Dilatometer for neutron diffraction in-situ investigations

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Motivation: in-situ industrial heat-treatment

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 environments 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

- 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: $\Delta l = 0.05 \mu m$, $\Delta T = 0.05^\circ C$, $\Delta t = 0.0005 s$

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

Alloy systems studied since 2009:

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

- low density, ~4 g/cm³ (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

- the blade is a forged product
- establishment of “forging window” for new alloys required
Continuous Cooling Transformation (CCT)

**fast and controlled cooling**

![Diagram showing CCT](image)

- **Temperature vs. Time**
  - 1200 °C
  - 200 K/s, 20 K/s, 2 K/s

- **Phase Transformations**
  - (a) dissolution temperature of γ phase
  - (b) α→α₂
  - (c) α→γ_massive
  - (d) α→α₂+γ

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

- **Microstructures**
  - γ absent
  - γ massive
  - γ lamellar

- **Predictions**
  - Phase content
  - Microstructure
  - Mechanical properties
Modeling of the Time Temperature Transformation (TTT) diagrams for TiAl

Temperature

Temperature

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

(a)

Scattering vector |q| / 2\pi \cdot \text{Å}^{-1}

900

1000

1100

1200

1300

\alpha \rightarrow \alpha_2

\gamma \rightarrow 0

\gamma_1 \rightarrow 0

\gamma_0 \rightarrow 0

\gamma_1 \rightarrow 0

\gamma_0 \rightarrow 0

\alpha \rightarrow \alpha_2

dissolution temperature of \gamma phase

dissolution temperature of \alpha phase

Time
In-situ high-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

Intensities 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

Slow \((1 \cdot 10^{-2} \text{ s}^{-1})\) deformation @ 1230 °C of Ti-43Al-4Nb-1Mo-0.1B (in at.%)
**Tensile test**

*Sheet steel / 900°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

*Al-alloys / 400°C*
Possible experiments with dilatometer at high temperatures:

- Quenching
- Compression
- Tension
The dilatometer is going to be combined with two neutron instruments: STRESS-SPEC and SANS-1.

Neutrons vs Synchrotron = Different contrast
Neutron Dilatometer at FRM II

Difference between synchrotron and neutron dilatometers:

- Windows shape and material
- Coil shape
- Blowing gas mechanism
- Hight of the main frame with measurement units

Foto from Weimin Gan
Dilatometer at STRESS-SPEC

Take off angle: 30° - 110°

1 Å – 2.4 Å
Neutron applications: Order–disorder transformations in TiAl

Neutron applications: TiAl alloys

... only neutrons allow the study of order–disorder transformations in TiAl alloys, e.g. of the $\beta/\beta_0$-phases. $b_{Ti} = -3,370 \, fm; b_{Al} = 3,449 \, fm$

Neutron applications: Order–disorder transformations

Temperature range: 1100 – 1450 °C

Heating ramp: 20 K/min

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

Detector

B e a m

Sample

Temperature, °C

5000 5100 5200 5300 5400

File N

Temp., °C

Elong, mkm

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

Vacuum high temperature furnace

STRESS-SPEC@MLZ
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

Dilatometer at SANS-1

- Precipitates and segregation in alloys
- Defects in materials

3.5 Å – 30 Å
Study of Sorption in monoliths with hierarchical pore structure


→ Potential for simultaneous in-situ SANS and dilatometry
Summary:

- 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.
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Thank you for your attention!
Dilatometer – an equipment for reproduction heat treatment conditions

Quenching- and deformation measuring head

Vacuum units: turbo molecular pump and pre-pump

Hydraulic aggregate

Control main frame

Measuring main frame

TA Instruments, Instruction for first use
In-situ phase analysis for TTT or CCT diagramms

Continuous Cooling Transformation (CCT)  Time Temperature Transformation (TTT)

(a) dissolution temperature of γ phase

Temperature

Time 200 K/s  20 K/s  2 K/s

α→α₂  α→γmassive  α→α₂+γ

dissolution temperature of γ phase

Temperature

Time

Neutron or synchrotron beam

Heated Sample

Helmholtz-Zentrum Geesthacht
Centre for Materials and Coastal Research
Neutron applications: Order–disorder transformations


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

β-phase important for forging.

→ is there ordered β-phase at high temperatures?

PhD work of Viktoria Kononikhina, HZG
Ordering and disordereding 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

Influence of the crystalline symmetry to a diffracton pattern:

Forbidden 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…
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Neutron applications: Order–disorder transformations

(100) Superreflex of $\beta_0$

For TiAl: Best contrast of $\beta/\beta_0$

- $b_{Ti} = -3.370 \text{ fm}$; $b_{Al} = 3.449 \text{ fm}$

Ti-42Al-2Mo 1100°C Neutron spectrum

Invisible with neutrons fundamental reflexes of $\beta_0$
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

Summary:

- 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
Welding temperature cycles

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

Initial slide produced by P. Staron
High-energy SAXS experiment at HEMS (P07)

- photon energy: 70 keV
  \[ \lambda = 0.177 \text{ Å} \]
- sample thickness: 4 mm
Welding temperature cycles

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

- temperature profile corresponds to a feed rate of 5 mm/s
- $T_{\text{max}} \leftrightarrow$ distance from weld line

Numerical modelling

Model predictions on AA7449-TAF:
Feed rate variation: 1.25 ... 20 mm/s

volume fraction $f$

$\rightarrow$ relation between welding speed and diffusion speed plays a crucial role!