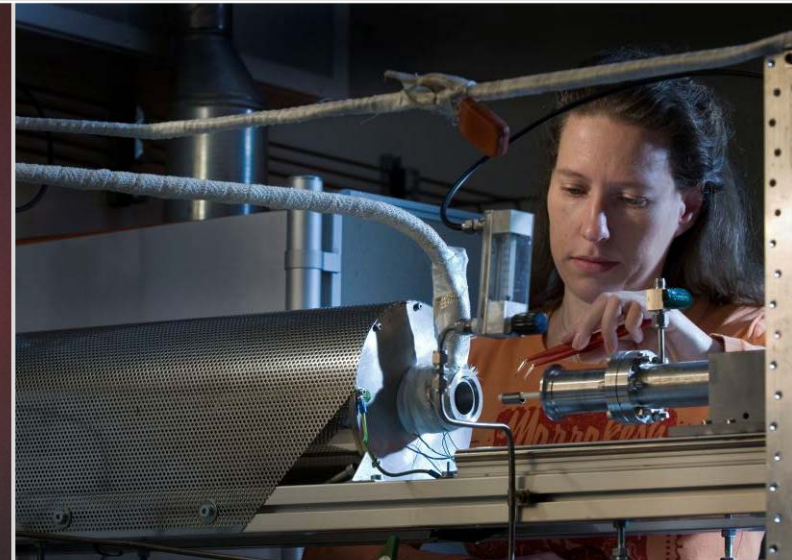
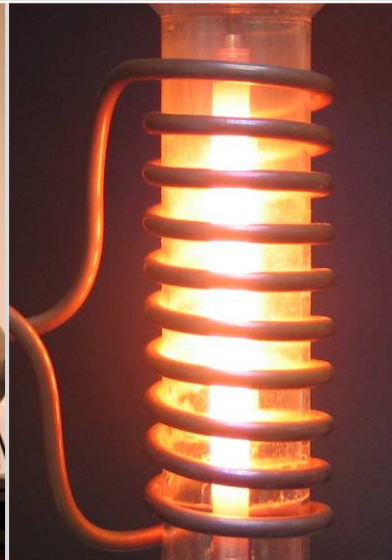


# 25 years QUENCH program. Highlights of separate-effects tests

M. Steinbrück, J. Stuckert, M. Große et al.

*25th International QUENCH Workshop, Karlsruhe, 22-24 October 2019*

Institute for Applied Materials IAM-AWP & Program NUSAFE



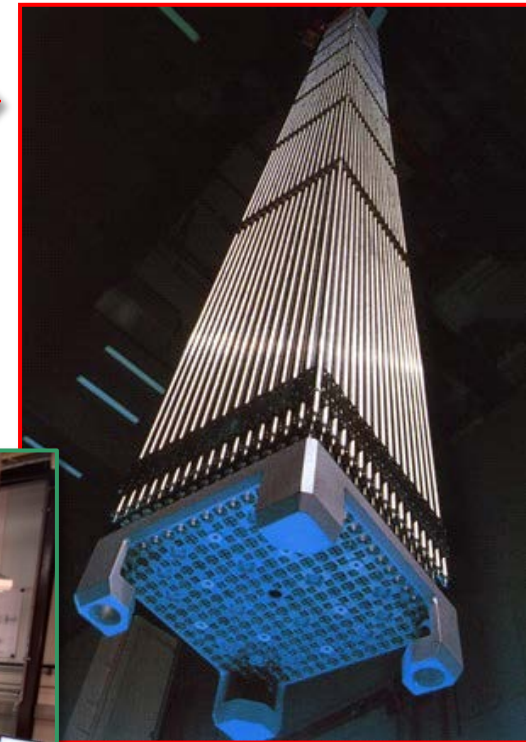
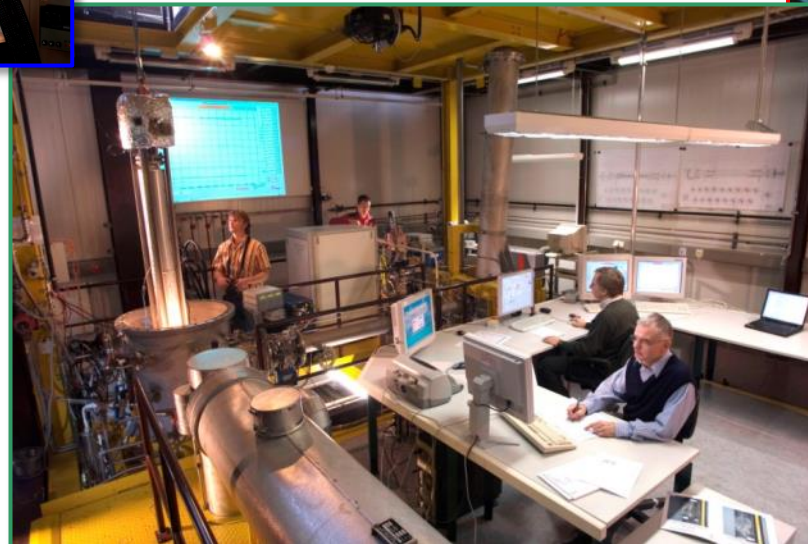
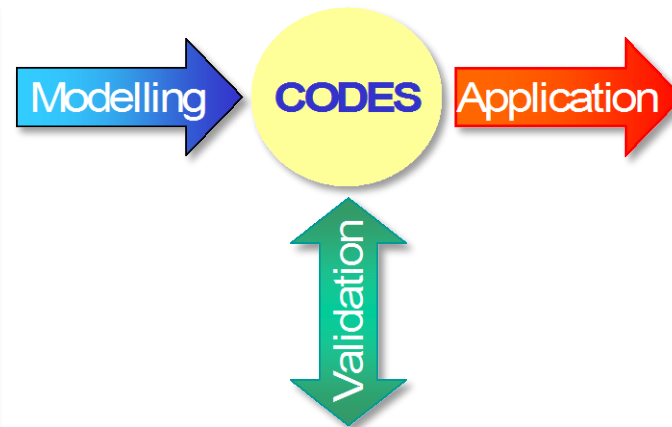
# QUENCH program at KIT

Investigation of hydrogen source term and materials interactions during LOCA and early phase of severe accidents including reflood



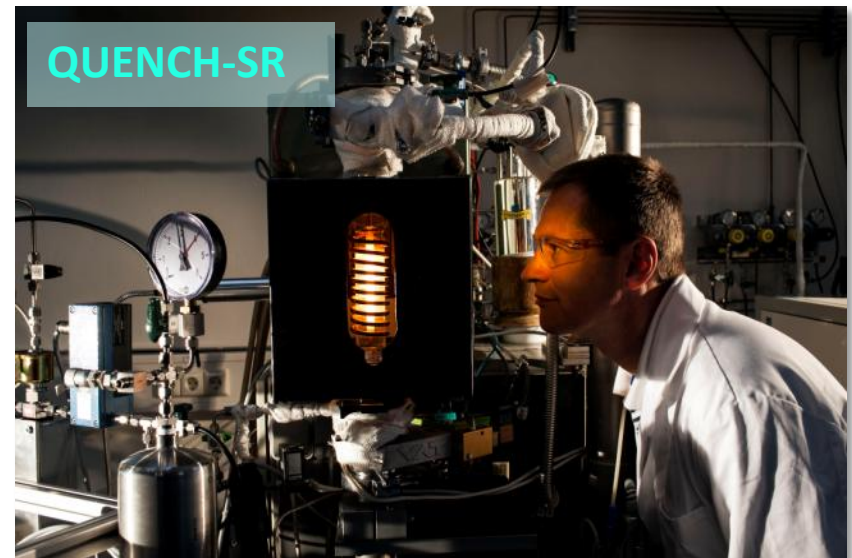
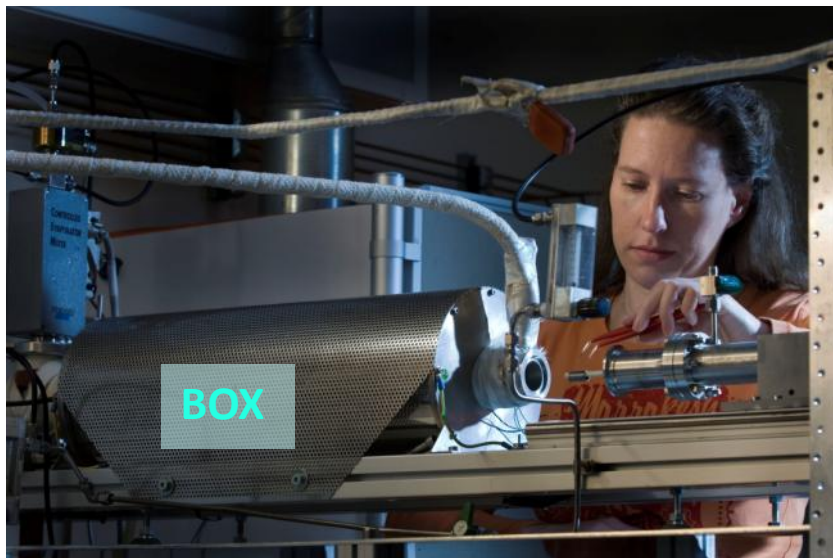
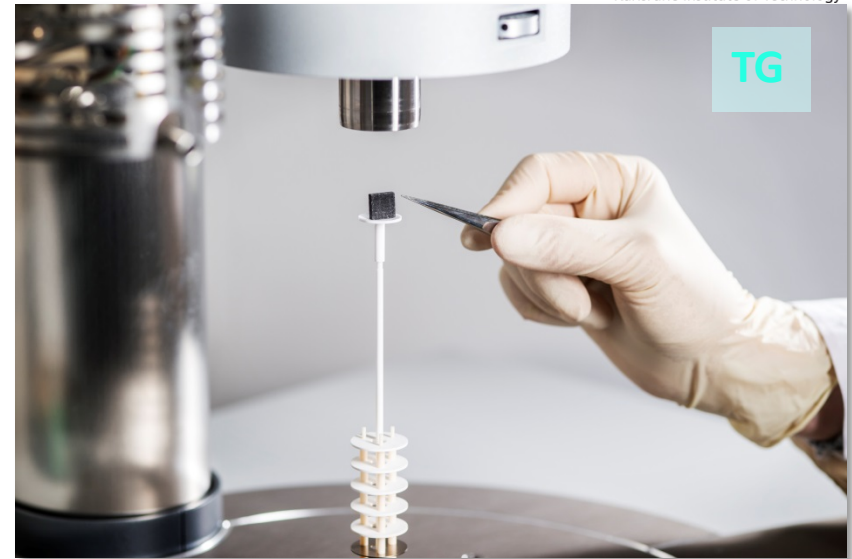
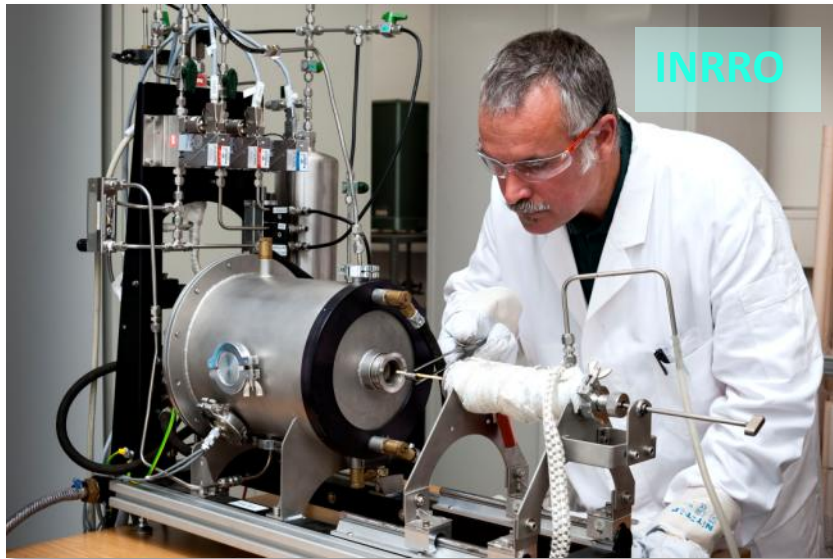
Separate-effects tests

Bundle experiments



PWR fuel element

# QUENCH separate-effects tests: Main setups



# Core materials in Light Water Reactors

- $\text{UO}_2$ (/ $\text{PuO}_2$ ) fuel: 100-200 t
- Zry cladding + grid spacers: 20-40 t
- Zry canister (BWR): 40 t
- Various steels, Inconel: >500 t (incl. RPV)
- $\text{B}_4\text{C}$  absorber (BWR, VVER, ...): 0.3-2 t
- AgInCd absorber (PWR): 3-5 t

## Environment

- **Water, steam**
  - Air
  - Nitrogen
- } After failure of RPV/primary circuit  
and in spent fuel pool



PWR fuel assembly



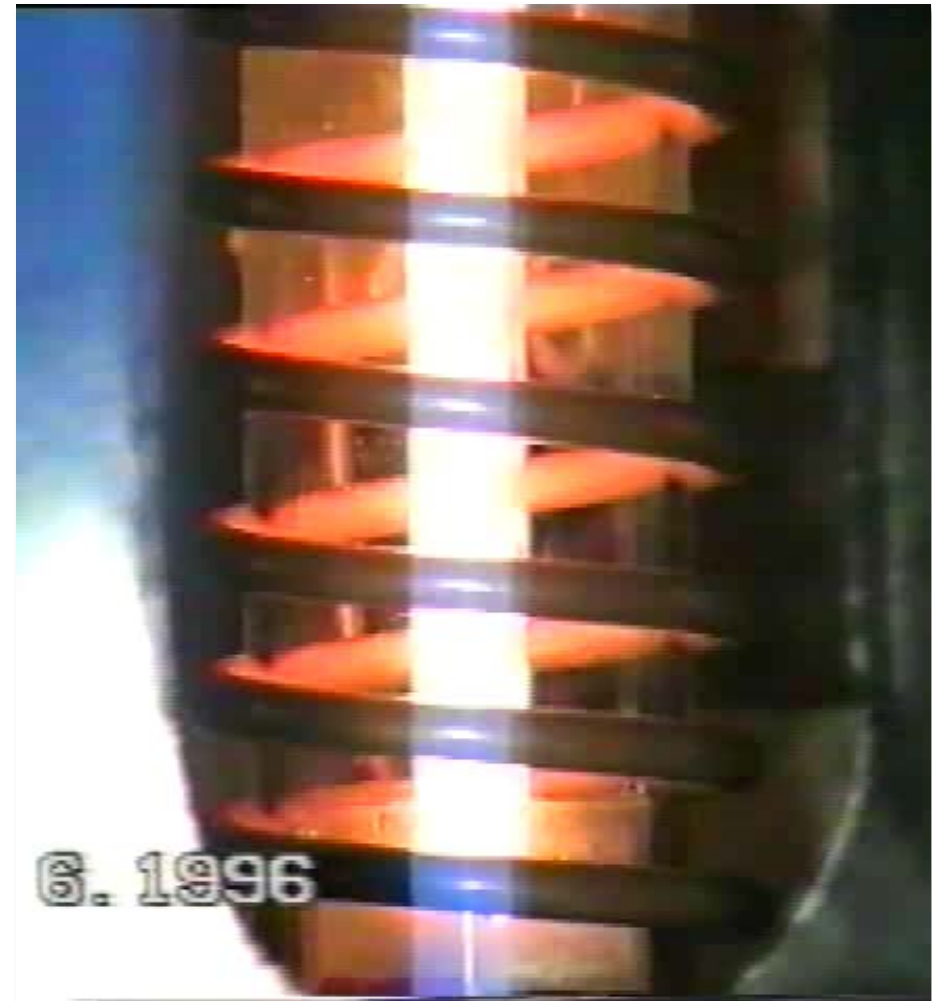
BWR control blade

- Generally, high-temperature ( $> 600^{\circ}\text{C}$ ) oxidation and materials interactions of zirconium alloys (cladding), absorber materials, and structure materials in well-defined atmospheres
- Quenching of pre-oxidized cladding
- Oxide shell failure criterion
- Interaction between Zr melt and  $\text{ZrO}_2$  ( $\text{UO}_2$ ) ceramic
- Zr alloy oxidation in steam, oxygen, air, mixtures
- Hydrogen release and absorption
- $\text{B}_4\text{C}$  absorber rod oxidation degradation
- AgInCd absorber rod failure
- ATF cladding materials
- ...
- SETs were made mostly in connection with corresponding bundle tests

