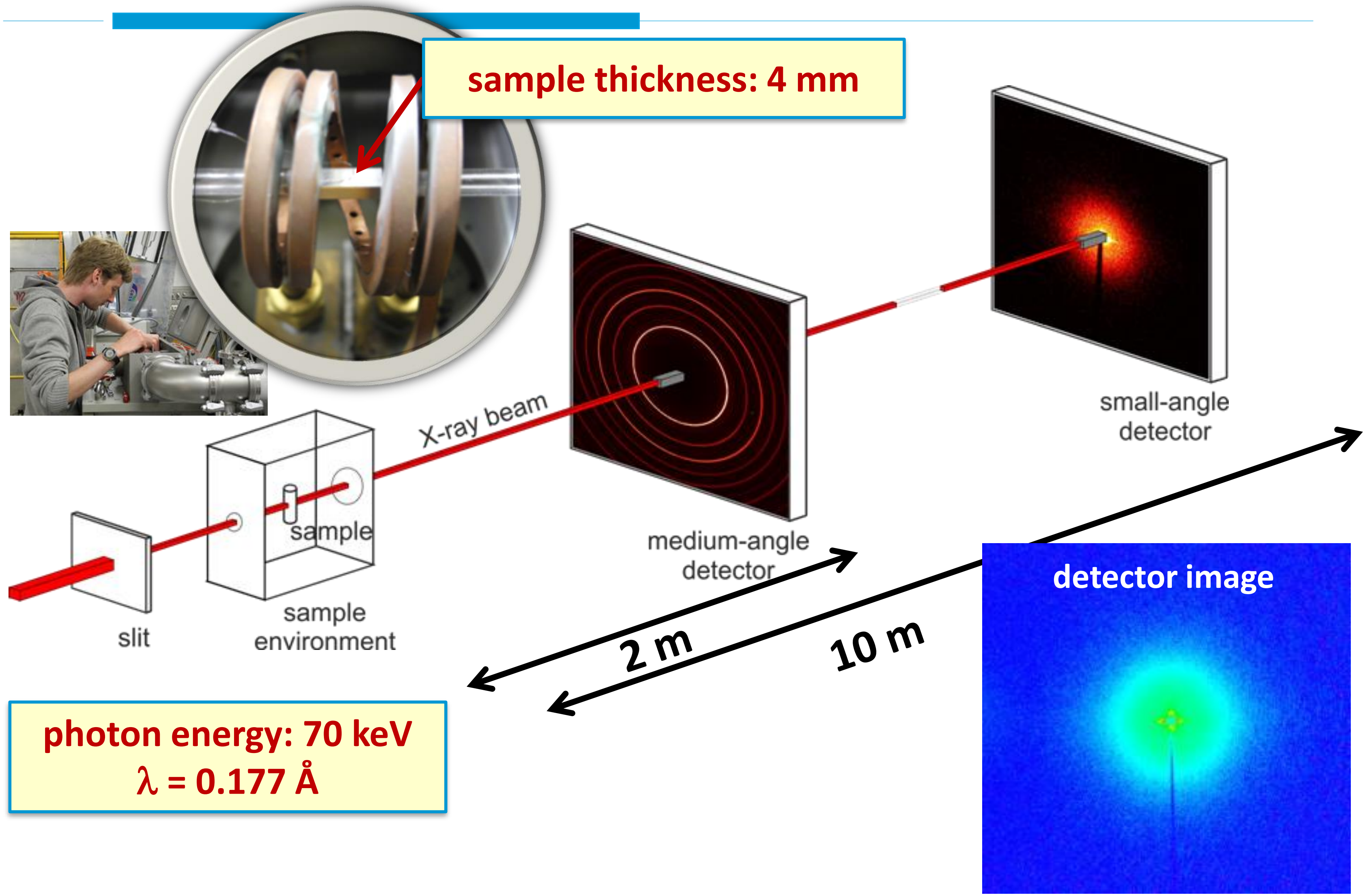
Friction stir welding of Al-Mg-Zn-Cu aircraft alloys



Development of precipitates during FSW?

Initial slide produced by P. Staron

High-energy SAXS experiment at HEMS (P07)



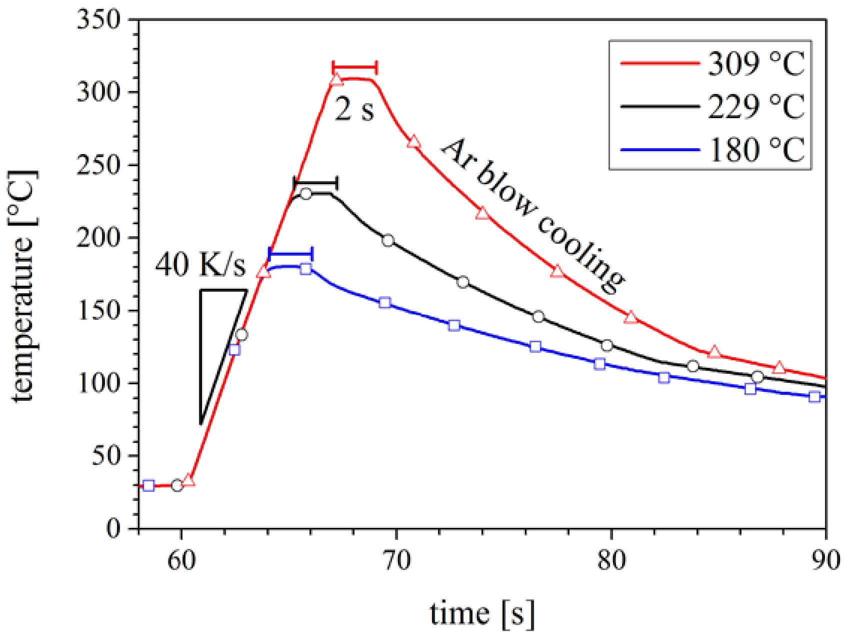
sample thickness: 4 mm

photon energy: 70 keV
λ = 0.177 Å

Initial slide produced by P. Staron

Helmholtz-Zentrum Geesthacht
Centre for Materials and Coastal Research

Dilatometer:
apply temperature cycles as they occur in friction stir welding



- **temperature profile corresponds to a feed rate of 5 mm/s**
- **T_{max} ←→ distance from weld line**

