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# Evaluating deformation behavior of a TBC-System during thermal gradient mechanical fatigue by means of high energy X-ray diffraction

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**Authors**

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# Evaluating deformation behavior of a TBC-system during thermal gradient mechanical fatigue by means of high energy X-ray diffraction



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Knowledge for Tomorrow



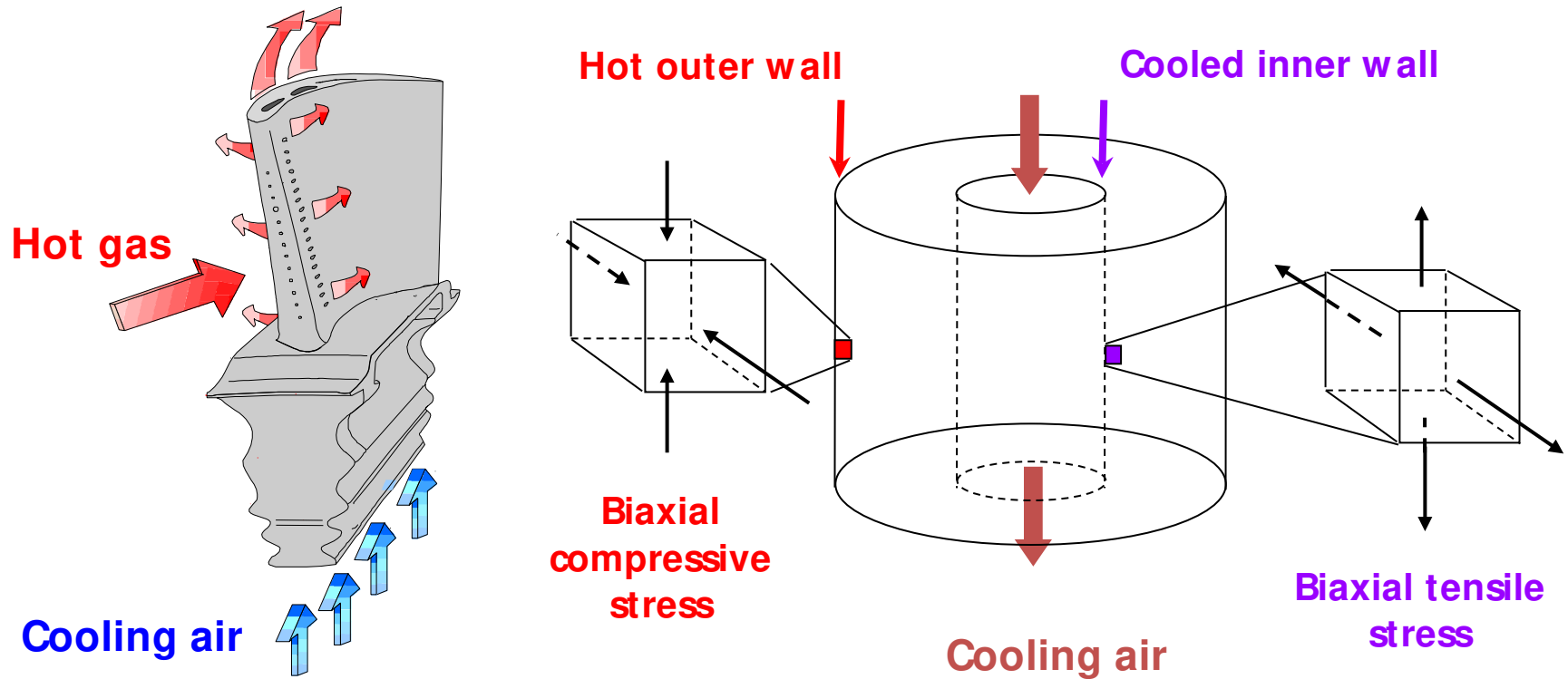
# Outline

1. Realistic thermomechanical testing with thermal gradients
2. Interpreting experimental results by means of numerical models
3. Model validation by means of in situ strain measurements via high energy X-ray diffraction at Argonne APS\*

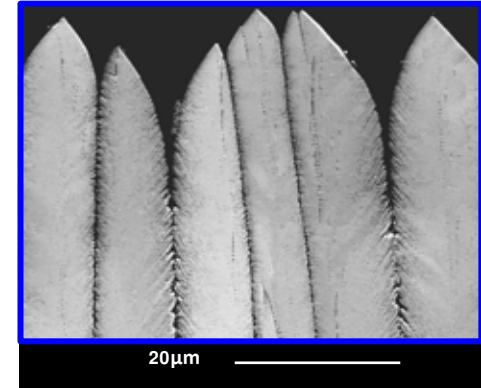
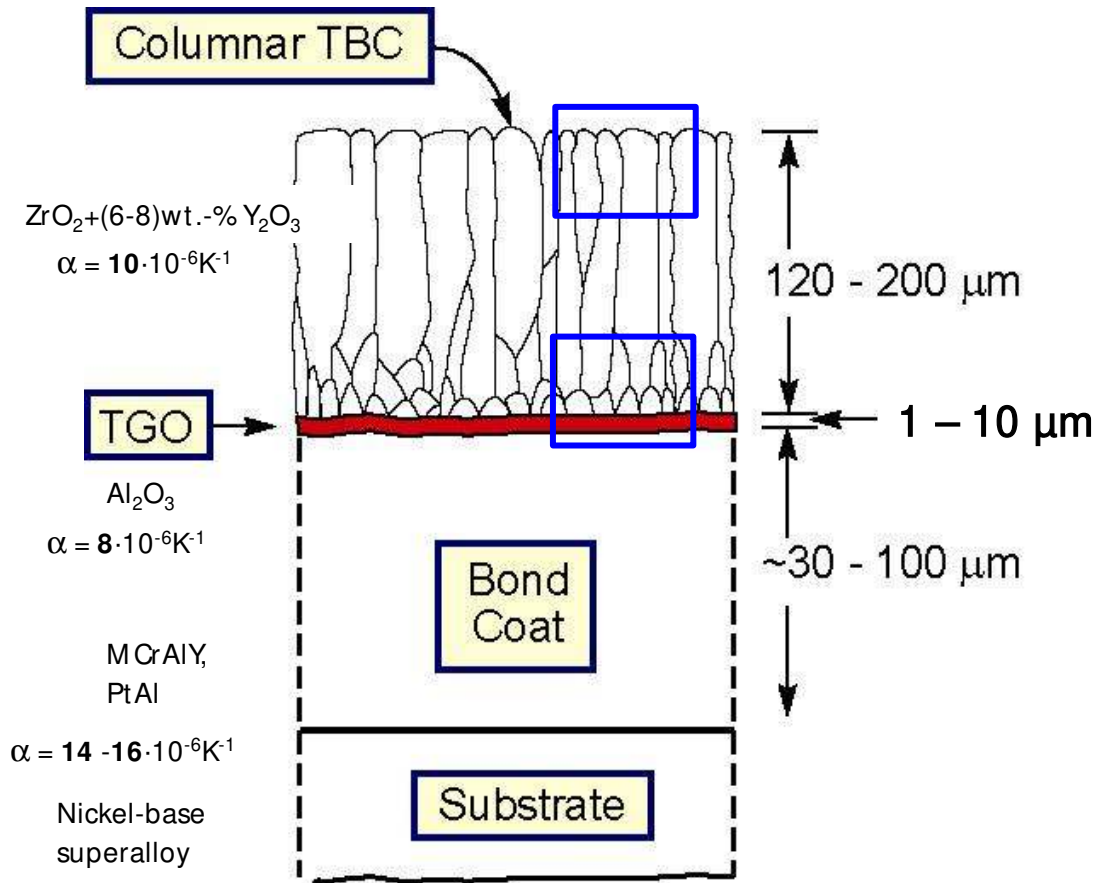
\* APS=Advanced Photon Source



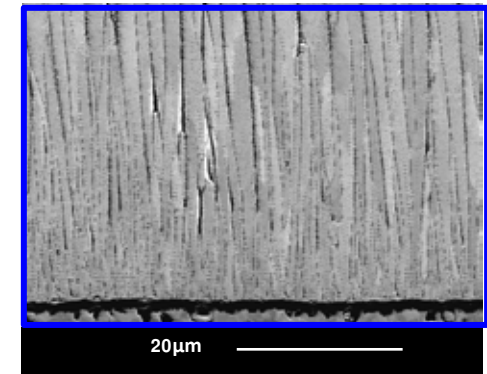
# Stress distribution due to thermal gradient



# Investigated coating system



near surface

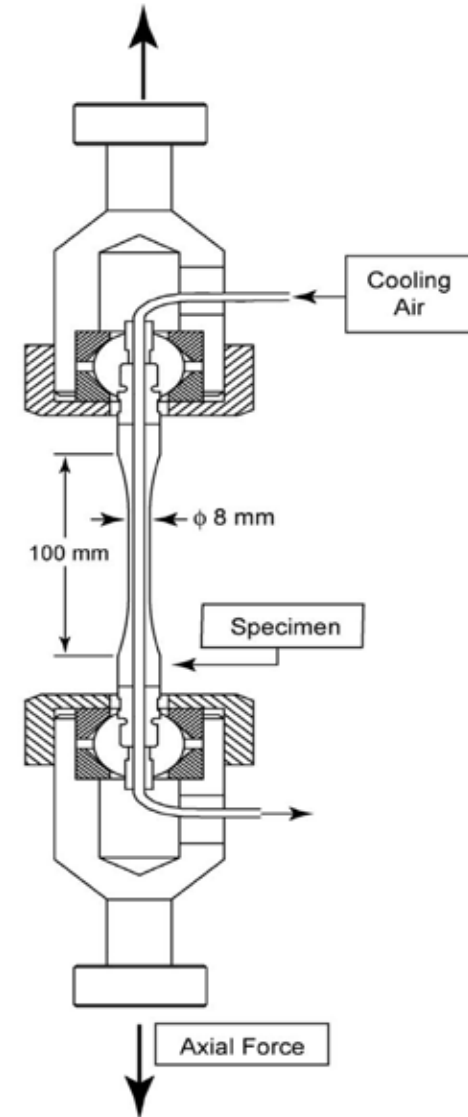
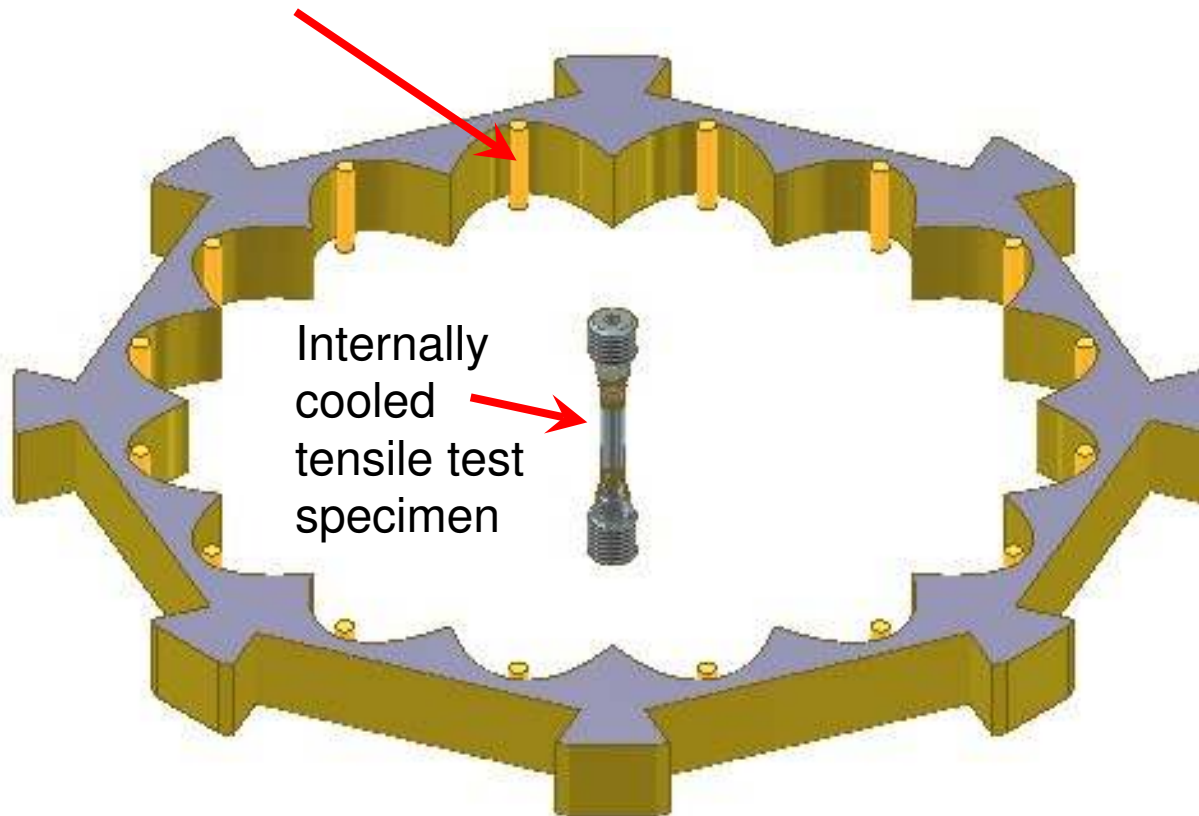