# Early experiments

## Single-rod QUENCH tests

- 15-cm rods filled with  $ZrO_2$  pellets
- Direct inductive heating
- Video recording
- Mass spectrometer for analysis of hydrogen release
- Parameters:
  - Pre-oxidation 0-350  $\mu\text{m}$
  - 1000-1600°C at onset of quenching
  - Quenching with hot/cold water or steam
  - Flooding rate 1.5 cm/s

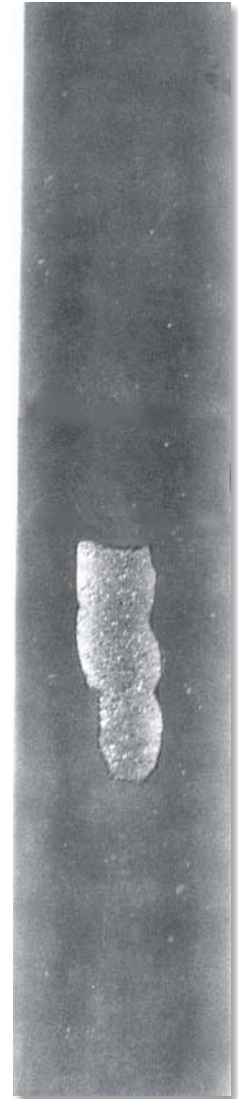
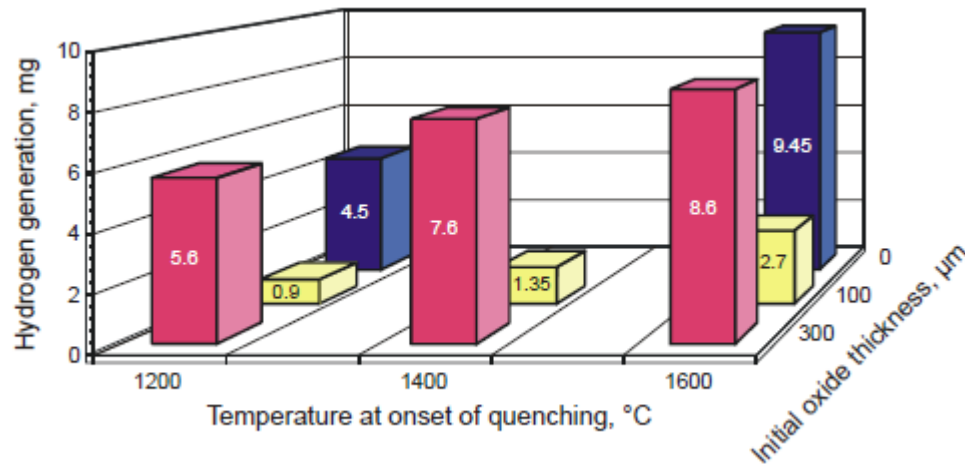
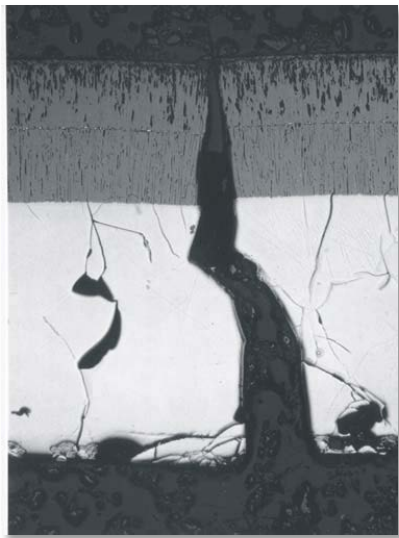


Reflood from 1400°C

# Single-rod QUENCH tests – Main results

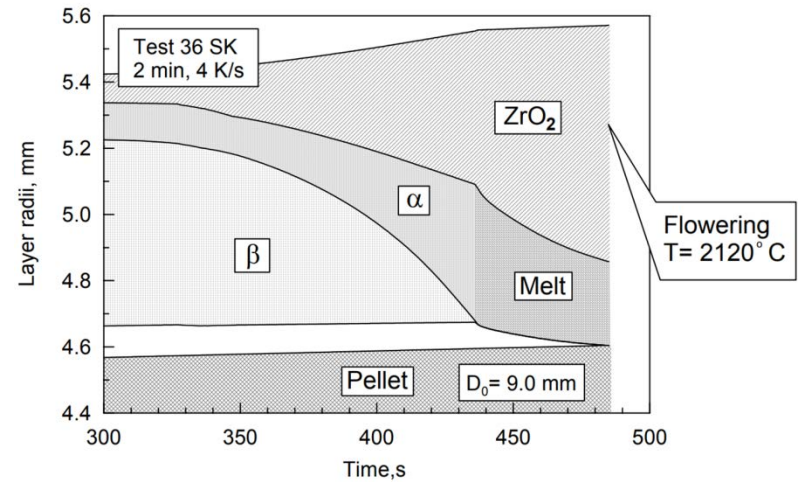
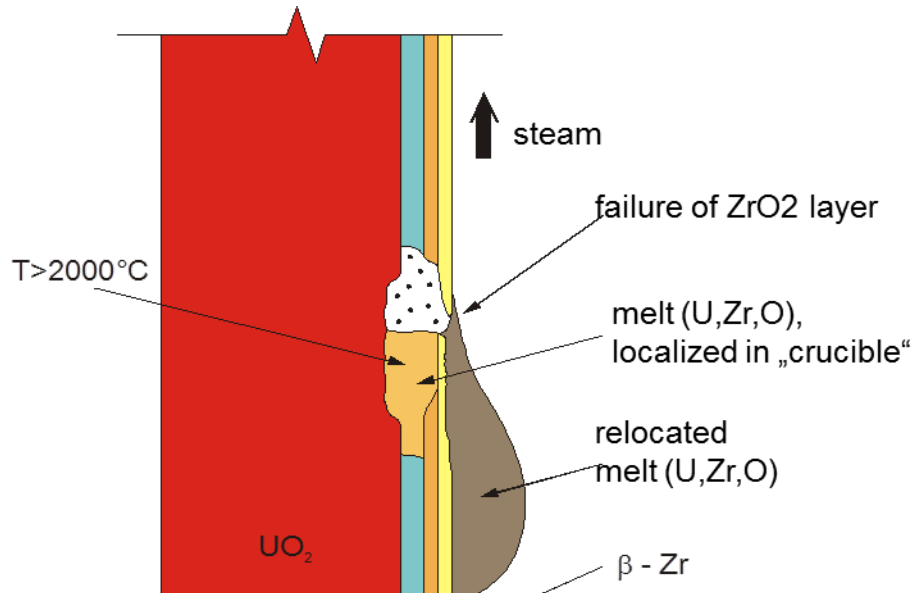


- Through-wall crack for pre-oxidation  $>200 \mu\text{m}$  with a density of  $0.5 \text{ mm/mm}^2$
- Oxidation of crack surfaces connected with hydrogen absorption by the metal
- Localized spalling of thick oxide scales

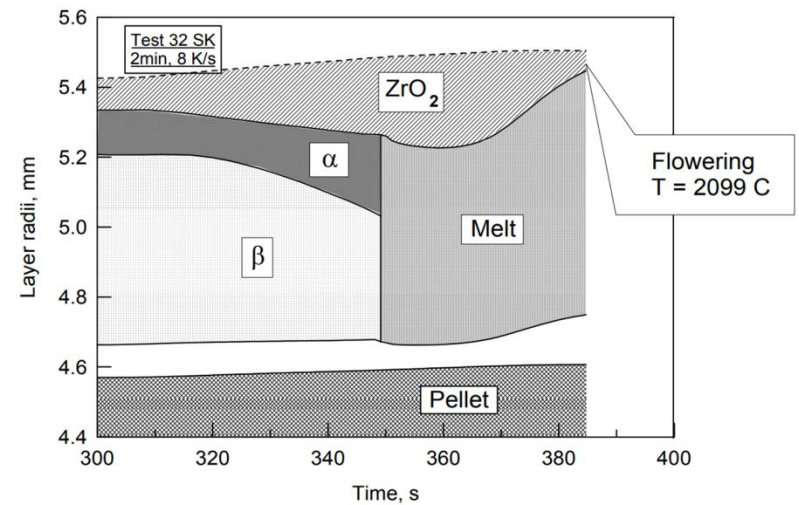




# Oxide shell failure

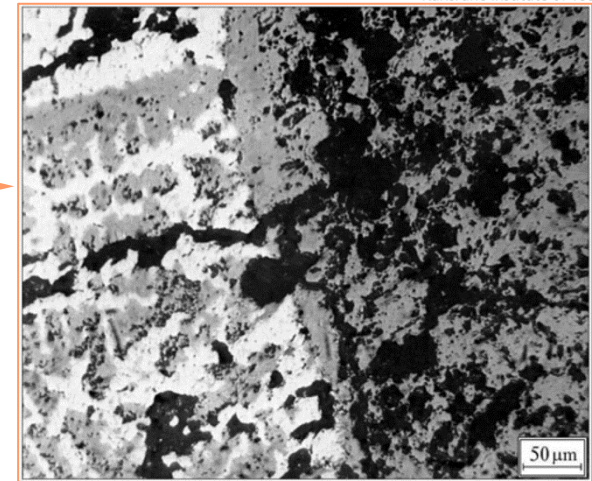
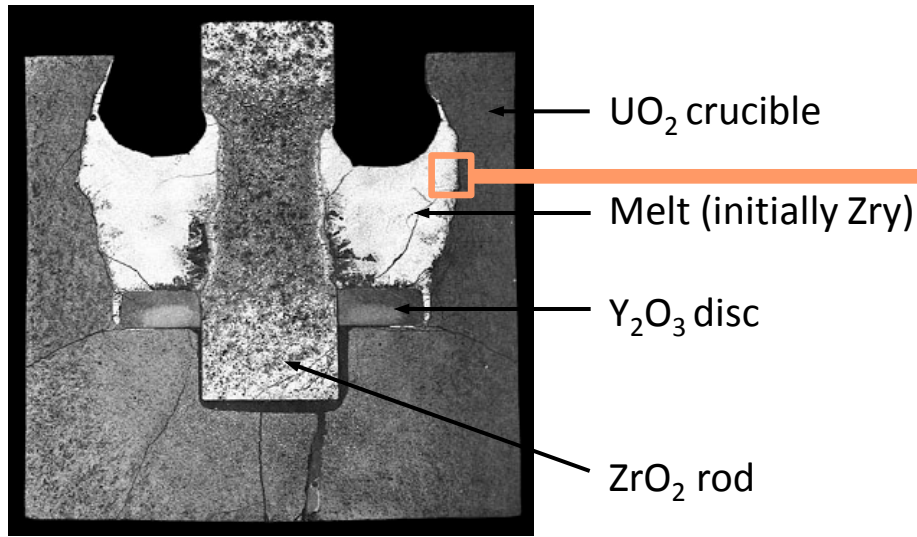


SVECHA simulation: slow heating

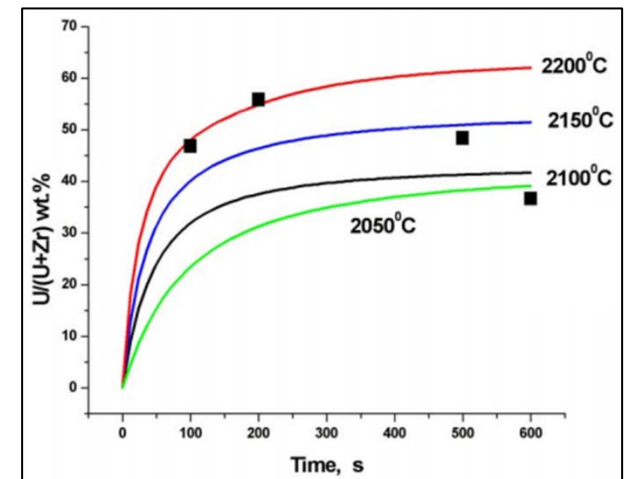
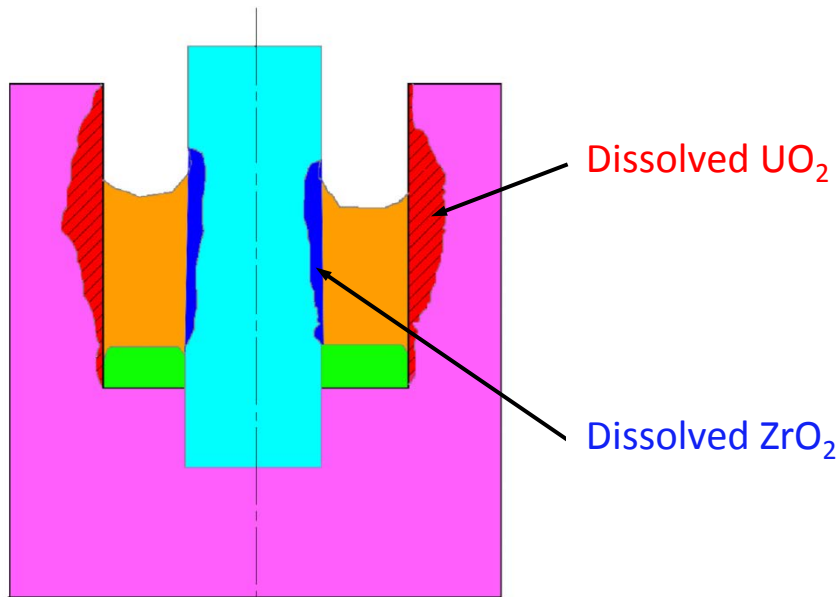