PhD work of Torben Fischer, HZG

Torben Fischer, PhD Thesis, University of Hamburg (2014)

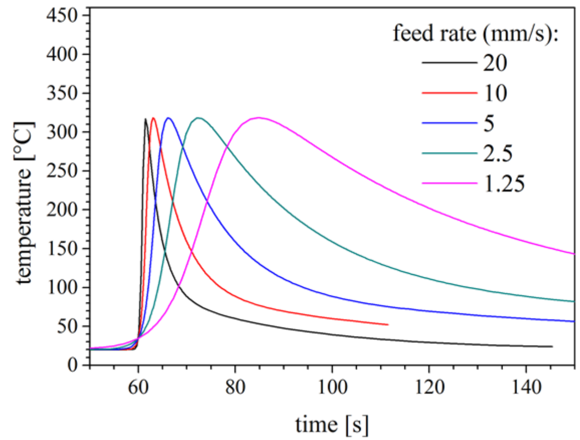
Initial slide produced by P. Staron

Numerical modelling

Helmholtz-Zentrum Geesthacht
Centre for Materials and Coastal Research

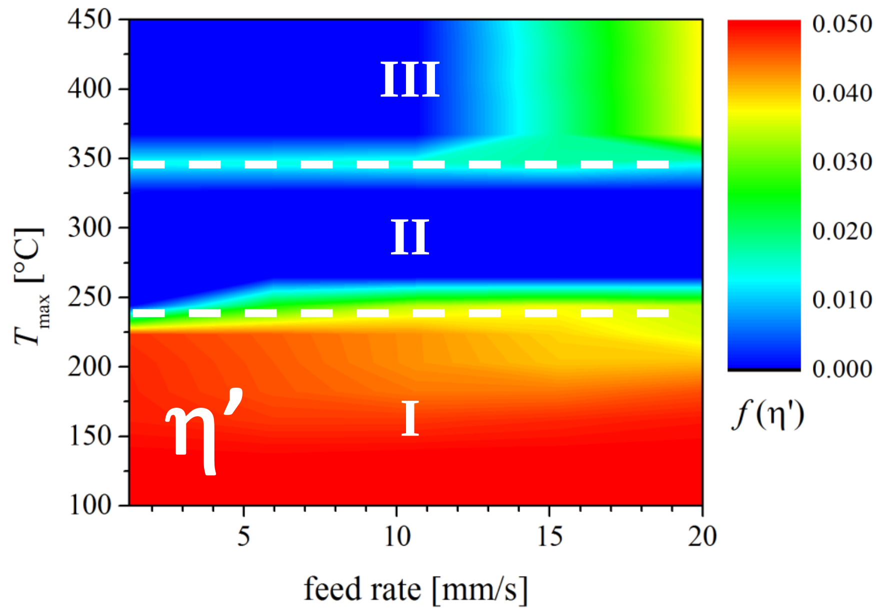
Model predictions on AA7449-TAF:

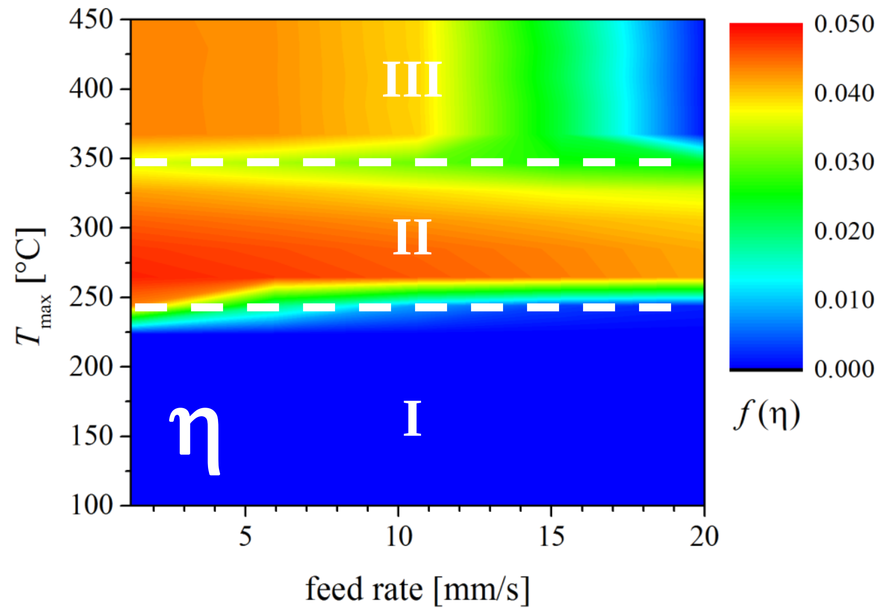
Feed rate variation: 1.25 … 20 mm/s



volume fraction *f*

slow → fast





→ relation between welding speed and diffusion speed plays a crucial role!

Initial slide produced by P. Staron