near TGO



# Test facility for thermal gradient mechanical fatigue

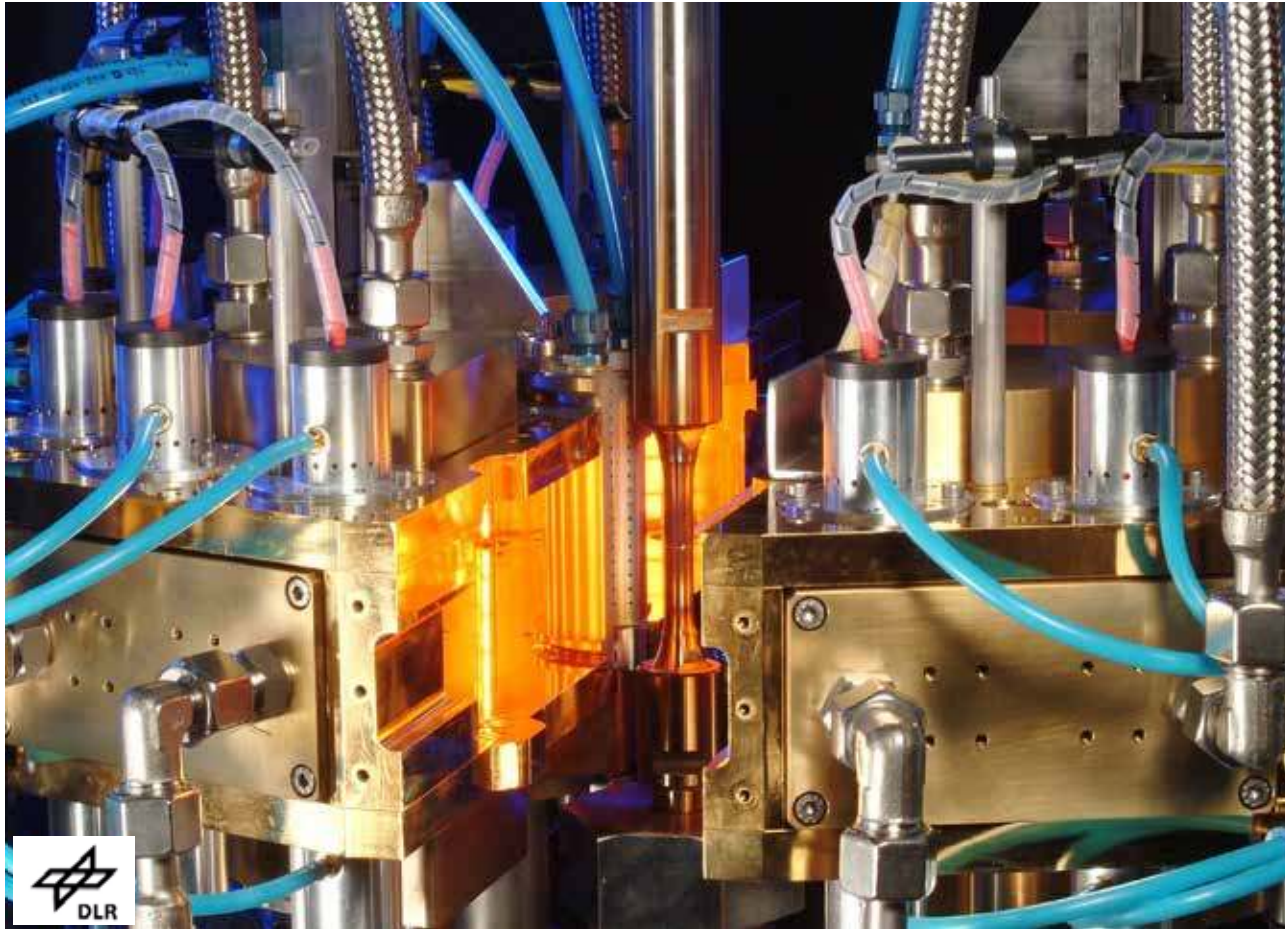
16 Quartz lamps, 1 kW each



Thermal Gradient Mechanical Fatigue = TGMF



# View of open furnace



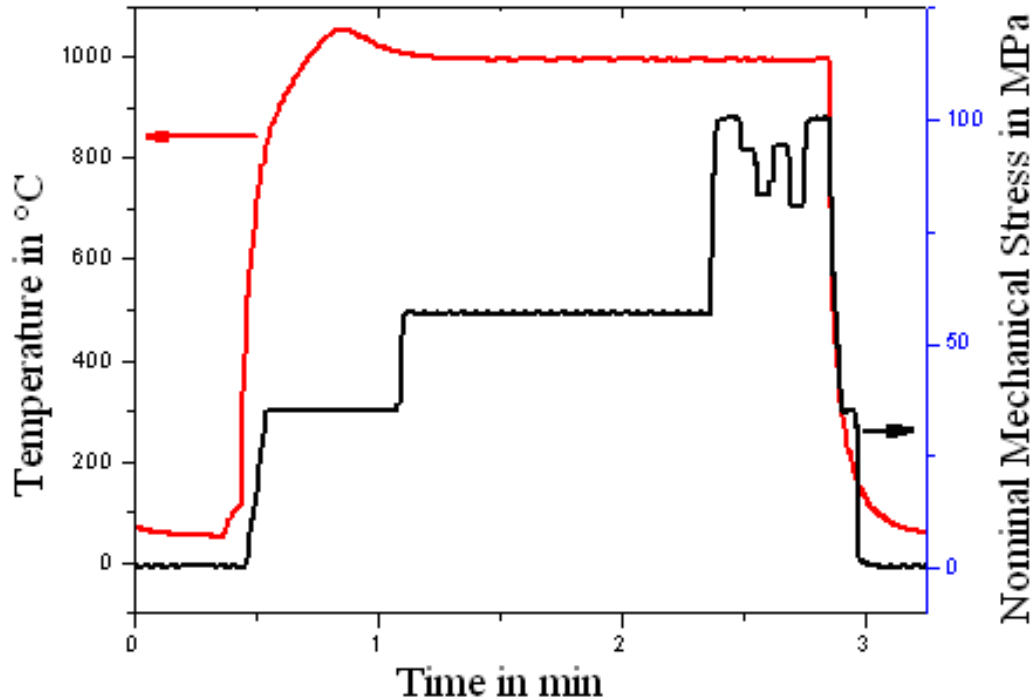


# Summarizing thermal and mechanical loads

- Maximal material temperatures ca. 1000°-1100°C
- Thermal gradient (temperature drop over a ceramic TBC of 100-200µm thickness of about 80°-150°C)
  - High thermal heat flux
  - Multiaxial thermally induced stresses
- High thermal transients (heating and cooling rates)
- Superposed mechanical loads (centrifugal forces on rotating blades)



# Thermal mechanical load cycle – representing the fatigue load of flight cycle



- It is not practical to perform test cycles with realistic cycle duration (e.g. 2 - 10 hour flights) - thus: reduced dwell times
- But: time at high temperature has major impact on lifetime of the coating



# Considering time dependent effects by pre-ageing

Time at 1000°C		TGMF-cycles
0 h	+	500 (25h)
250 h		1000 (50h)
500 h		until spallation

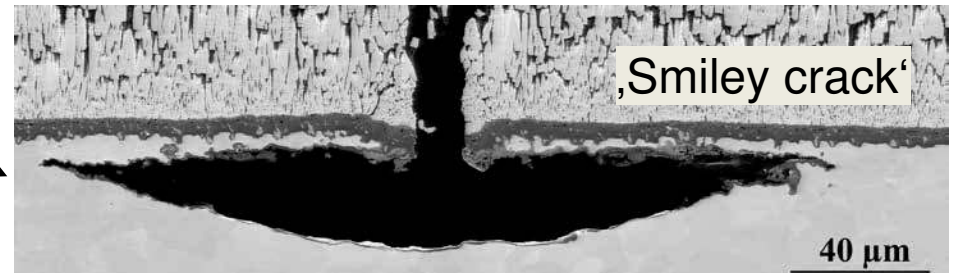
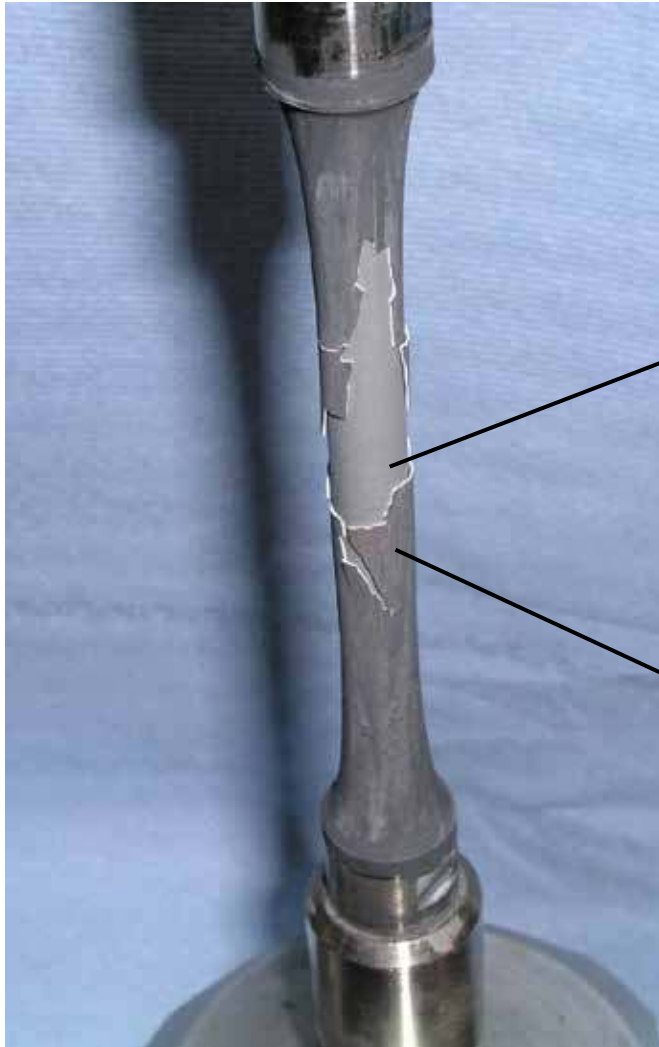
Pre-ageing