SVECHA simulation: rapid heating

# UO<sub>2</sub>/ZrO<sub>2</sub> dissolution by Zr melt (COLOSS project)

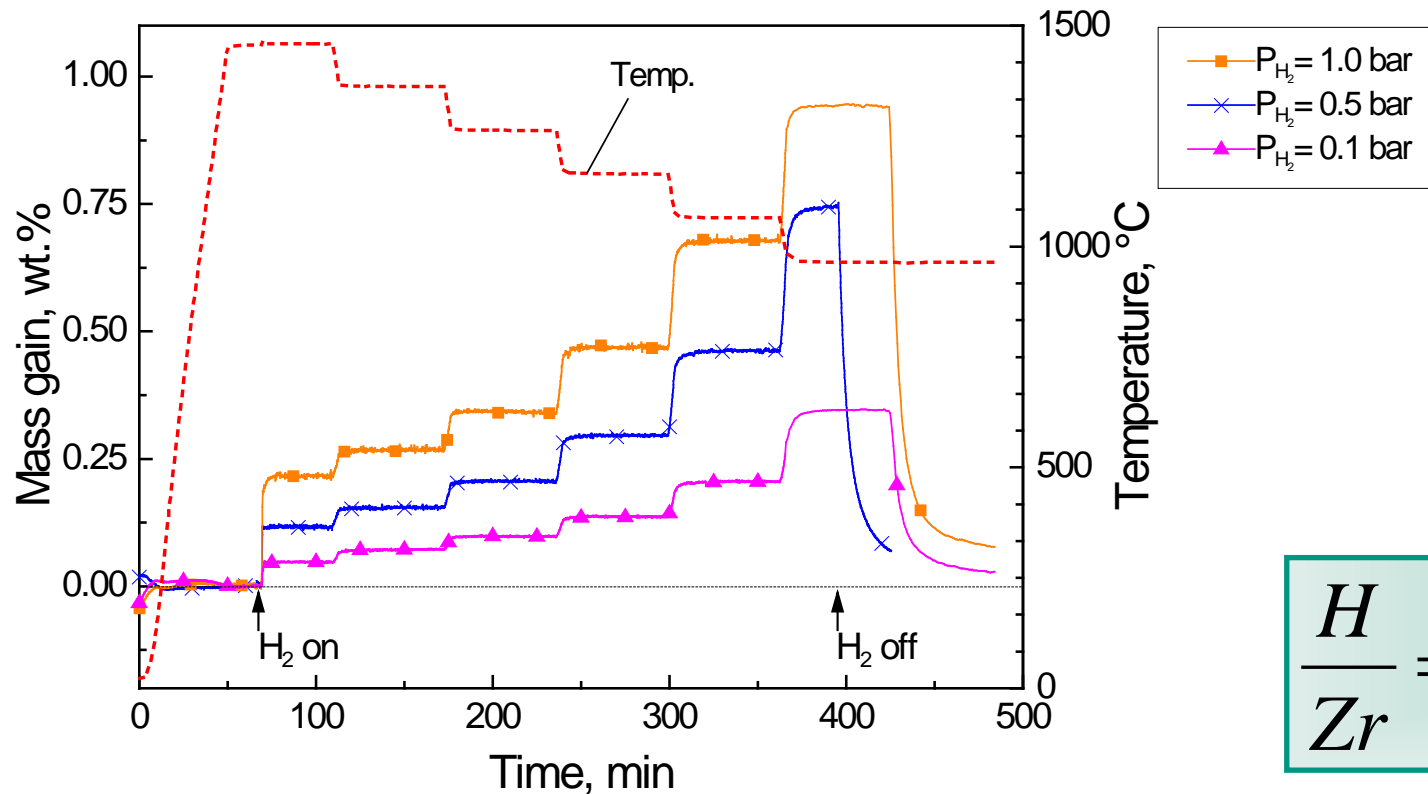


(U, Zr)O<sub>2-x</sub> precipitates in melt and transition zone between melt and crucible



SVECHA simulation: calculated and measured U content in the melt

# Hydrogen uptake of Zircaloy-4

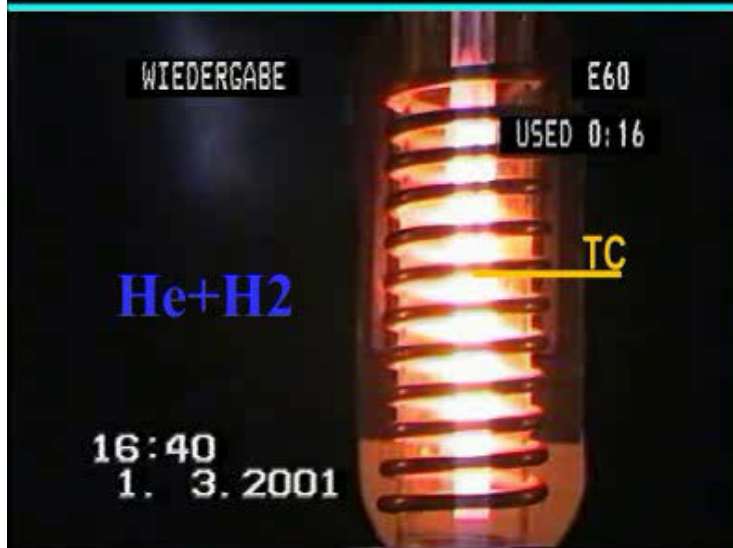
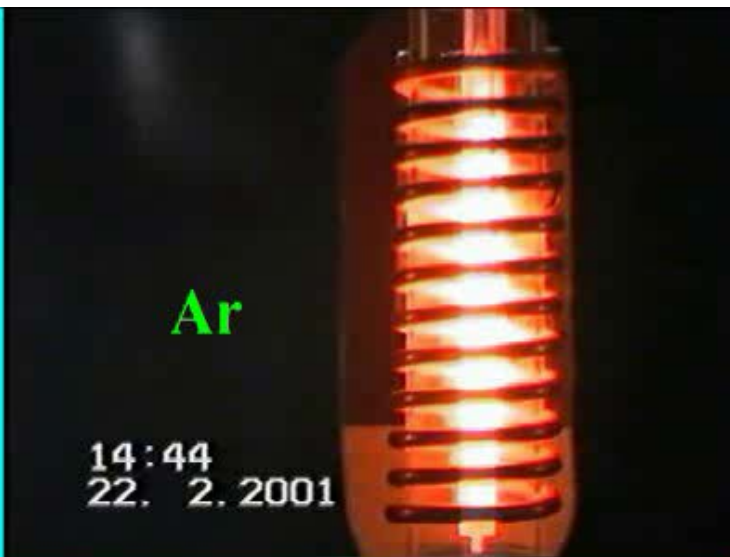
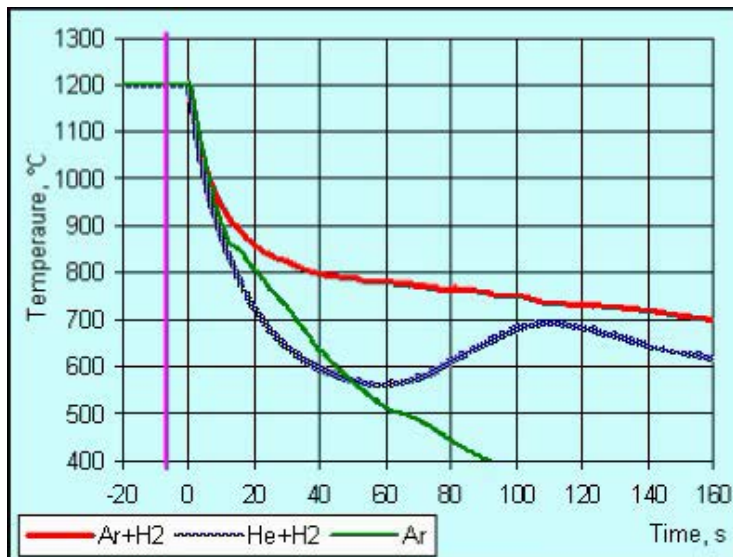


$$\frac{H}{Zr} = k_S \cdot \sqrt{P_{H_2}}$$

Sieverts' law

- ➡ Sieverts' law constant determined
- ➡ No difference between Zry-4 and M5<sup>®</sup>
- ➡ Reduced H solubility with increasing O content in the metal
- ➡ Fast establishment of equilibrium

# Exothermal effect of hydrogen uptake



# Oxidation of zirconium alloys and hydrogen behavior

# High-temperature oxidation of zirconium alloys

- Most cladding alloys consist of 98-99 wt% zirconium plus some alloying elements (Sn, Nb, Fe, Cr, ...)

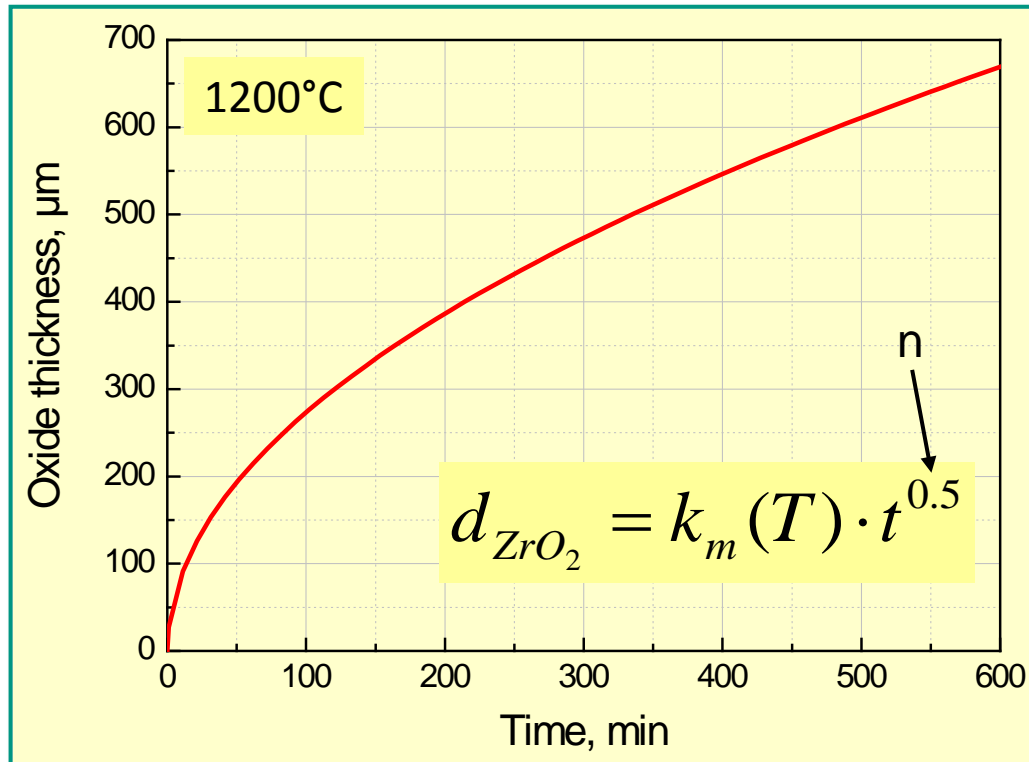
Element	Zircaloy-4	D4	M5	E110	ZIRLO
Nb	-	-	1	1	1
Sn	1.5	0.5	0.01	-	1
Fe	0.2	0.5	0.05	0.008	0.11
Cr	0.1	0.2	0.015	0.002	< 0.01

- In steam, oxygen, nitrogen, air, and various mixtures
- Temperature: 600-1600°C

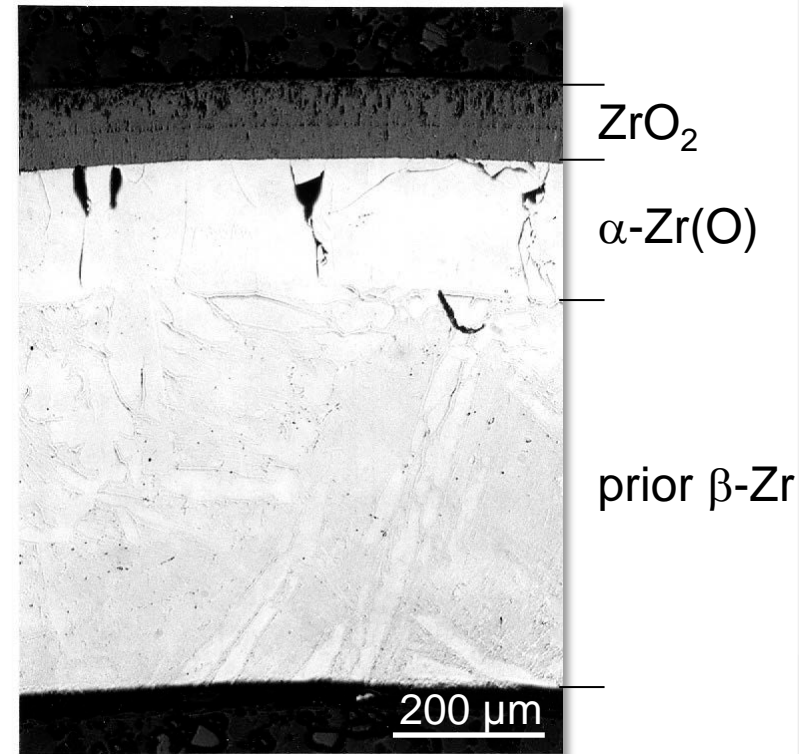


# Oxidation in steam (oxygen): Text book knowledge

- Parabolic oxidation correlations determined by the diffusion of oxygen through growing oxide scale



Oxide thickness during oxidation of Zry at 1200°C in steam



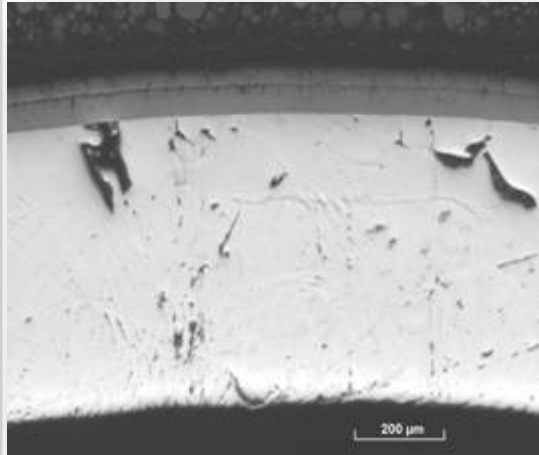
20 min at 1200°C in steam

## Deviation from parabolic kinetics

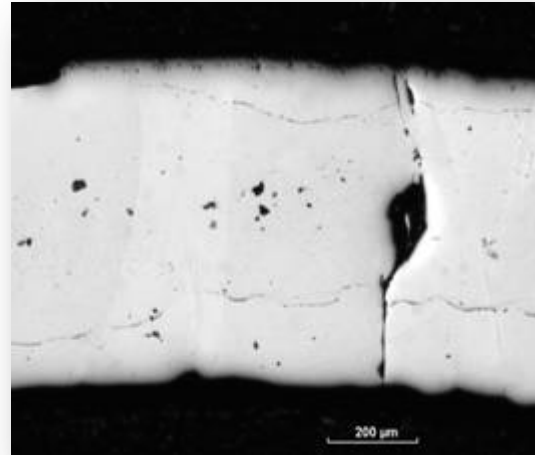
- Starvation conditions at low oxidant flow rates
- Cubic (sub-parabolic) kinetics for  $T < 1000^{\circ}\text{C}$  ( $n < 0.5$ )
- Breakaway ( $n \approx 1$  after transition)
- Nitrogen ( $n \approx 1$  after transition)



# Steam starvation

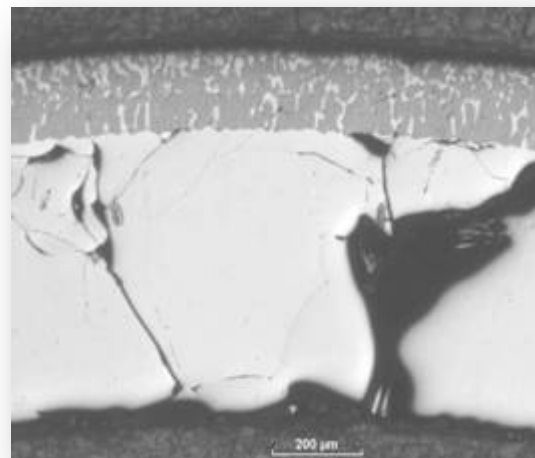
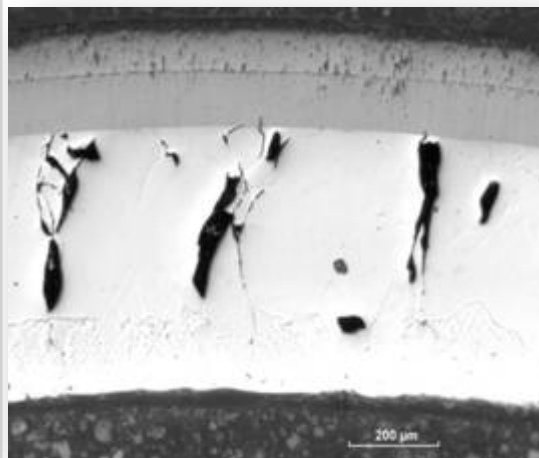


Oxidation



Steam starvation at 1700 K

Dissolution of  
oxide scale

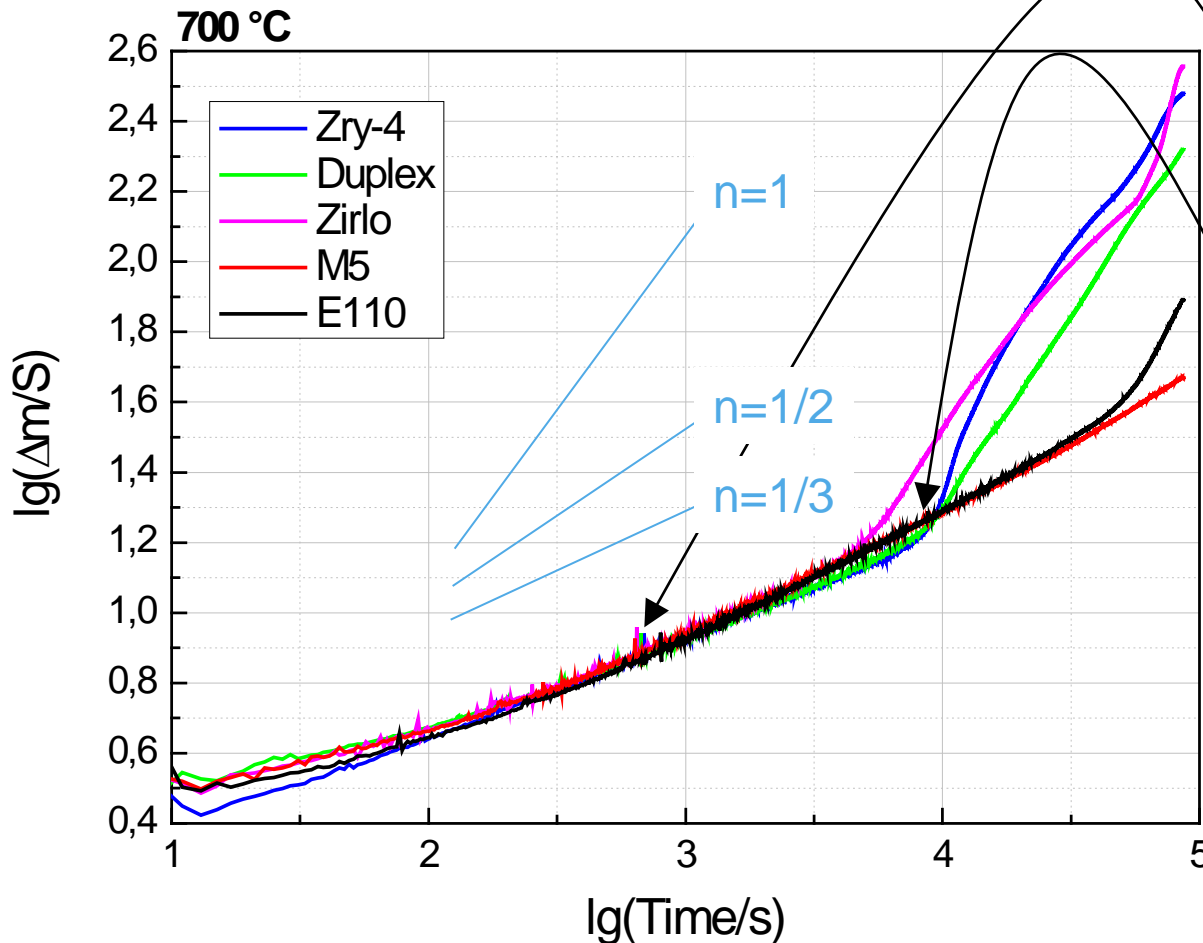


Thinning of oxide scale and  
precipitation of  $\alpha$ -Zr(O) in  
oxide

➔ Weakening of protective  
effect of  
ZrO<sub>2</sub> oxide layer

# Oxidation in steam (oxygen)

- Deviations from parabolic kinetics at temperatures  $<1050^{\circ}\text{C}$



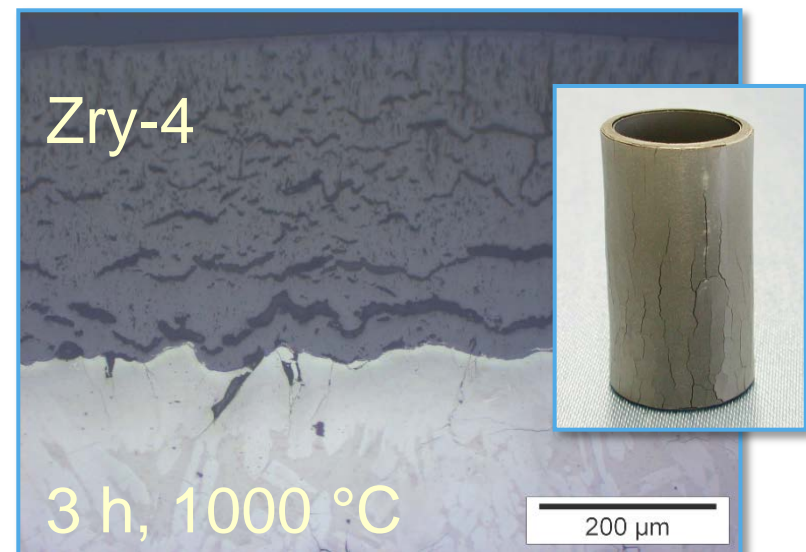
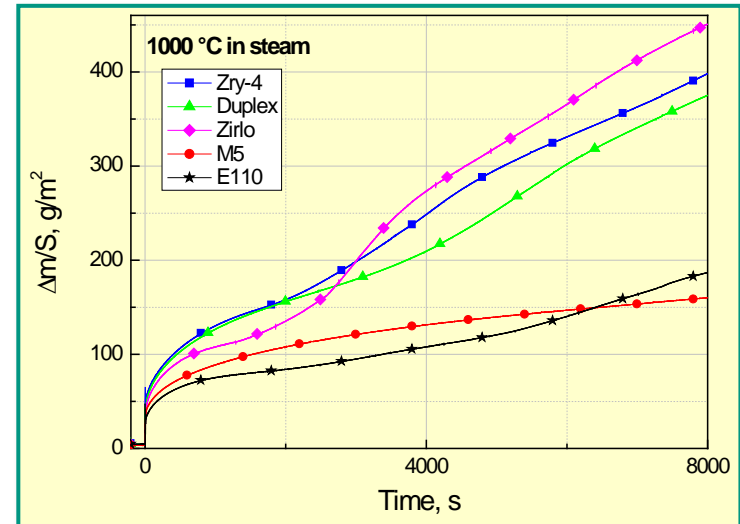
- Sub-parabolic (cubic) kinetics

- Transition from (sub-)parabolic to linear kinetics after critical time / oxide thickness due to breakaway

- Similar kinetics of all alloys before transition, but strongly varying behavior at and after transition

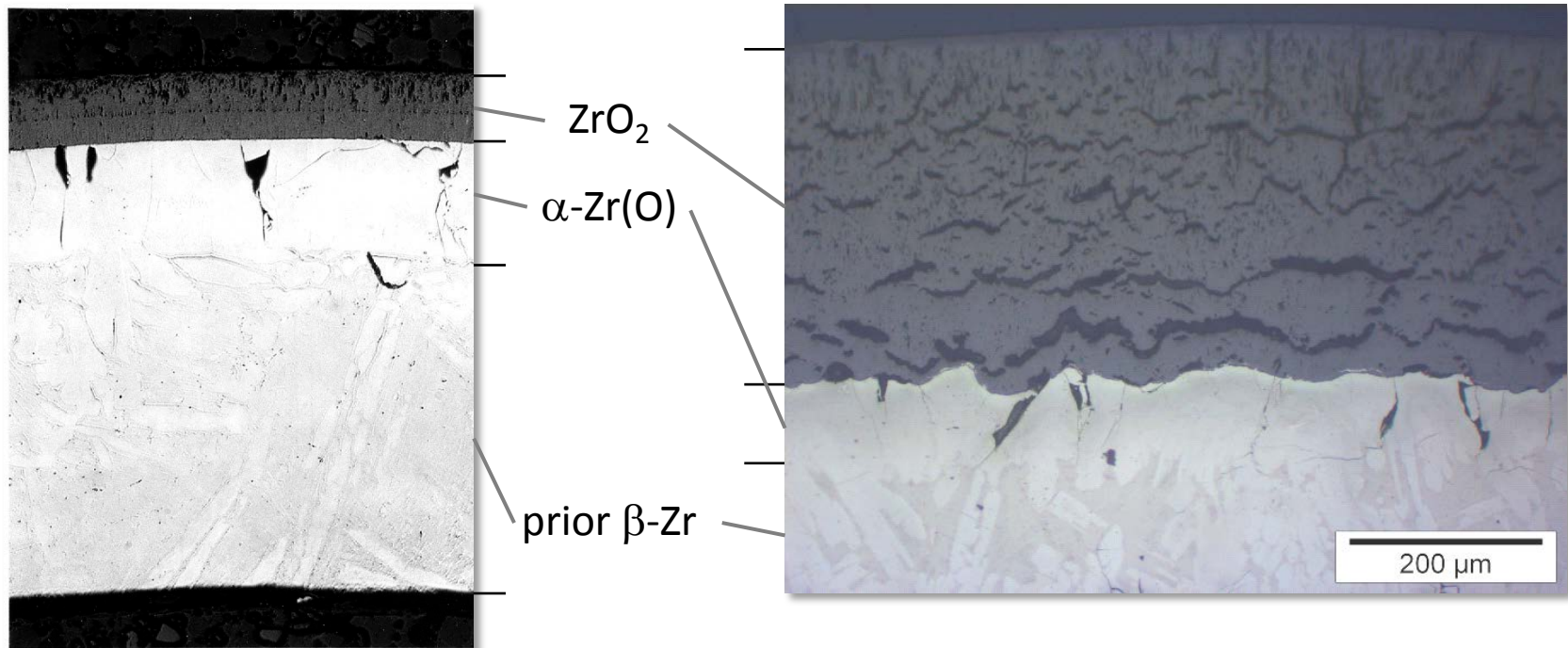
# Breakaway oxidation

- ➔ Loss of protective properties of oxide scale due to its mechanical failure.
- Breakaway is caused by phase transformation from meta-stable tetragonal to monoclinic oxide and corresponding change in density up to ca. 1050°C.
- Critical times and oxide thicknesses for breakaway strongly depend on type of alloy and boundary conditions (30-60 min at 1000°C and 8 h at 600°C).
- During breakaway significant amounts of hydrogen can be absorbed (>40 at.%, 7000 wppm) due to local enrichment of H<sub>2</sub> in pores and cracks near the metal/oxide boundary (“hydrogen pump”).