+

Thermomechanical fatigue



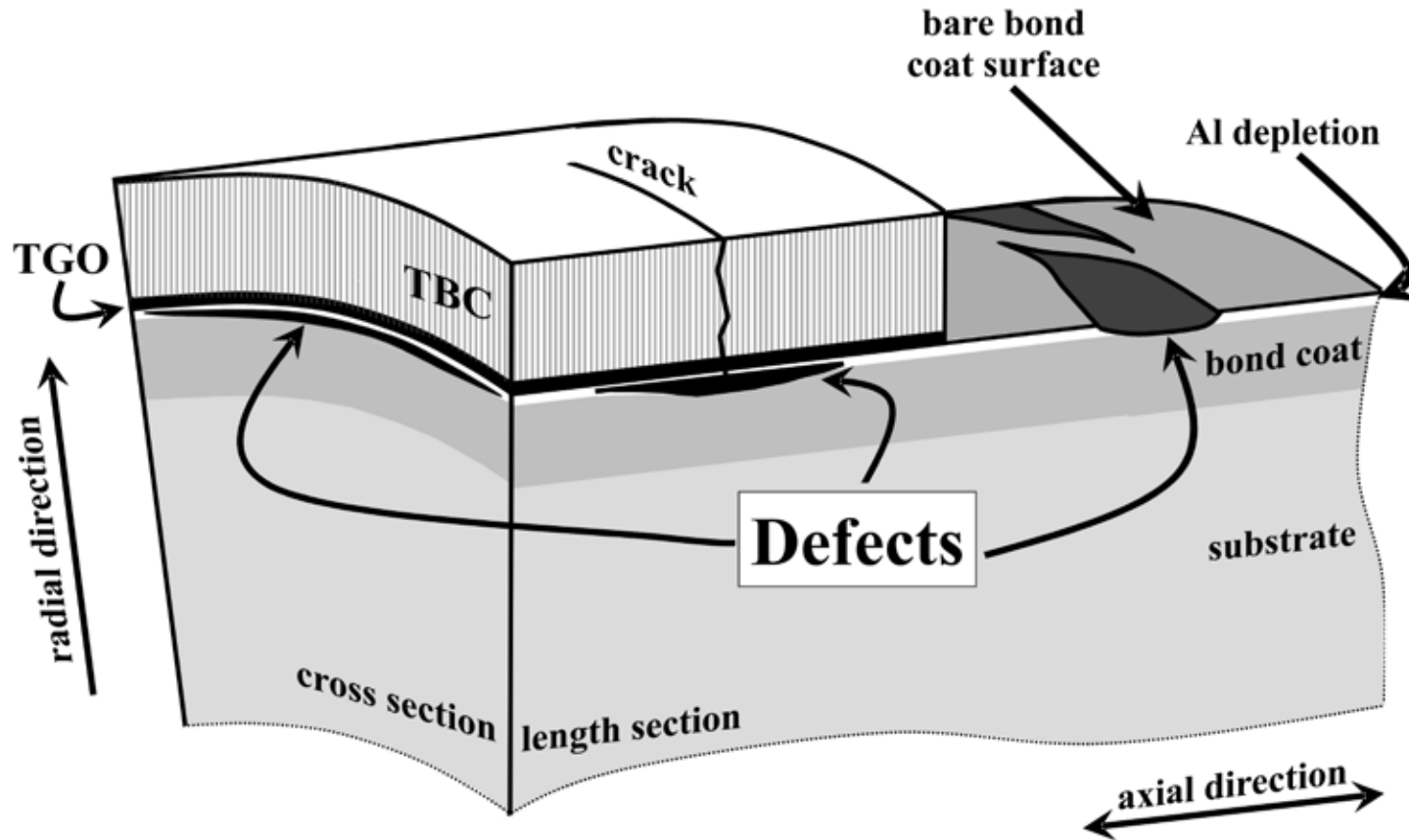
# Failure after thermomechanical laboratory testing



after 933 TGMF-cycles &  
500h pre-ageing at 1000°C



# 3 - dimensional sketch of defects



Sketch by Bernd Baufeld, in Key Eng. Mat. Vol. 333 (2007) pp. 147-154

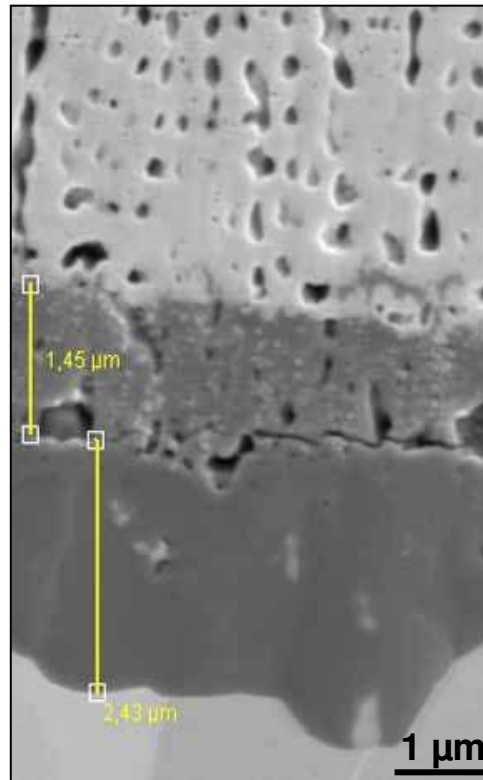


# Summary of experimental results

- **Without pre-ageing no spallation up to 7000 cycles**
  - **250h (500h) pre-ageing + 1000 cycles, open delamination cracks, spallation**
- Evolution of the ‚smiley‘ cracks is linked to cracks in the TGO, perpendicular to the applied mechanical load.
- Initial TGO cracks are generated due to axial tensile stresses
- The questions are
- **How can axial tensile stresses evolve in the TGO during TGMF tests?**
  - **Why do they only evolve in pre-aged specimens?**



# After pre-ageing: bi-layer thermally grown oxide



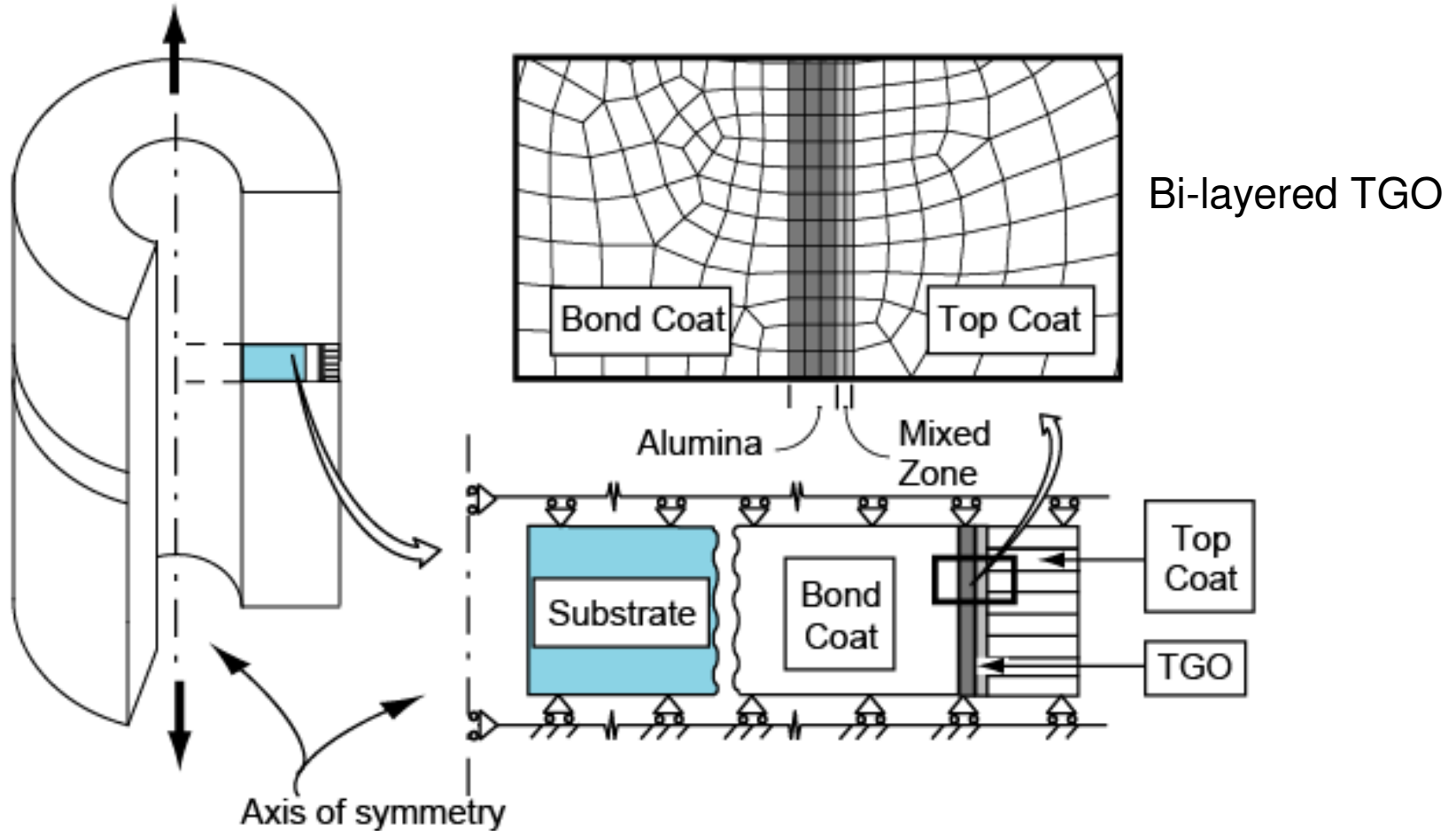
Fine grained  
intermixed zone  
 $\text{Al}_2\text{O}_3 + \text{ZrO}_2$