# Hydrogen uptake during HT oxidation of Zry in steam

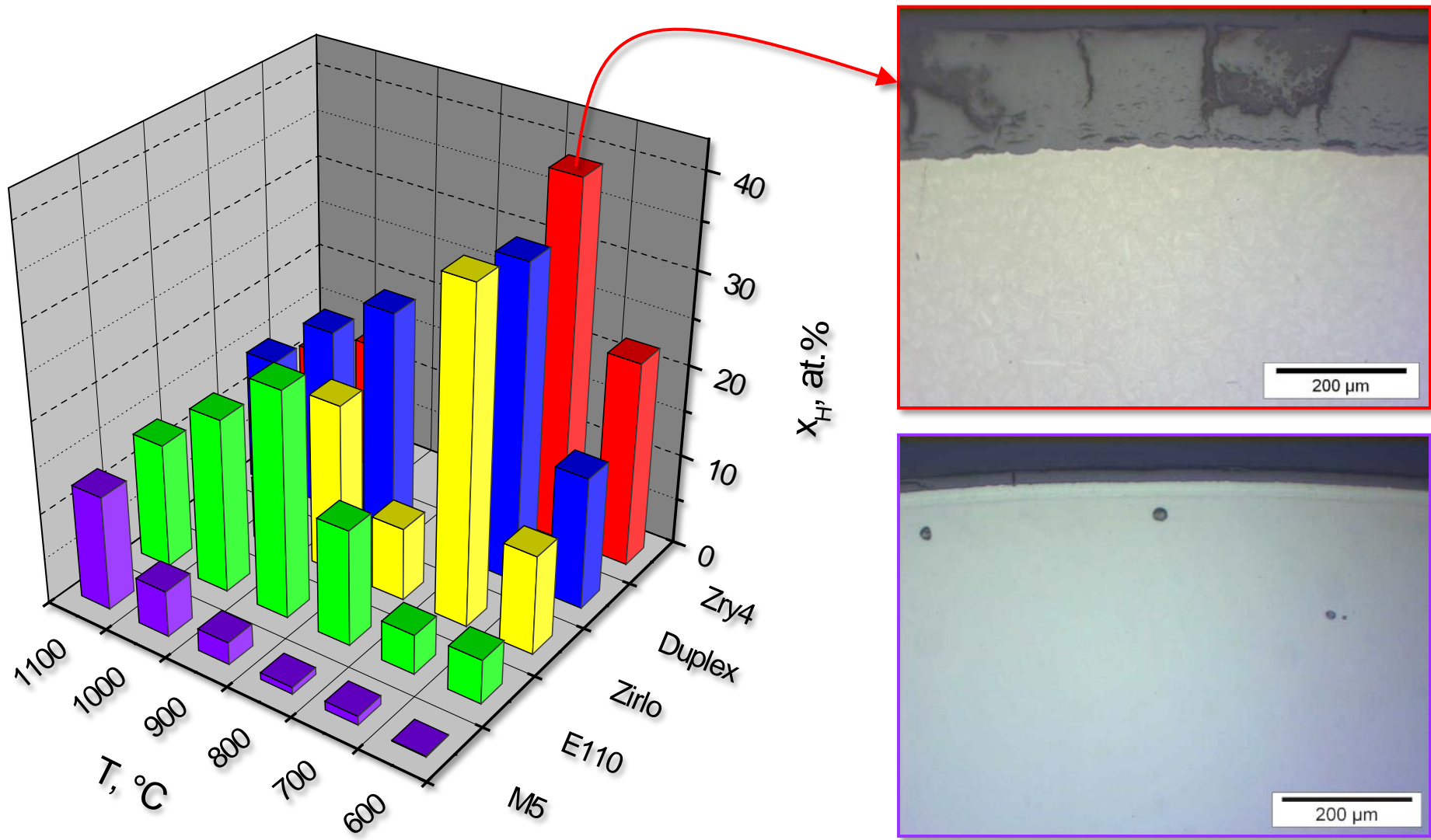
- $Zr + 2H_2O \rightarrow ZrO_2 + 2H_2$
- $H_2(gas) \leftrightarrow 2H(diss)$
- Oxide scale acts as a barrier for uptake and release of hydrogen



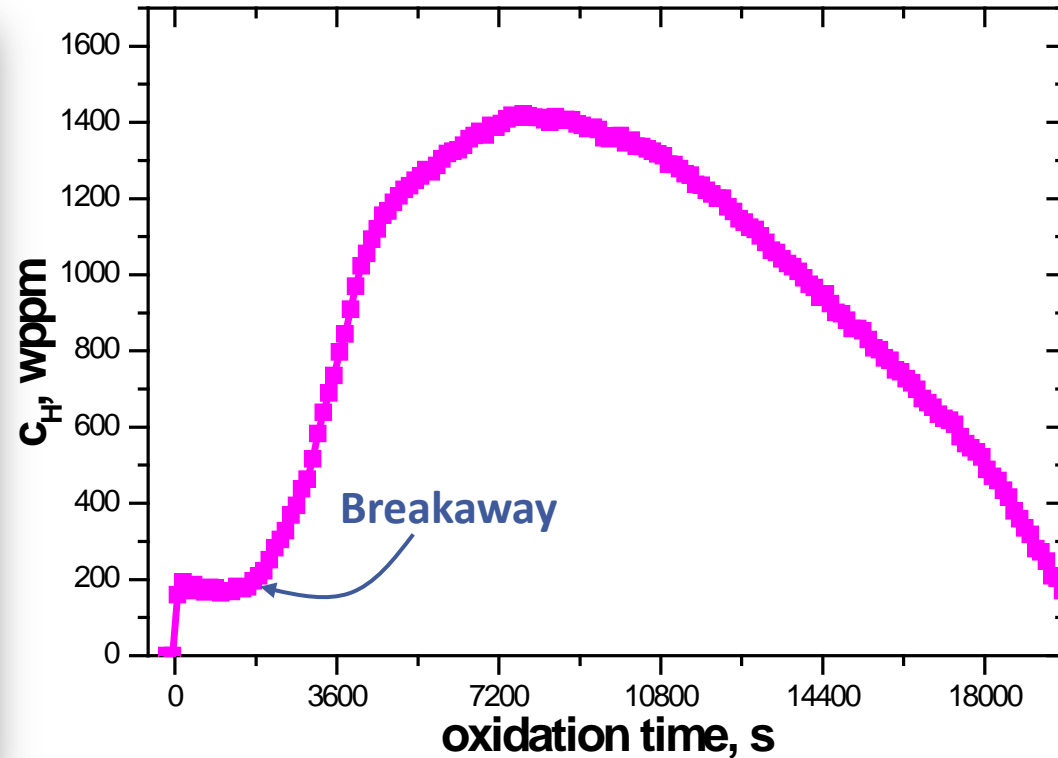
20 min at 1200°C in steam

3h at 1000°C in steam

# Correlation of H absorption and oxide morphology



# In-situ investigation of hydrogen uptake during oxidation of Zry in steam by neutron radiography



Zry-4, 1000°C  
30 g/h steam, 30 l/h argon

- ➡ Rapid initial hydrogen uptake
- ➡ Further strong hydrogen absorption after transition to breakaway

# Oxidation in atmospheres containing nitrogen

... under prototypical conditions, including

- Pre-oxidation in steam/ $O_2$
- Tests in mixed air( $N_2$ )-steam atmospheres

1 hour at 1200 °C in



steam

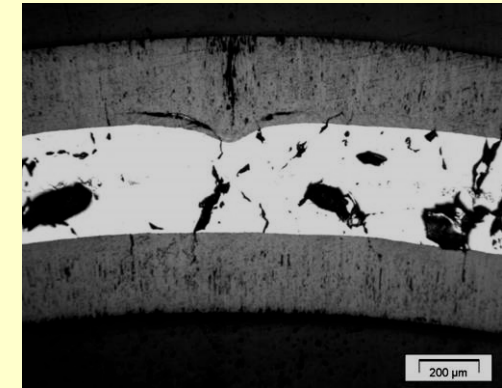


50/50 steam/air

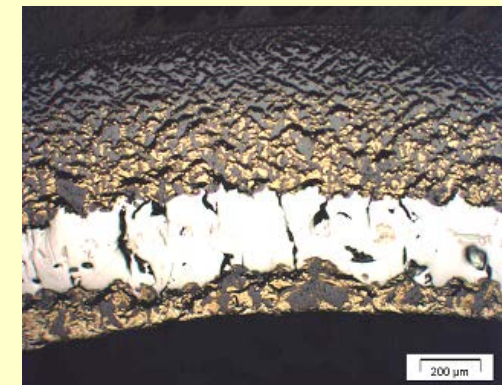


air

➡ Loss of barrier effect against FP product release



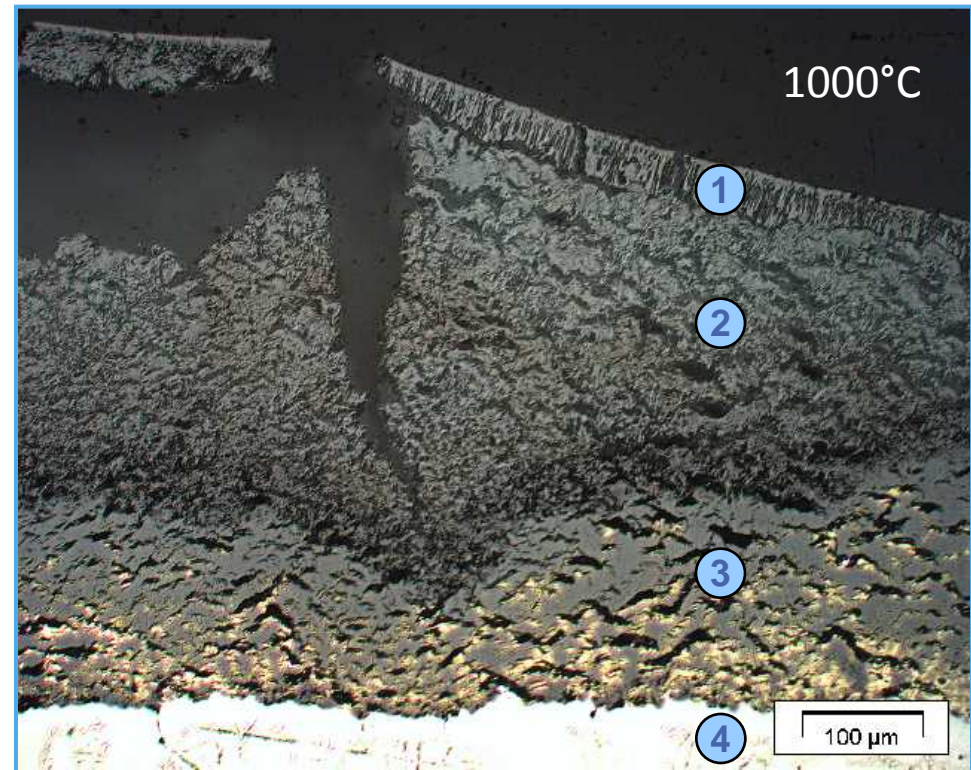
1.5 h steam



1 h steam/ $N_2$  (50/50)

## Mechanism of air oxidation

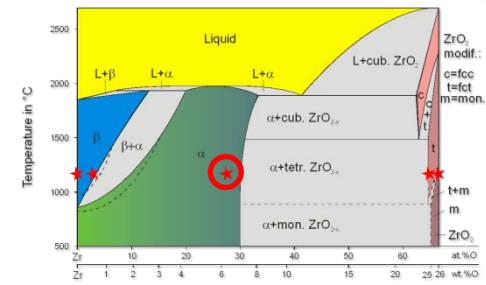
- Diffusion of air through imperfections in the oxide scale to the metal/oxide interface
- Consumption of oxygen
- Remaining nitrogen reacts with zirconium and forms ZrN
- ZrN is re-oxidized by fresh air with continuing reaction associated with a volume increase by 48%
- ➔ Formation of porous and non-protective oxide scales



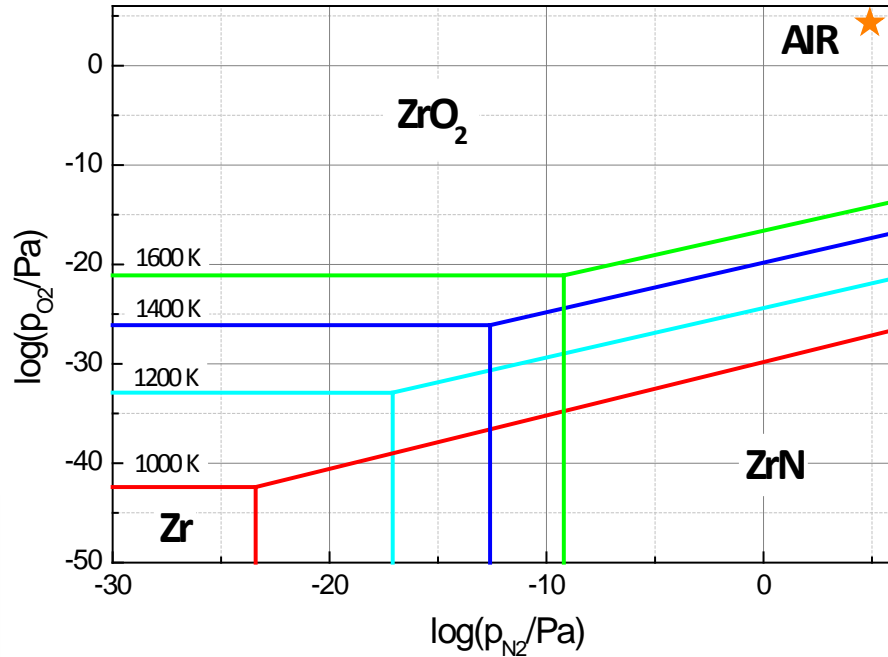
- 1 – initially formed dense oxide  $ZrO_2$
- 2 – porous oxide after oxidation of ZrN
- 3 –  $ZrO_2$  / ZrN mixture
- 4 –  $\alpha$ -Zr(O)



# Mechanism of air oxidation

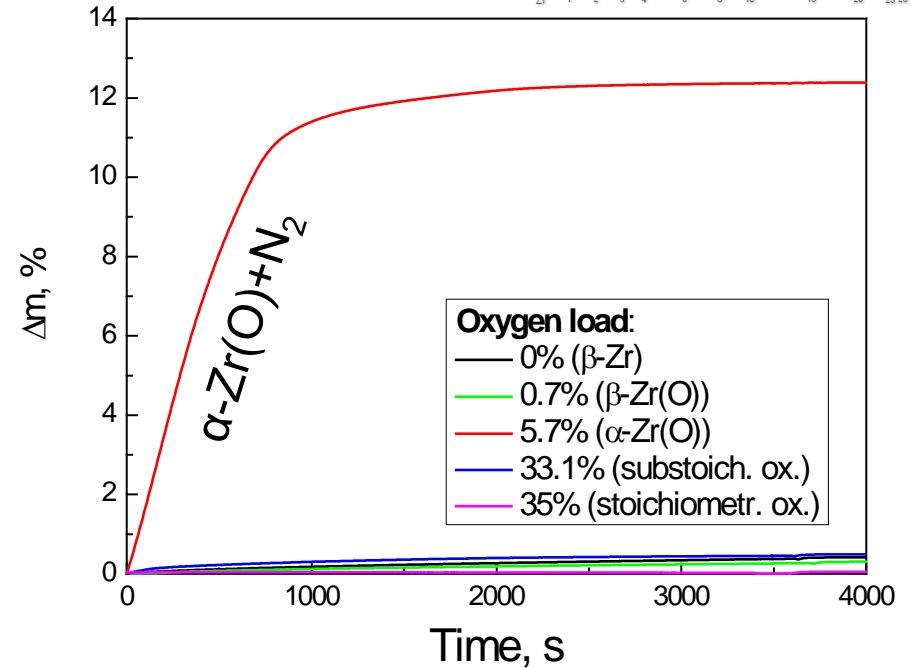


## Thermodynamic view



Stability diagram Zr-O-N

## Kinetic view



Reaction of  $ZrO_x$  with nitrogen ( $1200^\circ\text{C}$ )

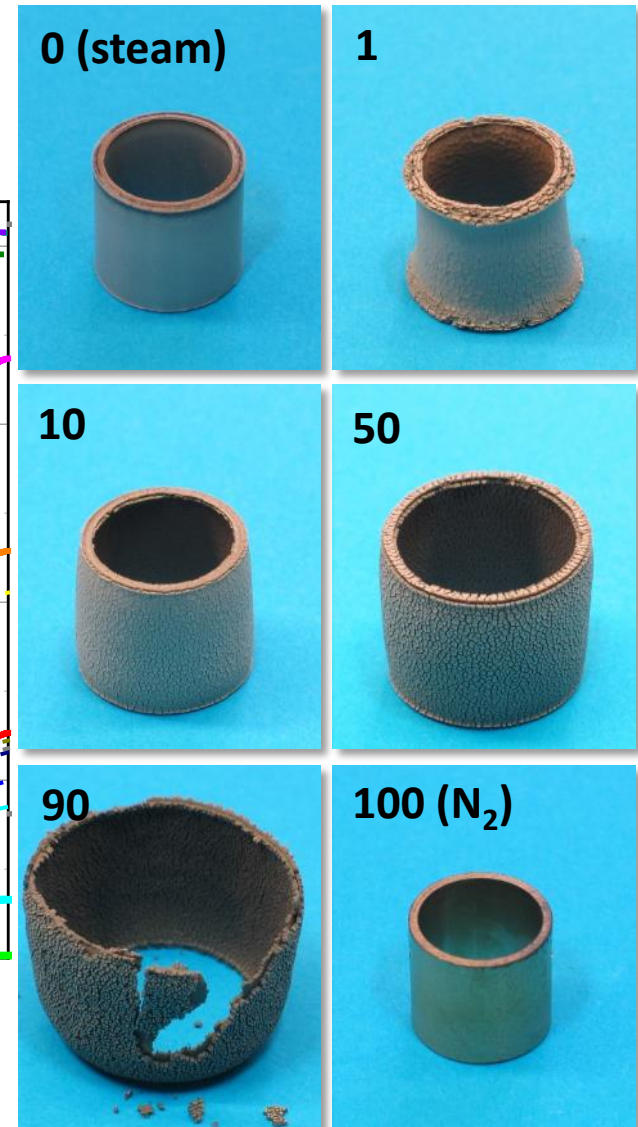
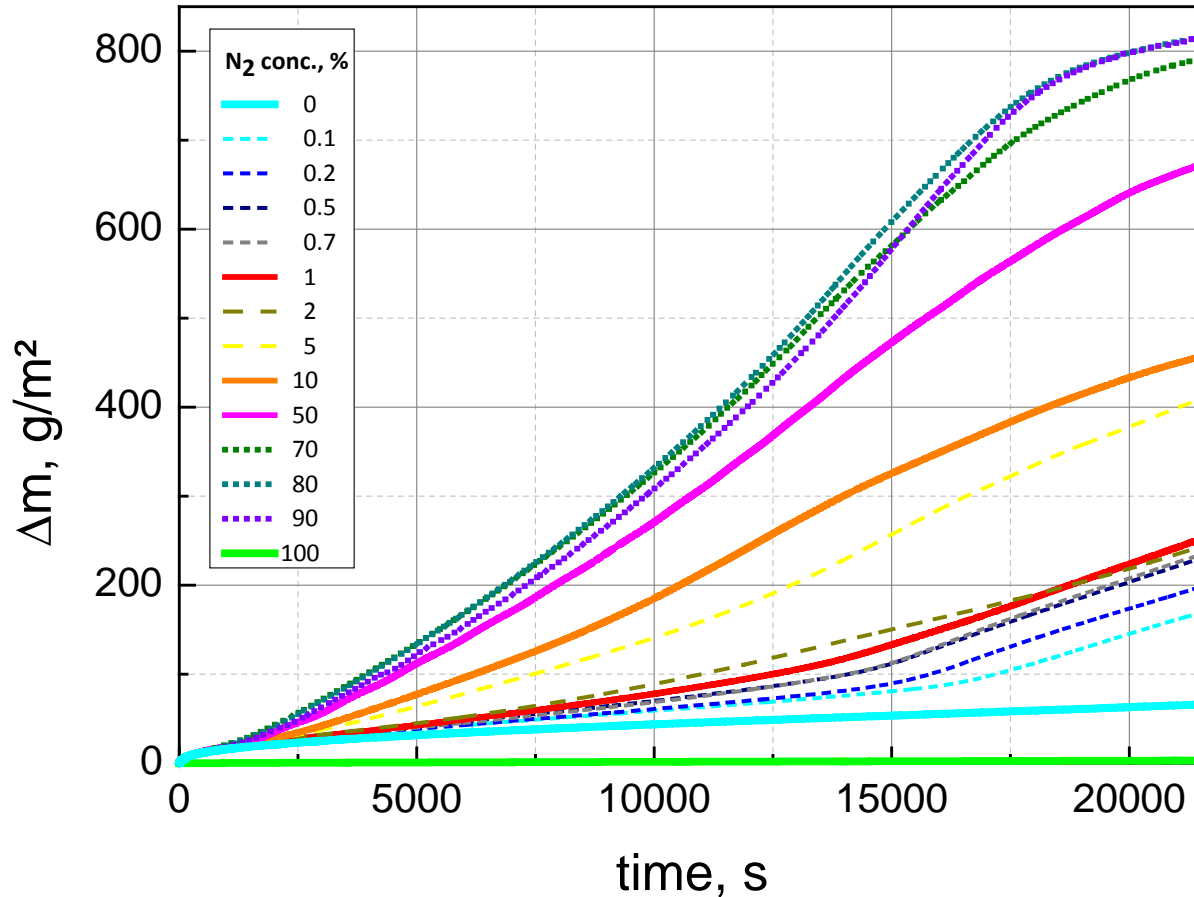
➡ ZrN only stable at very low oxygen partial pressure (activity)

➡ Fast reaction of oxygen-stabilized Zr with nitrogen

➡ Both conditions are present locally at the metal-oxide interface

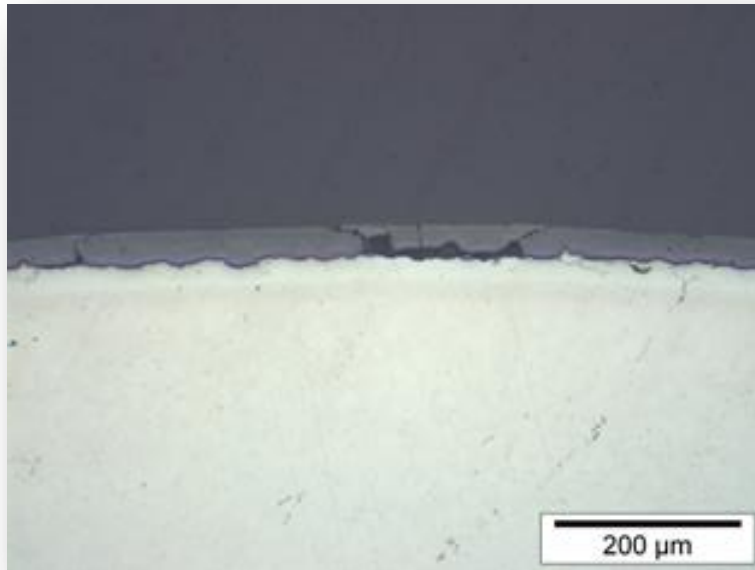
# Oxidation in mixed steam-nitrogen at 800°C