Coarse grained  
 $\text{Al}_2\text{O}_3$

200h/1000°C



# Numerical model: Geometry and boundary conditions

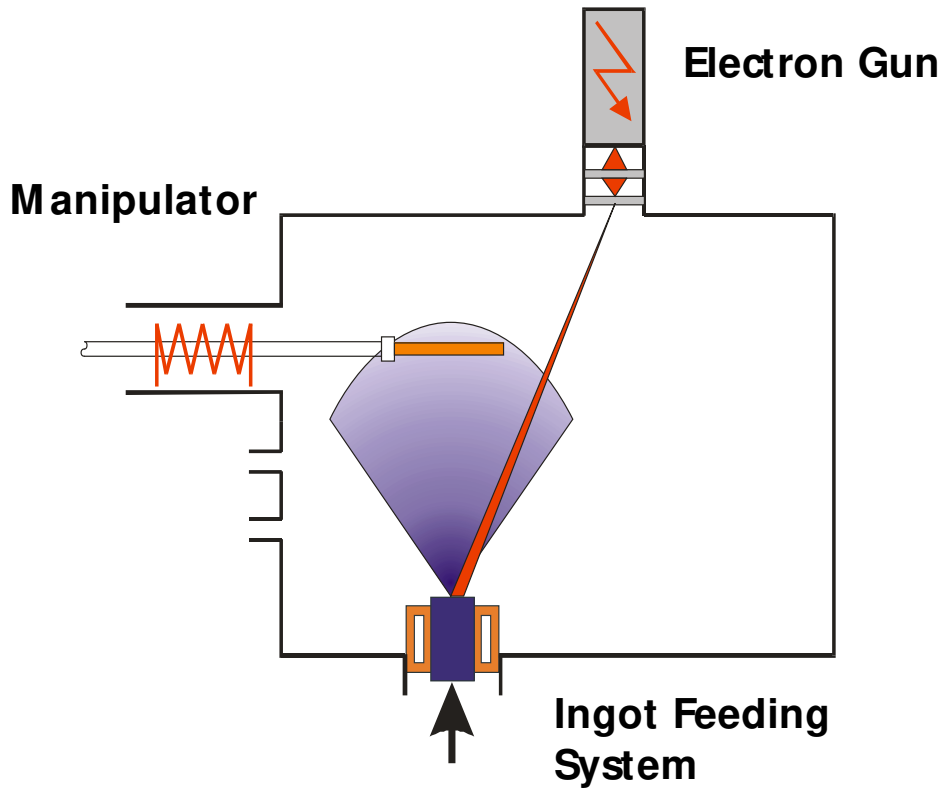


*M. Hernandez, A.M. Karlsson, M. Bartsch: Surface Coatings & Technology 203, 3549-58, 2009*





# Stress free at homogenous temperature of 1000°C



Deposition temperature:  
ca. 1000°C

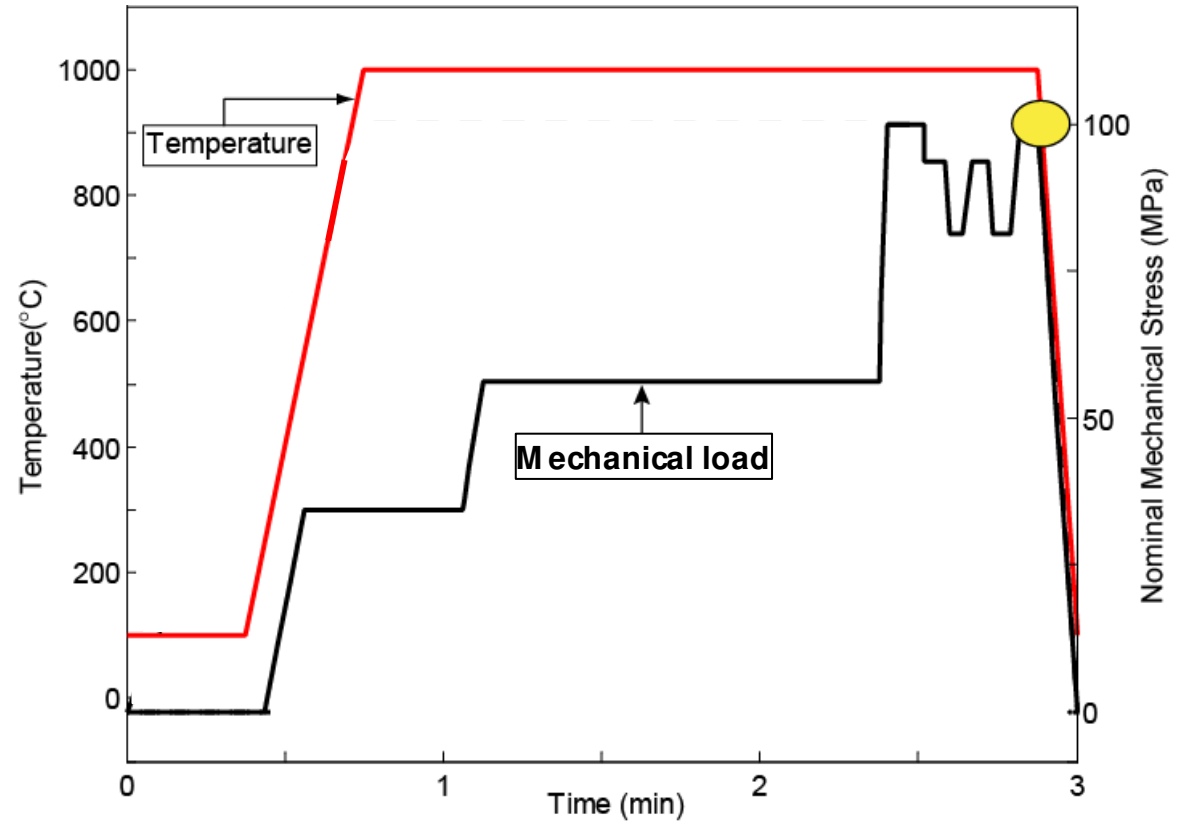
Electron Beam - Physical Vapor Deposition  
(EB-PVD)

⇒ **high residual stresses  
at ambient temperature**



# Numerical model: load cycle

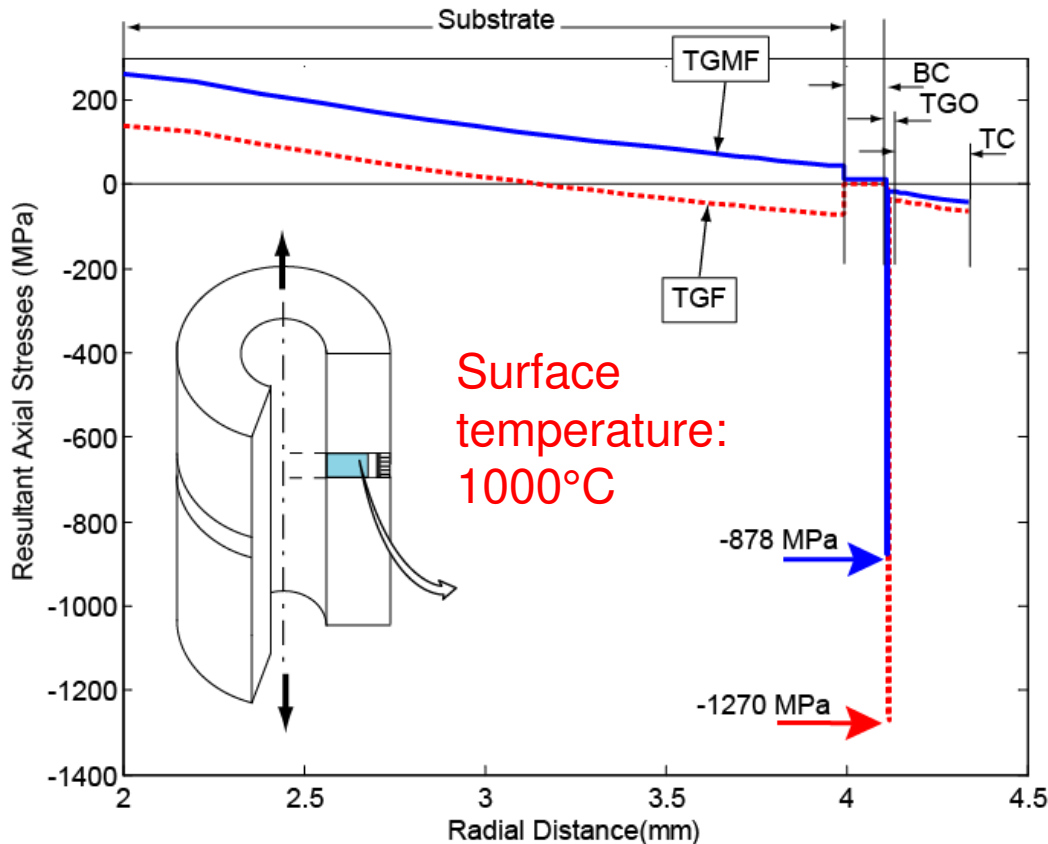
- Temperature at the outer surface is shown
- Thermal gradient: time dependent temperature difference between outer and inner wall (not shown)
- mechanical cycle TGMF



● Highest mechanical tensile load, thermal gradient near stationary conditions



# Axial stresses for elastic – plastic material properties



Stress free at  $T_{\text{processing}}$   
(1000°C, homogenous)

Axial stresses across the specimen wall due to

- thermal gradient
- mechanical load
- property mismatch

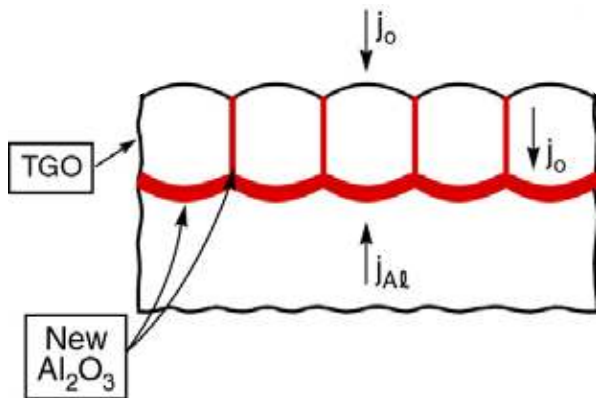
→ TGO always under compression

even at highest mechanical tensile load

M. Hernandez, A.M. Karlsson, M. Bartsch: *Surface Coatings & Technology* 203, 3549-58, 2009



# Including time dependent TGO properties: growth strain and creep / relaxation

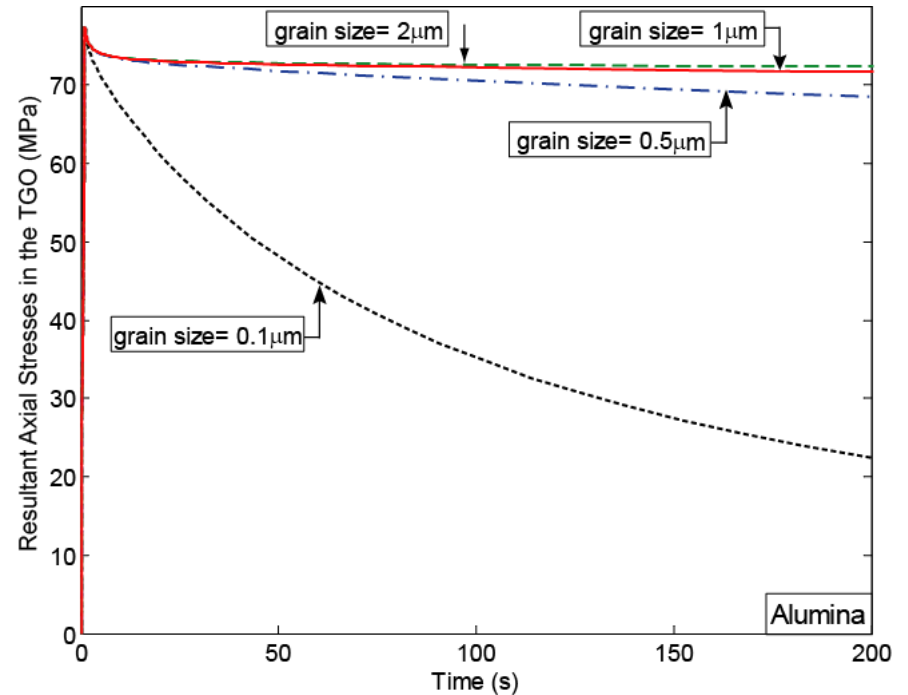


Thickening  $\epsilon_t$  and lengthening  $\epsilon_l$  growth strain

$$\epsilon_l = 0.1 \cdot \epsilon_t$$

**Growth strain increases the compressive stress in TGO!**

Karlsson, A.M. and A.G. Evans, *Acta Materialia*, 2001 **49**(10): p. 1793-1804

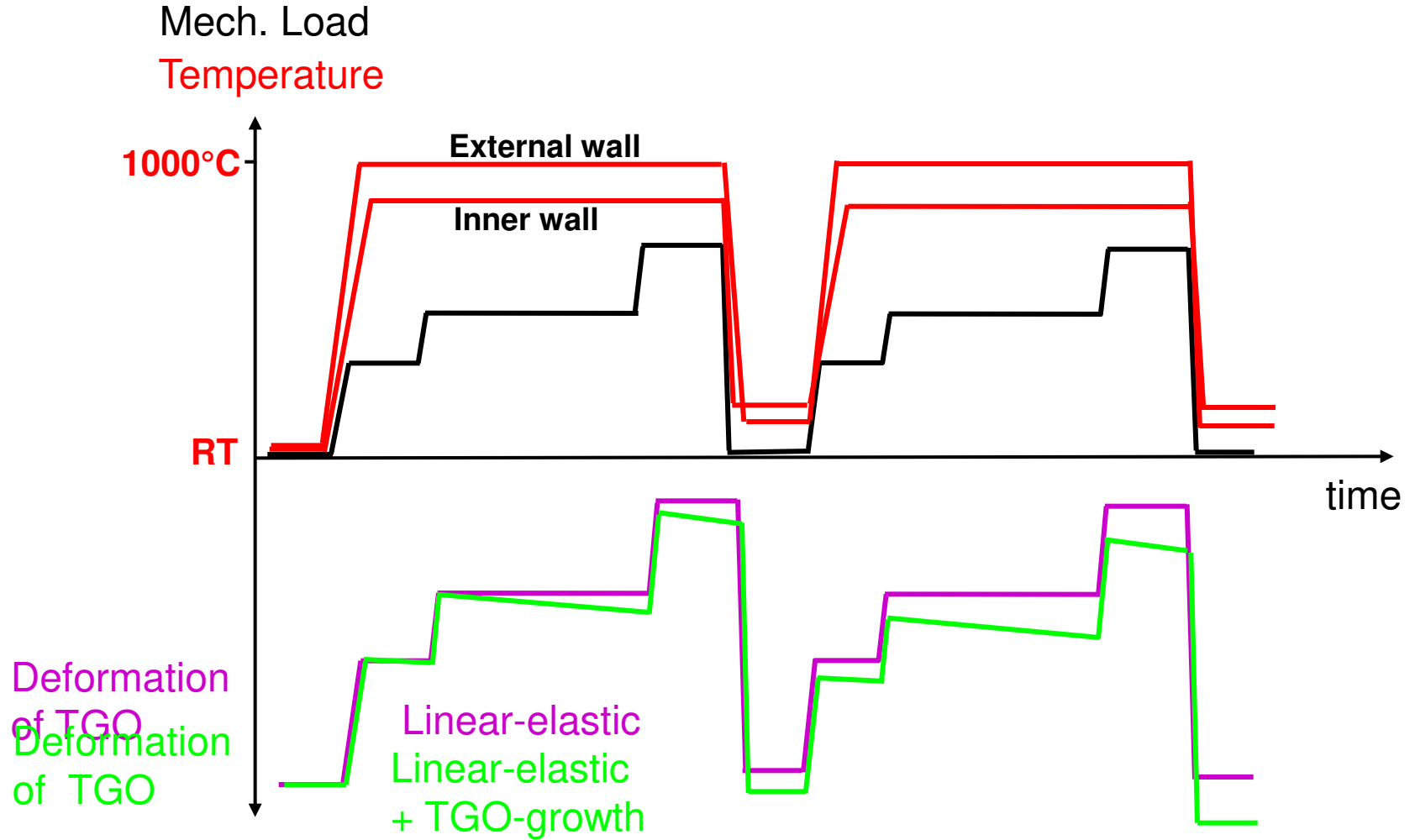


**Relaxation decreases the compressive stress in TGO!**

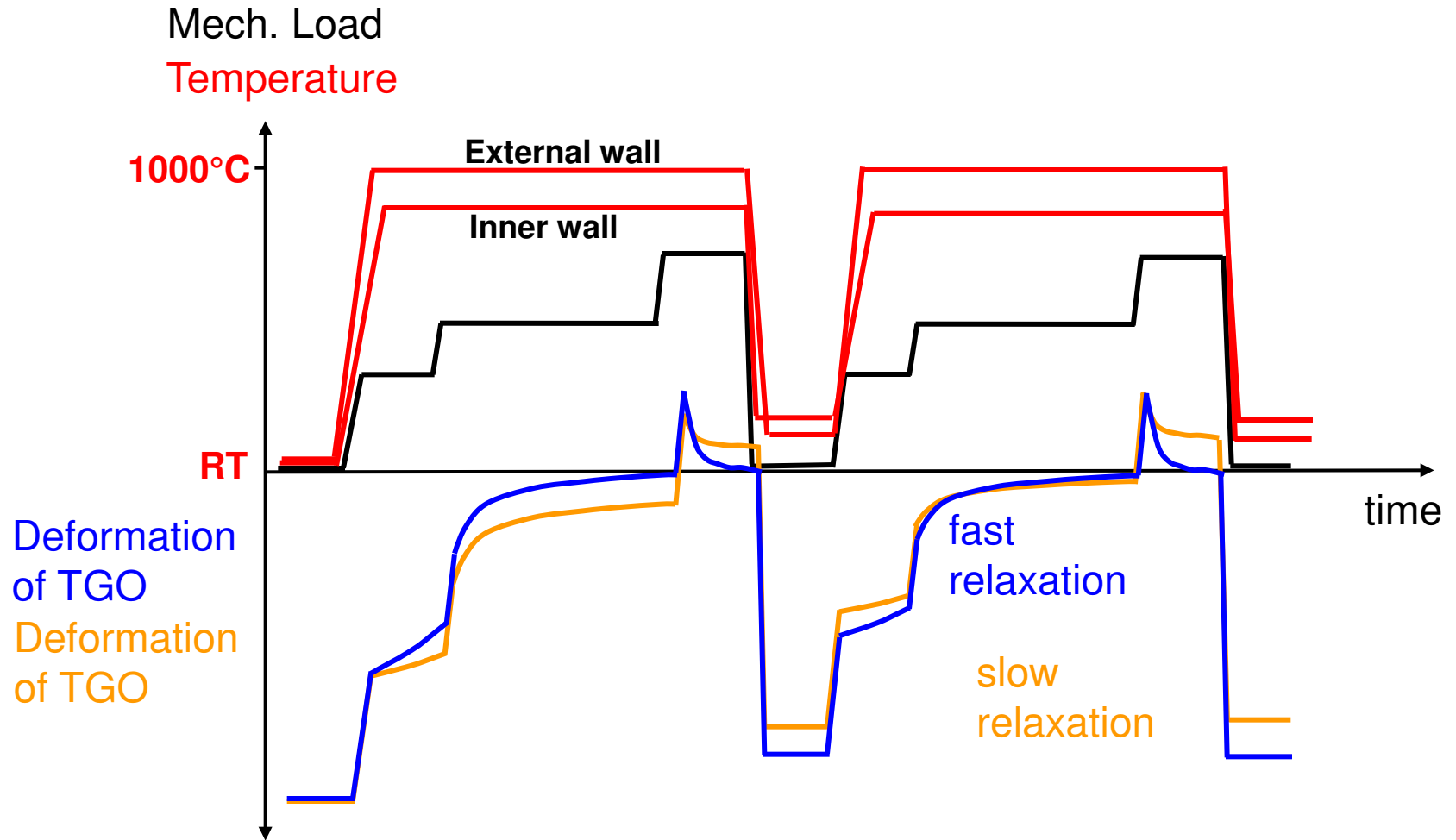
With data from J.D. French, J.H. Zhao, M.P. Harmer, H.M. Chan, G.A. Miller. *J. American Ceramic Society* 77 (1994)



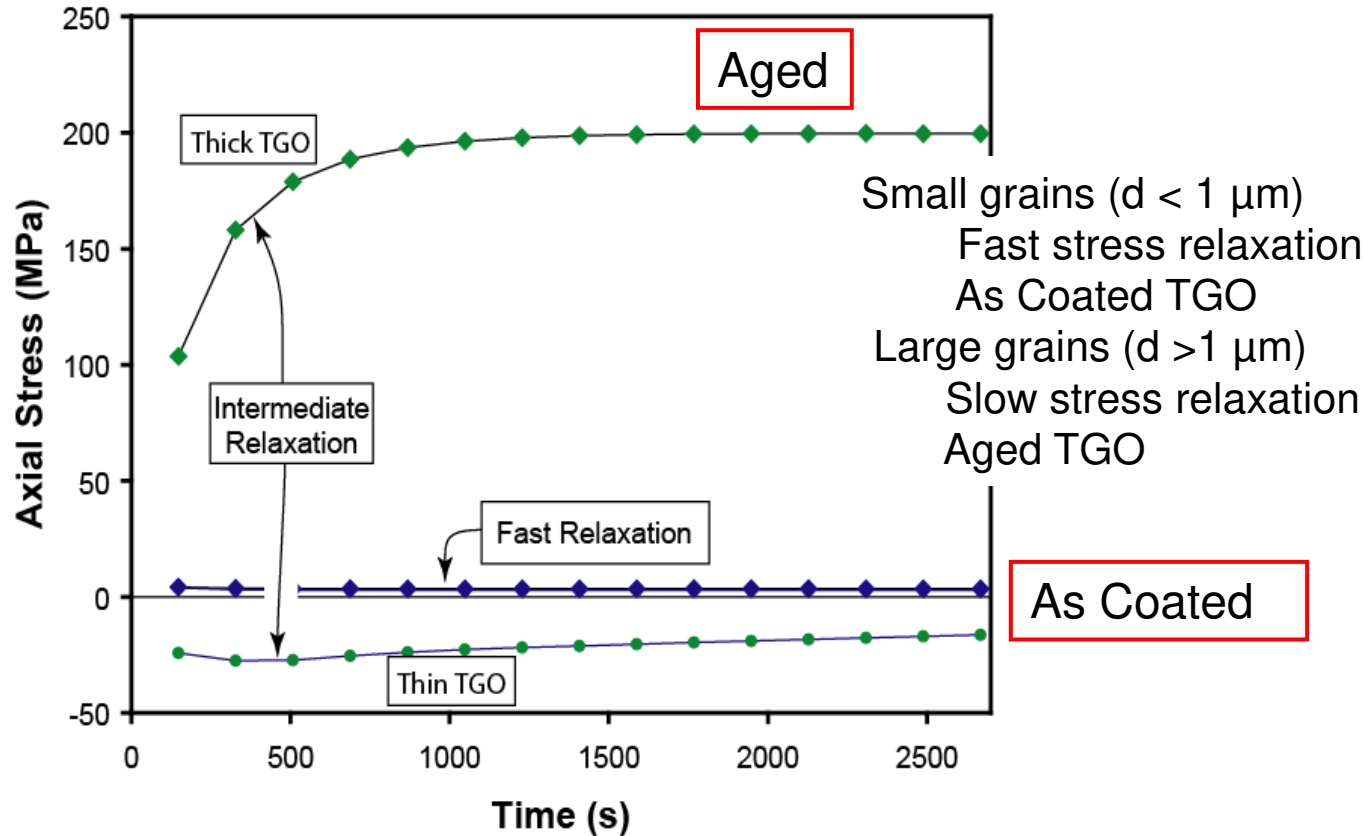
# Effect of TGO properties on stress accumulation