TG results at 800°C, 6 hours

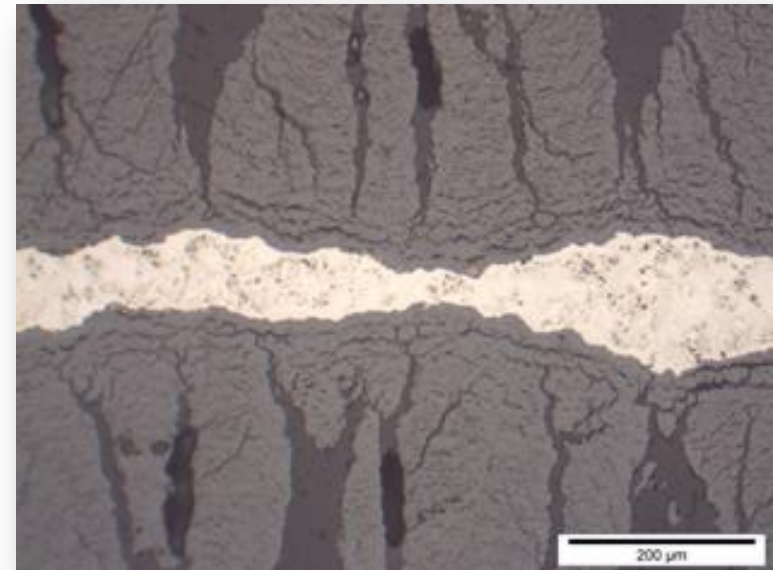


# Oxidation in mixed steam-nitrogen at 800°C

6 hour at 800 °C in steam

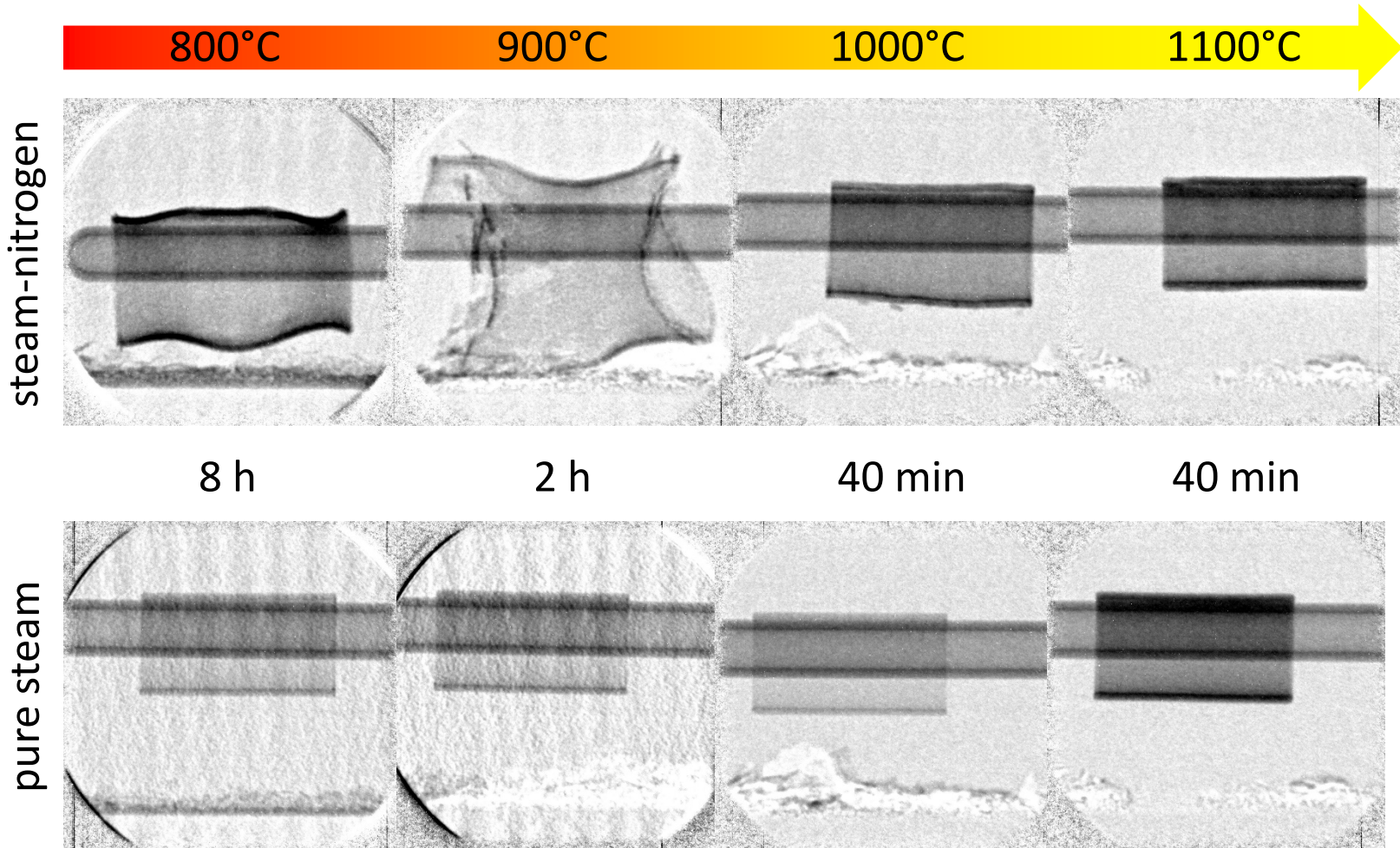


6 hour at 800 °C in 50/50 steam/N<sub>2</sub>



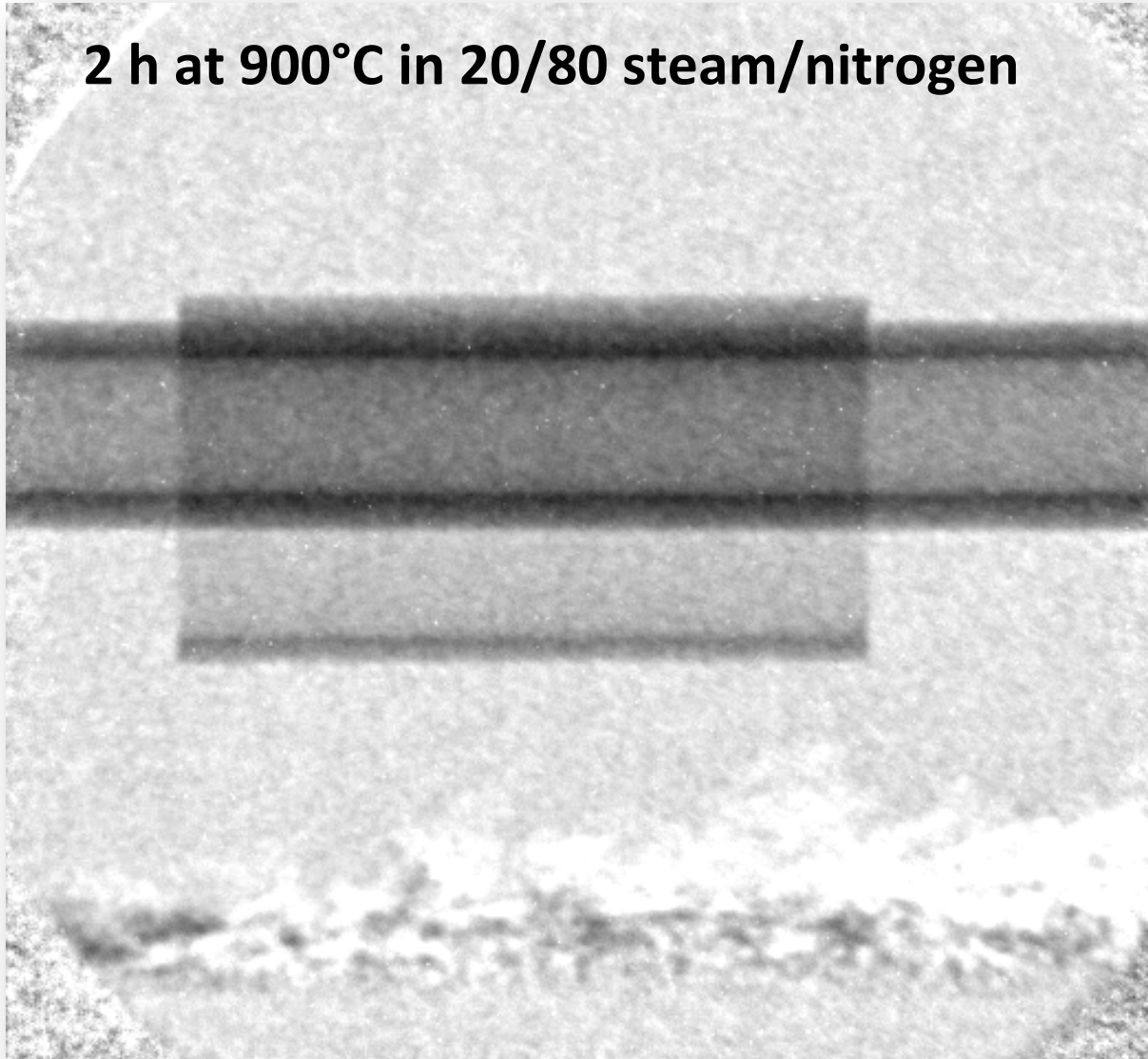
- Strong effect of nitrogen on oxidation and degradation
- Nitrogen acts like a catalyst (NOT like an inert gas)
- Enhanced hydrogen source term by oxidation in mixtures containing nitrogen

# In-situ NR of Zircaloy-4 in steam and steam-nitrogen



# In-situ NR of Zircaloy-4 in steam-nitrogen

**2 h at 900°C in 20/80 steam/nitrogen**

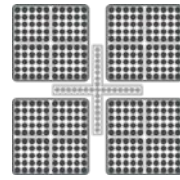


# Control rod behavior

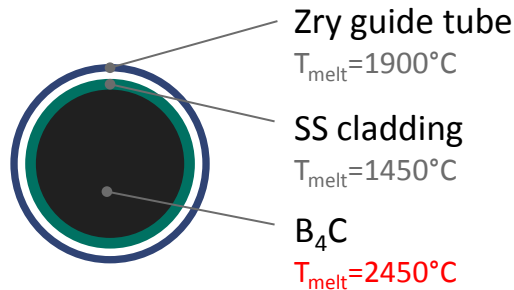
# Absorber materials in LWRs

## Boron carbide

- Used in boiling water reactors (BWR), VVERs, some pressurized water reactors (PWR)
- Control rods (PWR) or cross-shaped blades (BWR)
- Surrounded by stainless steel (cladding, blades) and Zry (guide tubes, canisters)



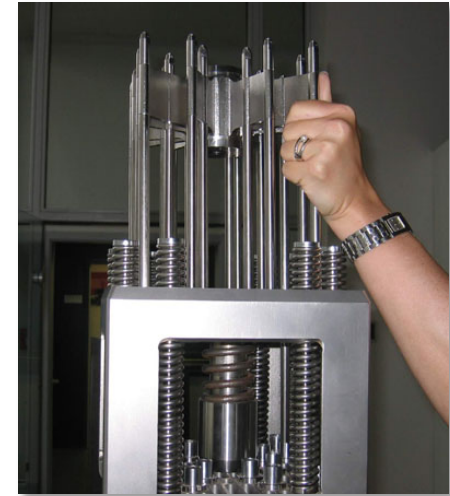
BWR control blade



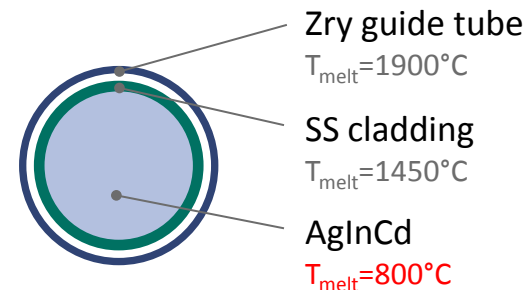
BWR control rod

## AgInCd alloy

- Used in PWRs
- Surrounded by stainless steel cladding and Zry guide tubes
- Rods in Zry guide tubes combined in control rod assemblies



PWR control rod assembly



PWR control rod

## Degradation of B<sub>4</sub>C control rods (1-pellet)

Post-test appearance and axial cross section of B<sub>4</sub>C/SS/Zry specimens after 1 hour isothermal tests at temperatures between 1000 and 1600 °C



1000°C



1200°C



1300°C



1400°C



1500°C

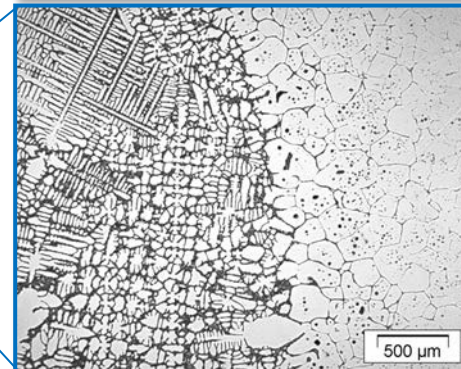
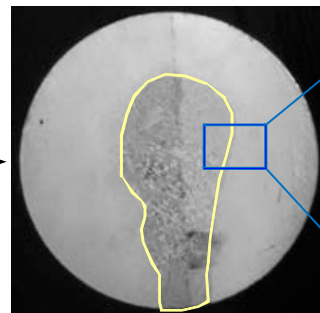
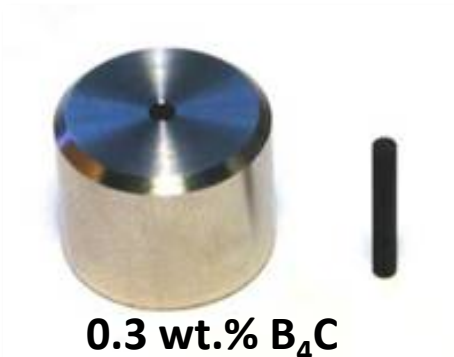
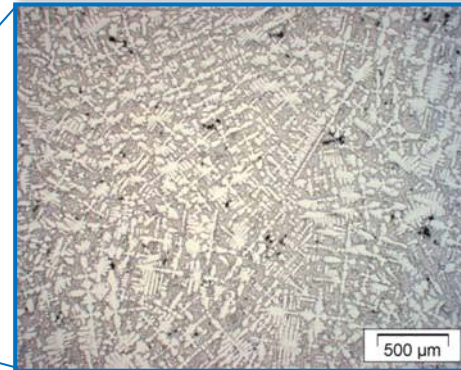
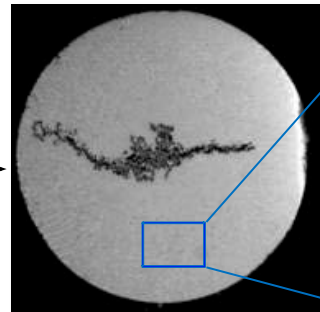
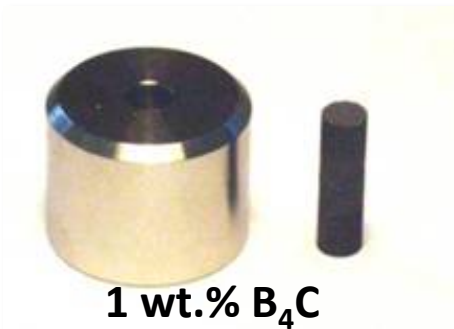
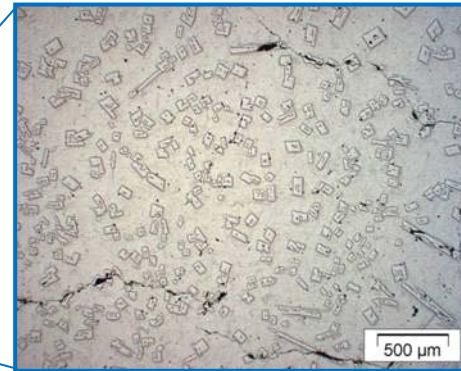
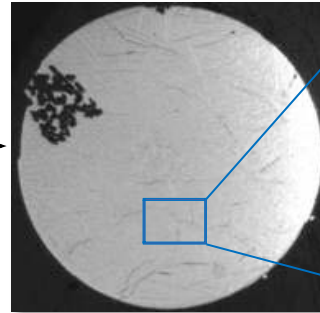
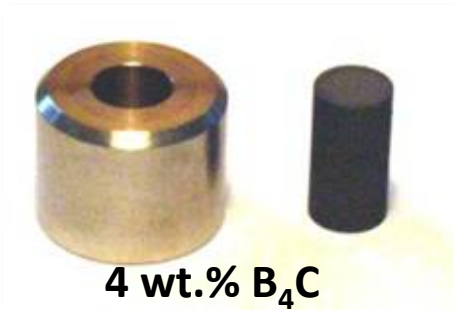


1600°C



# Eutectic interaction of stainless steel with $B_4C$

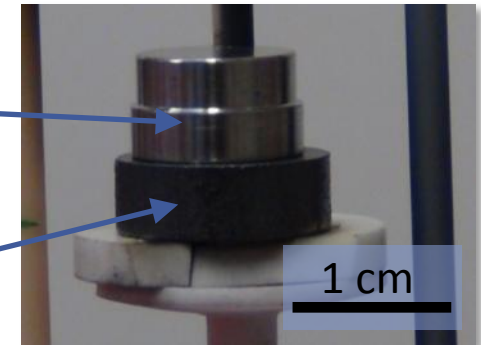
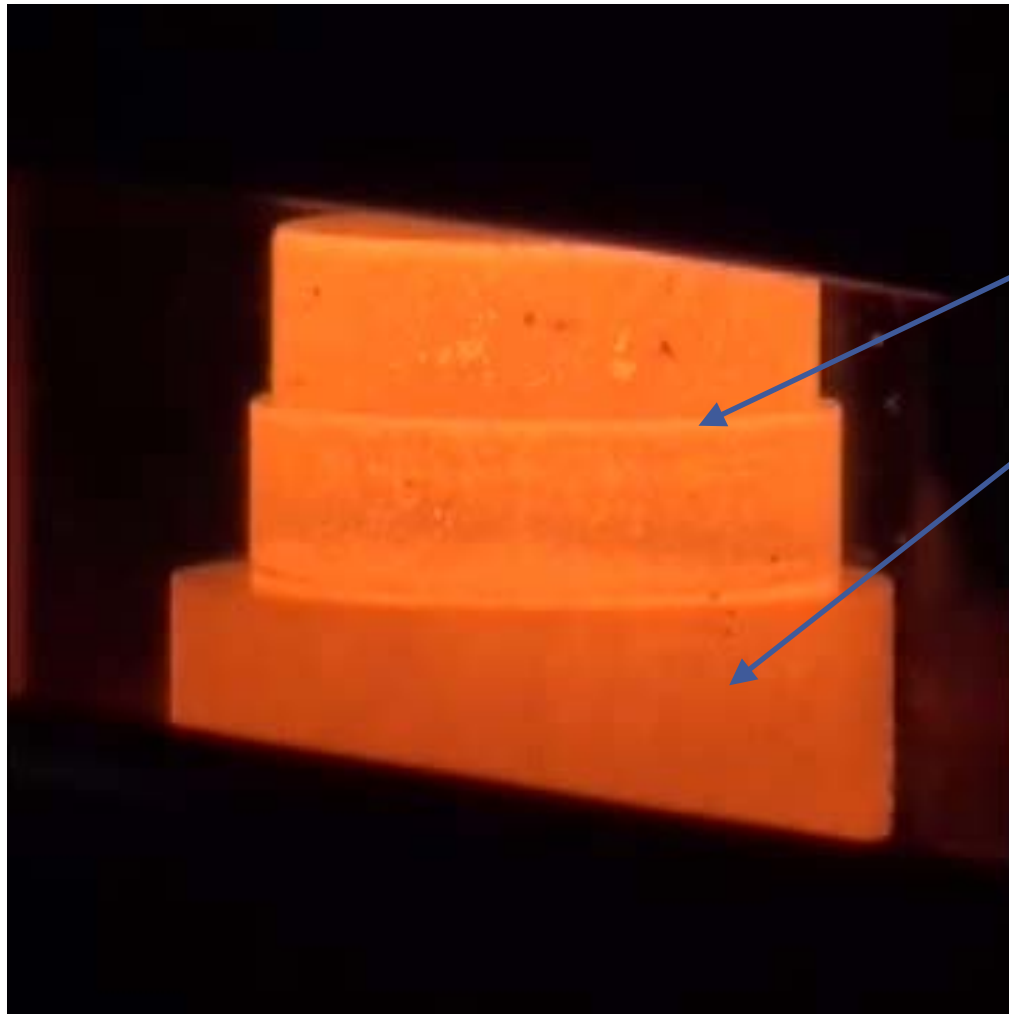
1 h at approx. 1250 °C