# Effect of TGO properties on stress accumulation



# Evolution of axial TGO-stresses



Hypothesis: Initiation of fatigue crack in TGO due to accumulation of tensile stress during subsequent TGMF-cycles



# Open questions – and a method to get answers

- Mechanical material properties of the coating materials are still unknown: Temperature dependent elastic properties, yield strength, creep laws of TGO (intermixed zone and coarse grained layer), bond coat and TBC
- Strategy:
  - measuring the strains in the coating system during TGMF by means of high energy X-ray diffraction
  - calculating (fitting) the respective material properties by means of finite element simulation





# Experimental set-up at Argonne Advanced Photon source

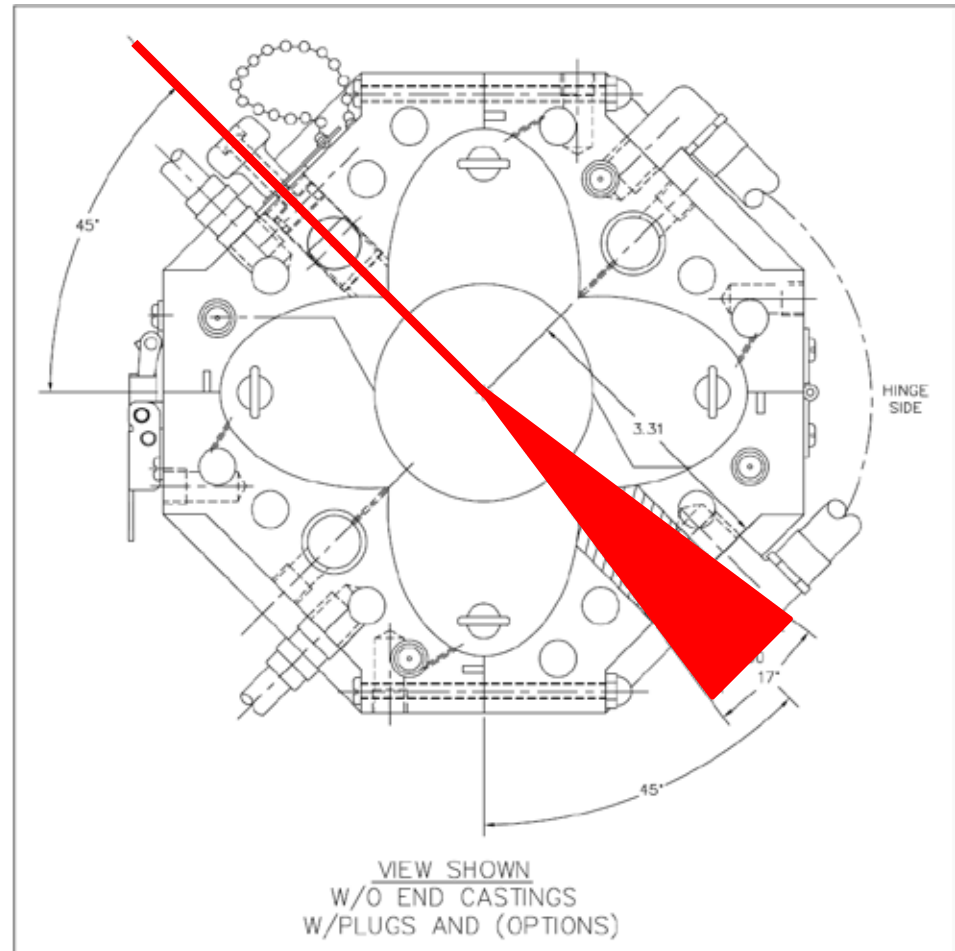
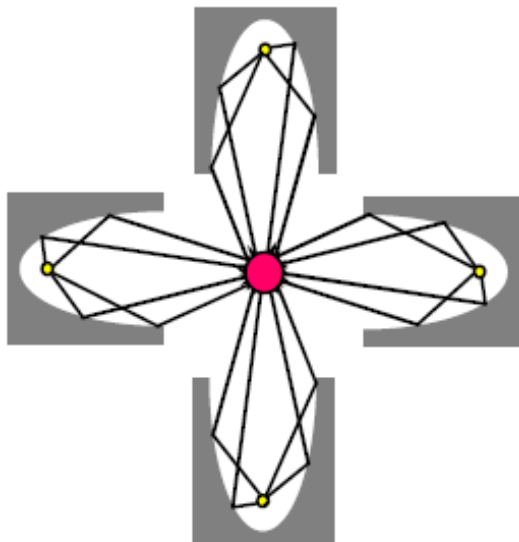


- Argonne National Laboratory, Argonne, Illinois
- Synchrotron high energy X-Ray beam-line; 65 keV beam energy



# Top view of heater and beam

- 4 focused infrared lamps
  - 8 kW total
- Beam exit window
  - $17^{\circ} 40'$



S. F. Siddiqui et al., *Rev. Sci. Instr.*, 84 - 083904 (2013)



# Servo-hydraulic testing machine on $\mu\text{m}$ - positioning rig



Assembling heater, grips and specimen at Argonne APS



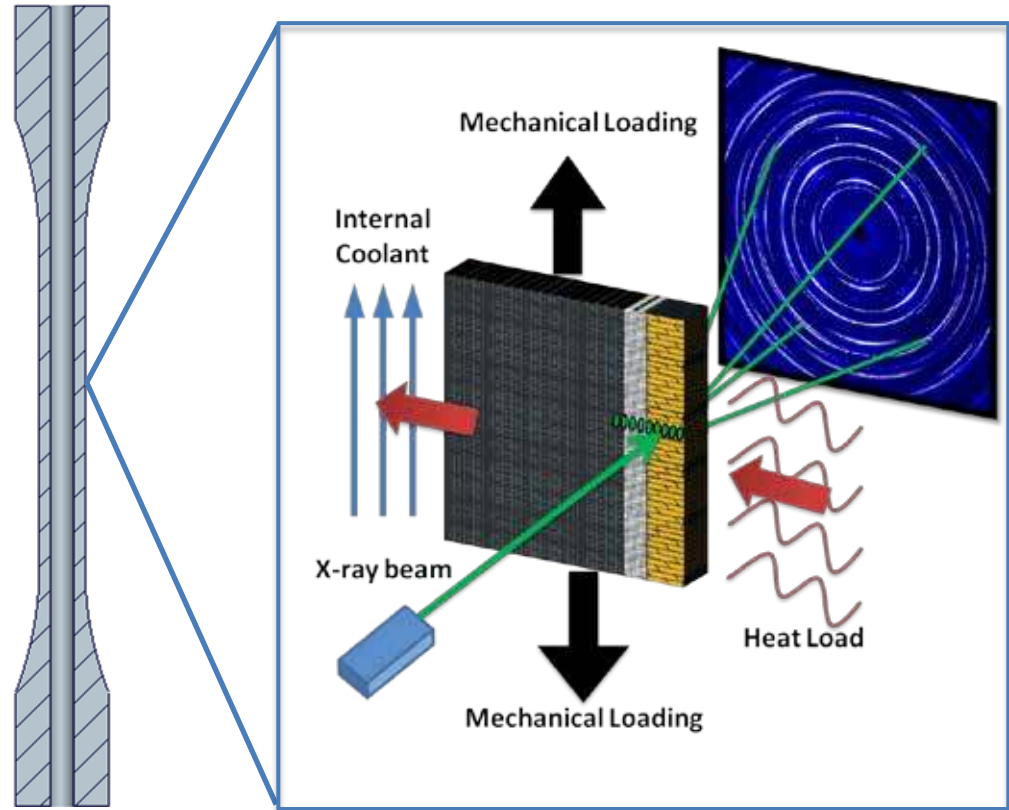
# Measurement method

## Loading parameter:

- thermal cycle (80 min)
- outer surface temperature max. 1000°C, temperature difference between outer and inner surface ca. 150°C
- variation of thermal gradient by variation of cooling flow rate
- superposition of mechanical load

## Beam parameter:

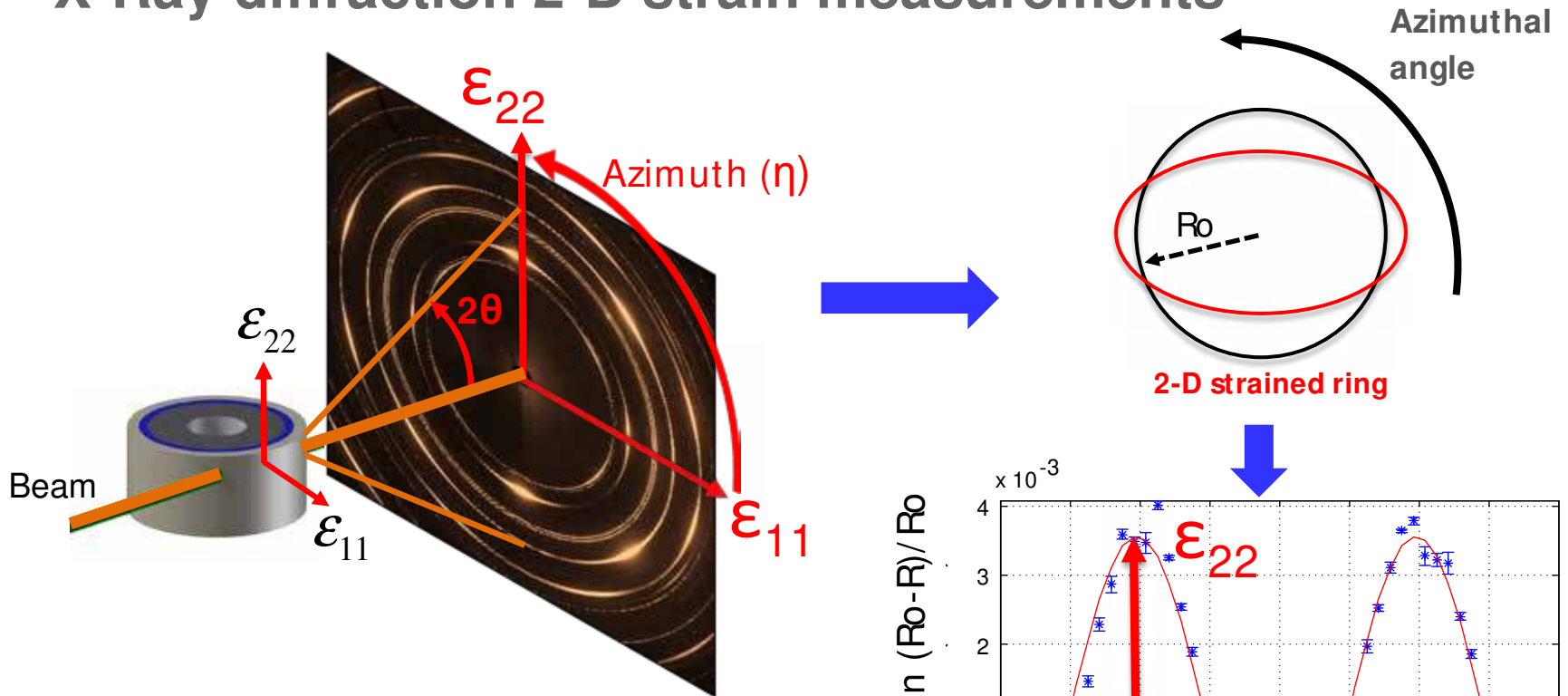
- 65 keV beam energy
- exposure time 0.5 to 15 sec.



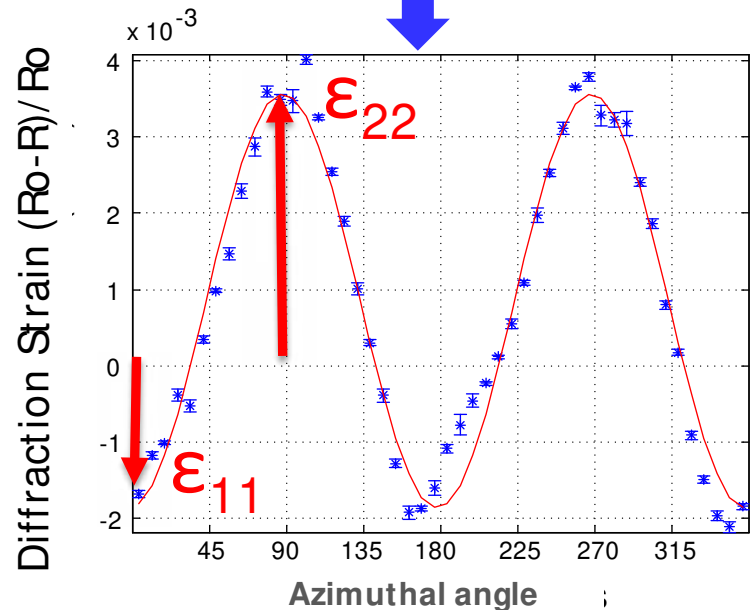
*K. Knipe et al., AIAA Structures, Struct. Dynamics & Mat. Conf., Boston, MA, 2013*



# X-Ray diffraction 2-D strain measurements



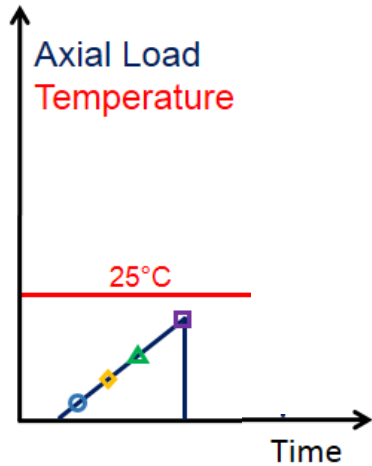
- Measure radial position around azimuthal angle
- Calculate each directional strain using  $(R_0-R)/R_0$ 
  - $R$  = measured radius
  - $R_0$  = strain free radius



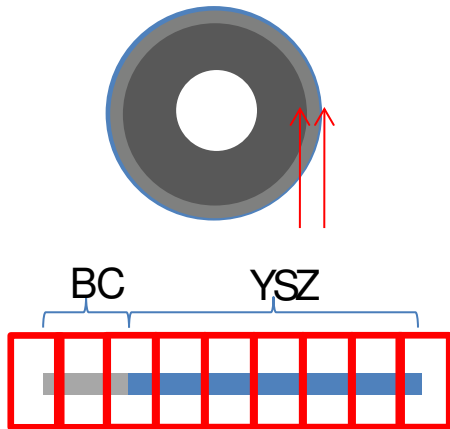
K. Knipe, Nature Comm. 5 (2014) article Nr. 4559



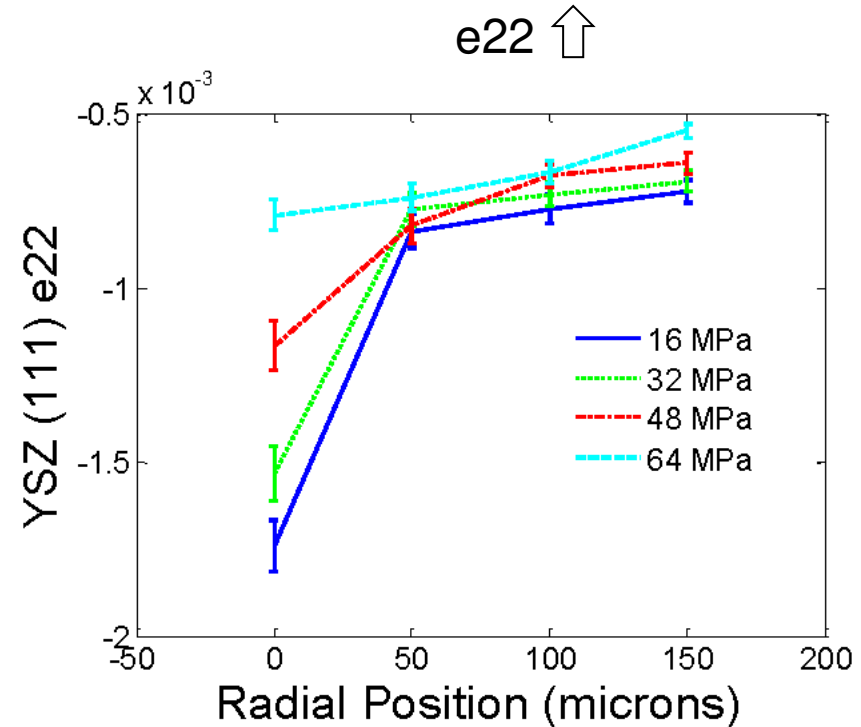
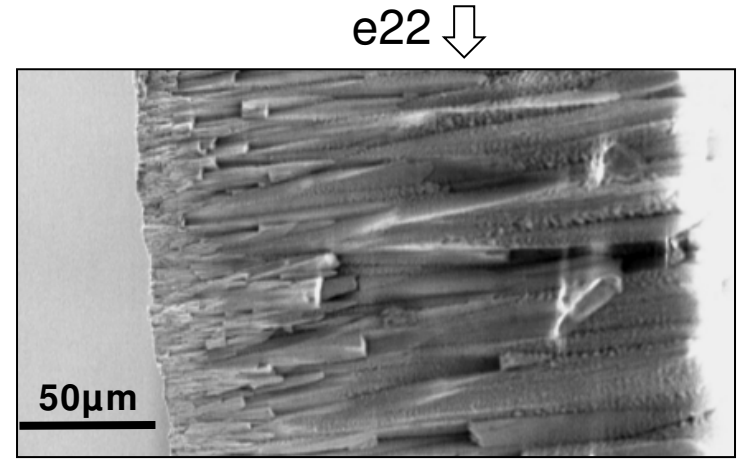
# YSZ - strain results



- No thermal gradient
- 25°C
- variation of mechanical load

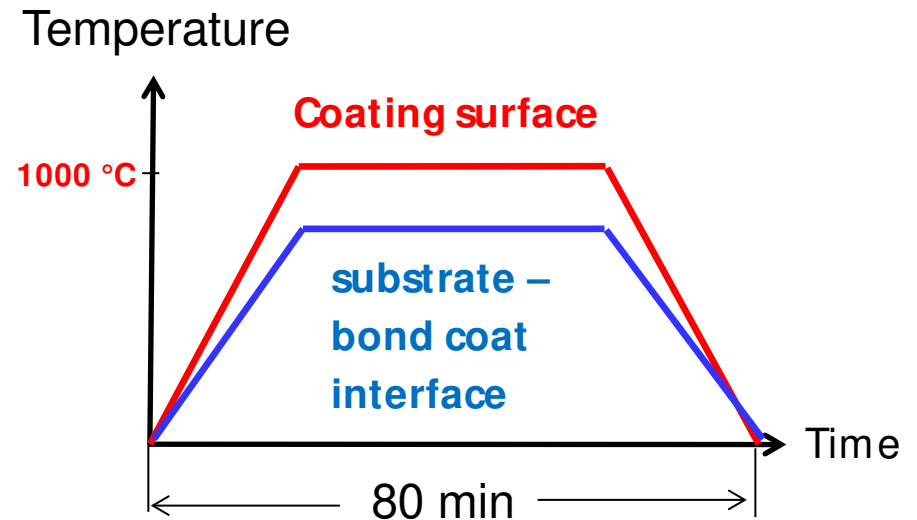


- X-Ray scan through coating thickness
- every 3.5 minutes
- window size 30 x 300 microns
- 10 window scan



# Strain measurement during cyclic loading

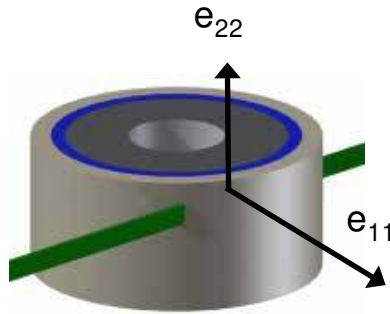
- Outer surface ramped up to 1000°C in 20 minutes and then held for 40 minutes
- Coolant flow rate for gradient varied
  - 30, 50, and 75 % max. flow (100 SLPM\* max)
- Constant nominal mechanical stress
  - 32, 64 and 128 MPa applied



SLPM\* = standard liter per minute



# Strain in YSZ during thermal cycle



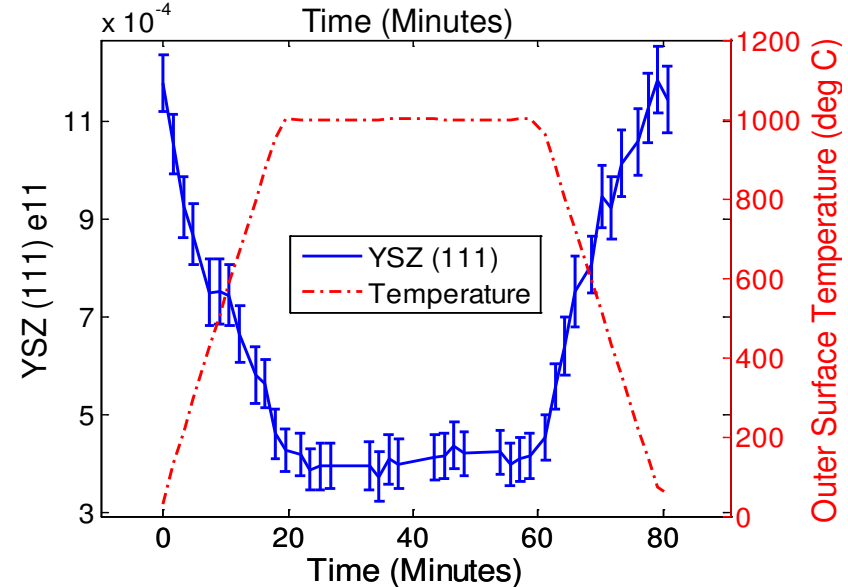
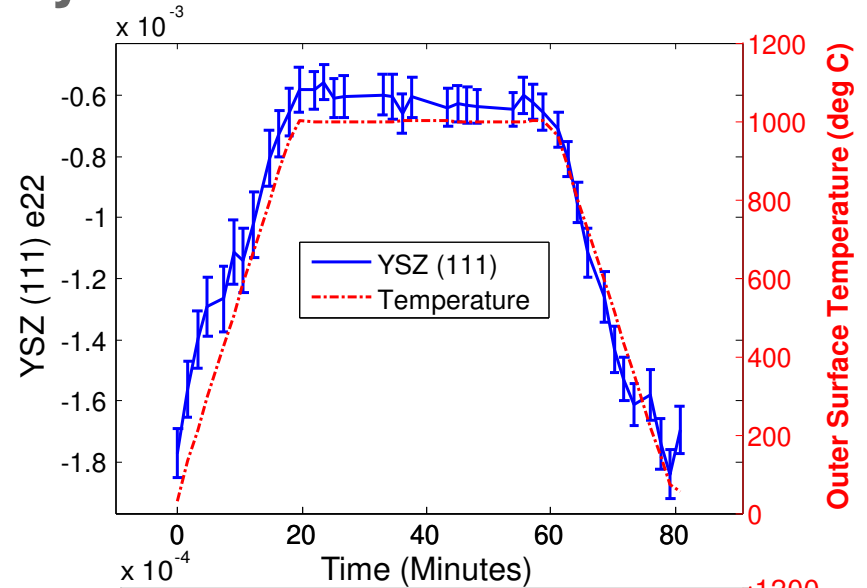
- 64 MPa
- 75% cooling air flow rate

at room temperature:

- compressive in plane strain  $e_{22}$
- tensile out of plane strain  $e_{11}$

at high temperature:

- strain reduces (closer to stress free condition at manufacturing temperature)

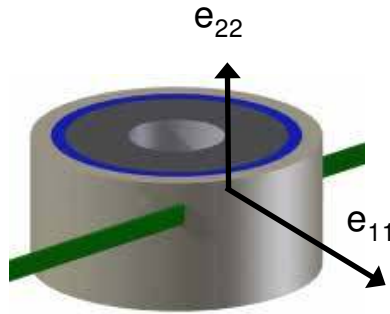


K. Knipe, Nature Comm. 5 (2014) article Nr. 4559





# Strain in bond coat $\beta$ -NiAl during thermal cycle



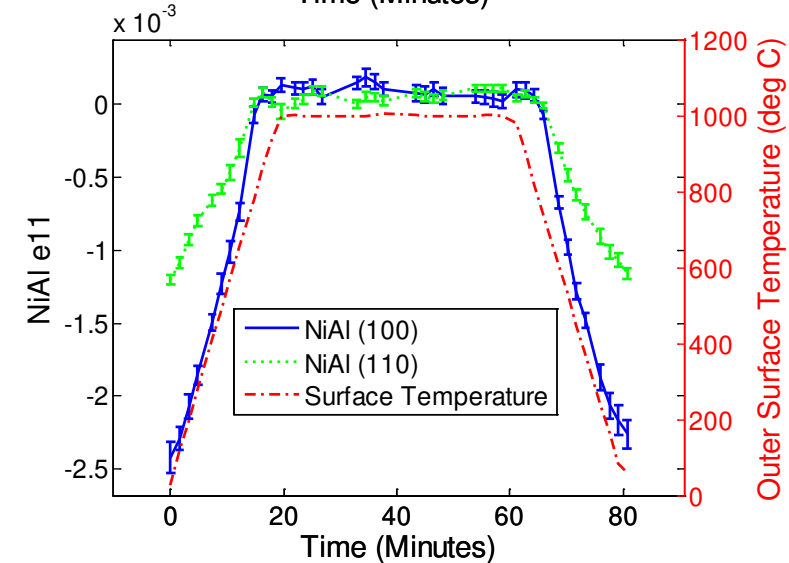
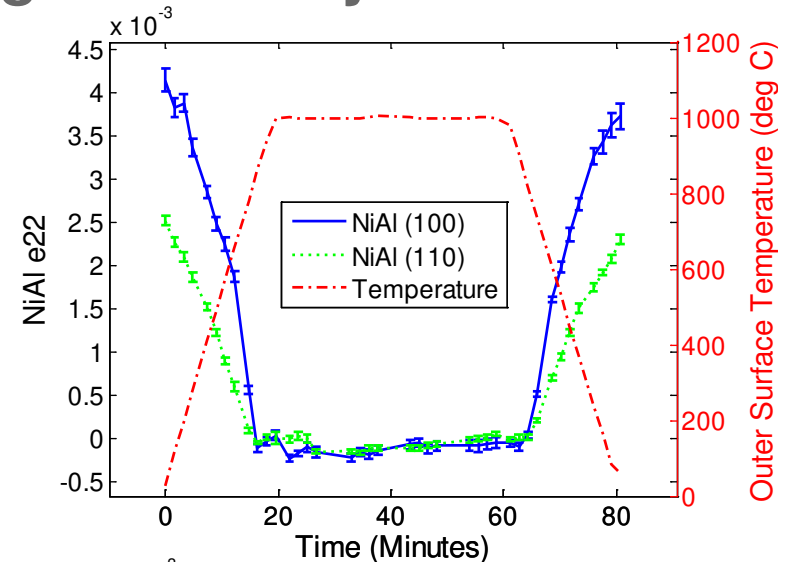
- 64 MPa
- 75% cooling air flow rate

at room temperature:

- tensile in plane strain  $e_{22}$
- compressive out of plane strain  $e_{11}$

at high temperature:

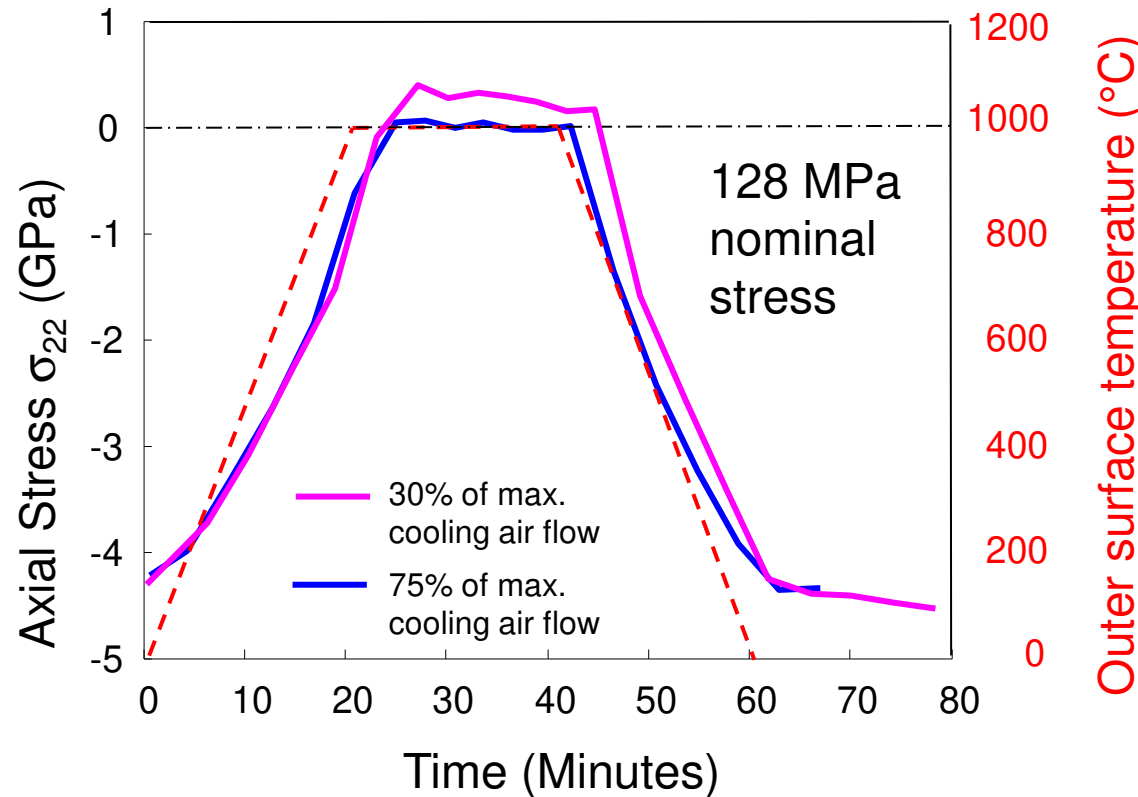
- strain reduces (stress free at manufacturing temperature)



K. Knipe, Nature Comm. 5 (2014) article Nr. 4559



# TGO stress in pre-aged specimen during thermal cycle



Pre-aged specimen:  
304h at 1000°C

- the TGO experience tensile stresses under TGMF loading depending on applied mechanical tensile load and thermal gradient.
- Relaxation occurs during dwell time at high temperature, which is a condition for accumulating tensile stress during cycling.



# Conclusions and outlook

- In situ strain measuring by X-ray diffraction
  - gives for each load case an equation for determining the respective material properties
  - test results can be used for validating numerical models and adapting laboratory experiments to more realistic conditions, e.g.
    - are dwell times and transients appropriate, e.g. time for relaxation processes within one load cycle appropriate? – example: stress accumulation in TGO
    - effect of time dependent processes captured?– TGO growth? Material property changes?
- Aim: validated realistic laboratory test for turbine blade materials for investigating damage mechanisms and contributing to life time modelling.
- Relevance-check of laboratory test: are observed damage mechanism and failure mode realistic?



# Thank you for your attention!

## Questions?

### Acknowledgements:

- This material is based upon work supported by the National Science Foundation Grants OISE 1157619 and CMMI 1125696
- German Science Foundation (DFG) grant SFB-TRR103, project A3
- Use of the Advanced Photon Source, an Office of Science User Facility operated for the U.S. Department of Energy (DOE) Office of Science by Argonne National Laboratory, was supported by the U.S. DOE under Contract No. DE-AC02-06CH11357.

Publication list 



# Publications

- J. Shi, A.M. Karlsson, B. Baufeld, M.Bartsch: *Evolution of surface morphology in thermo-mechanically cycled NiCoCrAlY- bond coats*, Mat. Sci. & Eng. A 434 (2006) 39-52
- M.Bartsch, B. Baufeld, M. Heinzelmann, A. M. Karlsson, S. Dalkilic, L. Chernova: *Multiaxial thermo-mechanical fatigue on material systems for gas turbines*, Materialwiss. & Werkstofftechnik 38, (2007) 712-719
- B. Baufeld, M. Bartsch, M. Heinzelmann: *Advanced thermal mechanical fatigue testing of CMSX-4 with oxidation protection coating*, Int. J. fatigue 30 (2008) 219-225
- M. Bartsch, B. Baufeld, S. Dalkilic, L. Chernova, M. Heinzelmann: *Fatigue cracks in a thermal barrier coating system on a super alloy in multiaxial thermomechanical testing*, Int. J. fatigue 30 (2008) 211-218
- M. Hernandez, A. Karlsson, M. Bartsch: *On TGO creep and the initiation of a class of fatigue cracks in thermal barrier coatings*, Surf. Coat. Techn. 203 (2009) 3549-3558
- M. T. Hernandez, D. Cojocar, A. M. Karlsson, M. Bartsch: *On the crack opening of a characteristic crack due to thermo-mechanical fatigue testing of thermal barrier coatings*, Comp. Mat. Sci. (50) (2011) 2561-2572
- S. F. Siddiqui, K. Knipe, A. Manero, C. Meid, J. Schneider, J. Okasinski, J. Almer, A.M. Karlsson, M. Bartsch, S. Raghavan: *Synchrotron X-Ray Measurement Techniques for Thermal Barrier Coated Cylindrical Samples under Thermal Gradients*, Review of Scientific Instruments, 84 - 083904 (2013)
- K. Knipe, A. Manero, S. F. Siddiqui, C. Meid, J. Wischek, J. Okasinski, J. Almer, A. M. Karlsson, M. Bartsch & S. Raghavan: *Strain response of Thermal Barrier Coatings captured under extreme engine environments through Synchrotron X-ray Diffraction*, Nature Communications 5 (2014), article number 4559

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