Complete  
liquefaction  
of stainless  
steel

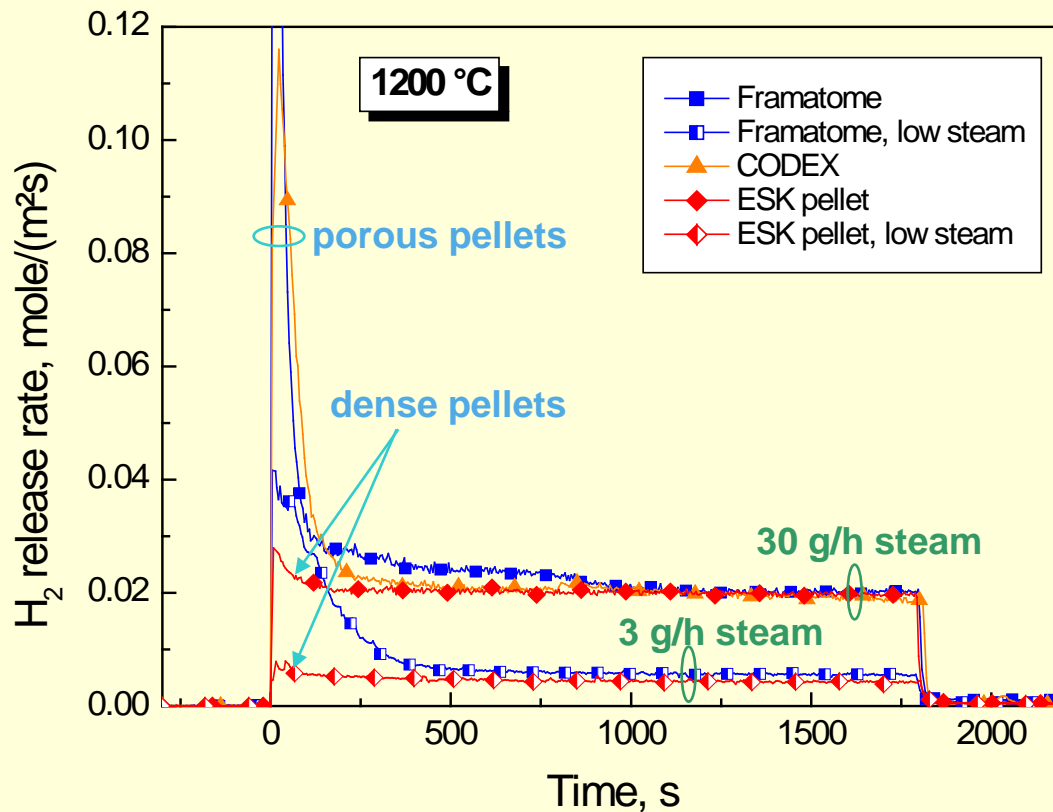
1/3 of SS  
liquefied

# Eutectic interaction of stainless steel with $B_4C$



- ➡ Rapid and complete melting of SS at  $1250^\circ C$  starting at  $B_4C/SS$  interface

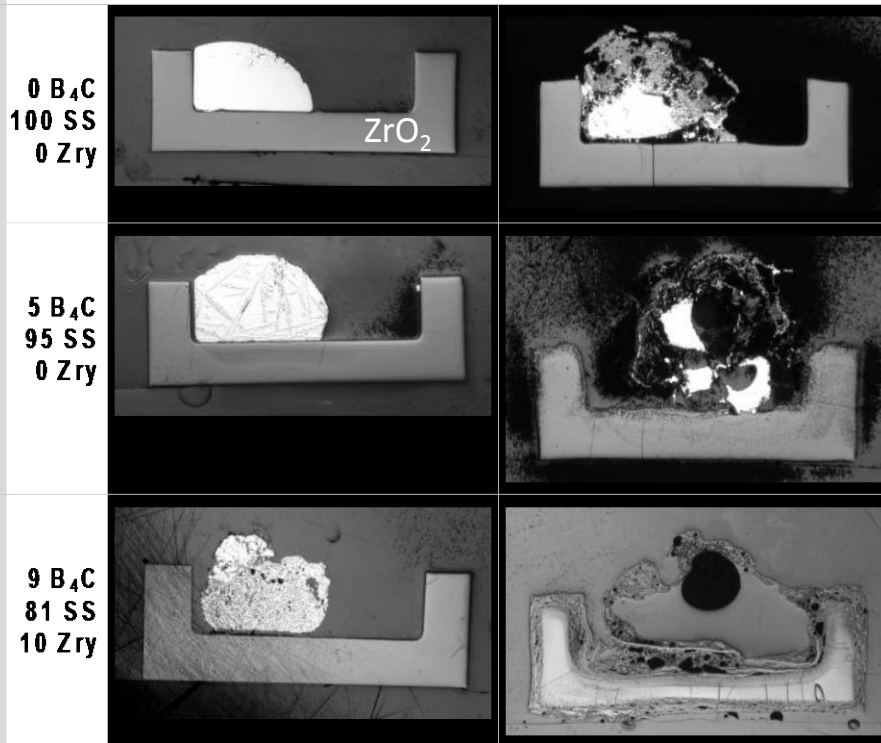
# Oxidation kinetics of B<sub>4</sub>C in steam



Strongly dependant on B<sub>4</sub>C structure and thermo-hydraulic boundary conditions like pressure and flow rate

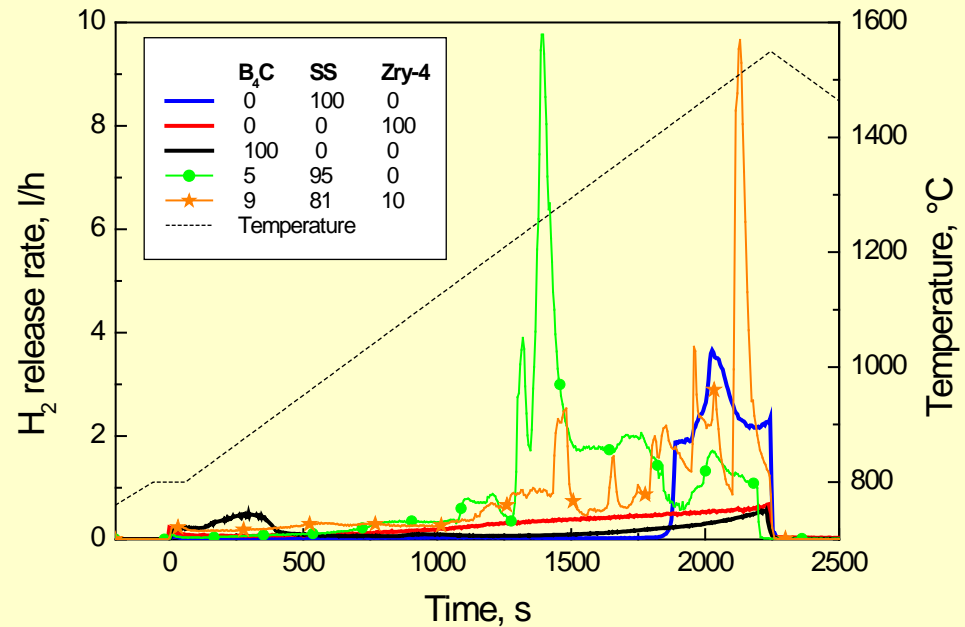
# Oxidation of B<sub>4</sub>C absorber melts

Transient oxidation of B<sub>4</sub>C/SS/Zry-4 absorber melts  
in steam between 800 and 1550 °C



before oxidation

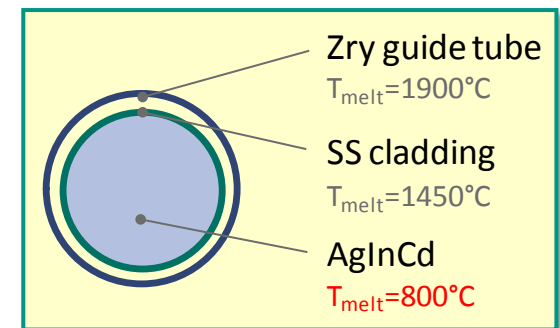
after oxidation



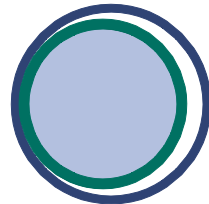
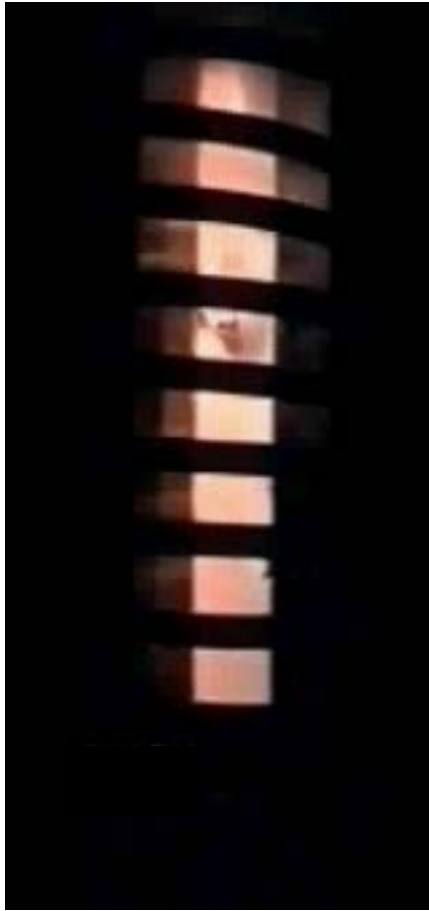
Oxidation rate during reaction of absorber melts and pure CR components in steam

## Failure of AgInCd absorber rod

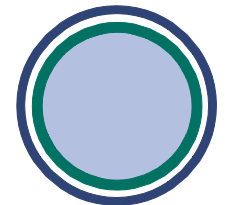
- Ag-In-Cd control rods fail at temperatures above 1200°C due to the eutectic interaction between SS and Zry-4
- Failure is very stochastic (from local to explosive) with the tendency to higher temperatures for symmetric samples and specimens with inner oxidation
- No ballooning of the SS cladding tube was observed before rupture
- Burst release of cadmium vapour is followed by continuous release of indium and silver aerosols and absorber melt



# Different failure types of AgInCd absorber rod



SIC-02 (asym. rod)  
Local failure at 1230°C



SIC-05 (symmetric rod)  
Global failure at 1350°C

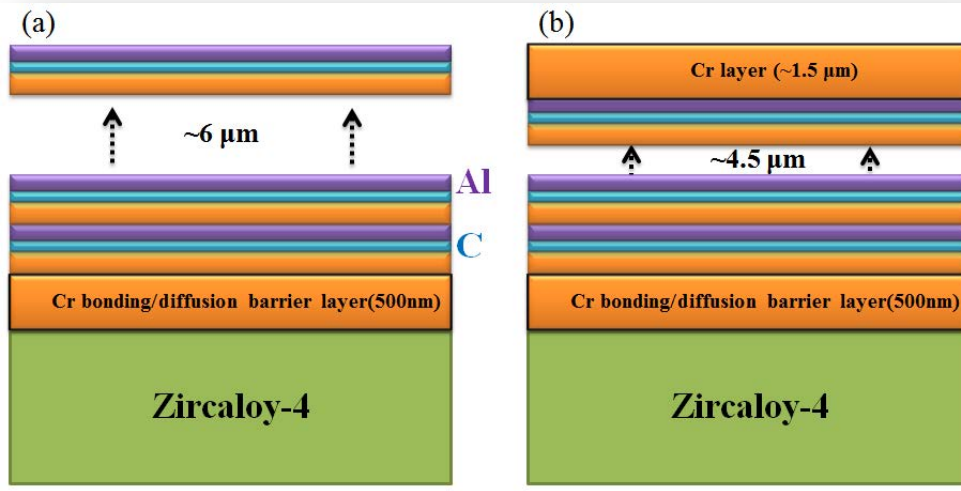
# ATF cladding

## KIT activities on ATF

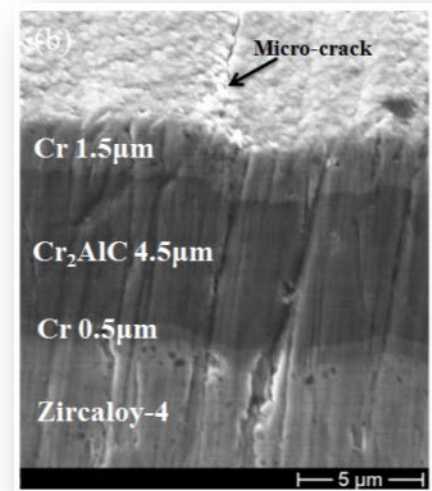
- QUENCH bundle tests with FeCrAl cladding in cooperation with ORNL
- Single-rod oxidation and quench tests with Cr-coated Zr alloy
- Ultra-high temperature oxidation tests with SiC<sub>f</sub>-SiC
- Development of MAX phase coatings for Zr alloys
  
- Participation in various international collaborations on ATF
  - EC IL TROVATORE
  - IAEA ACTOF
  - OECD NEA EGATFL, TOPATF, and QUENCH-ATF (under discussion)
  - Westinghouse CARAT



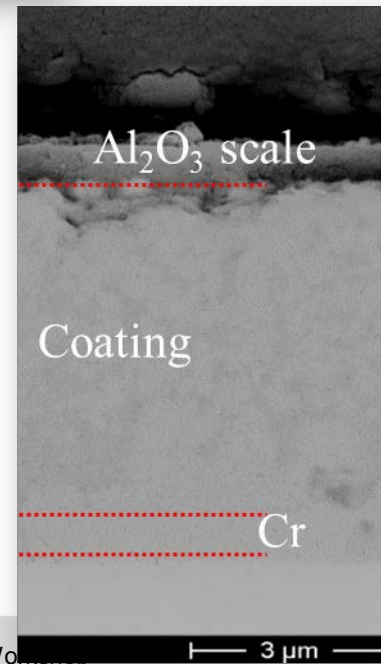
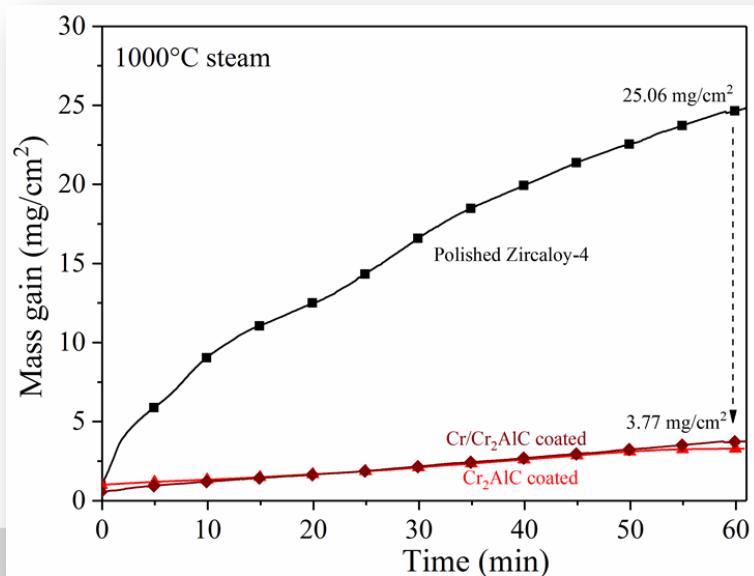
# MAX-phase coated cladding alloys ( $\text{Cr}_2\text{AlC}$ )



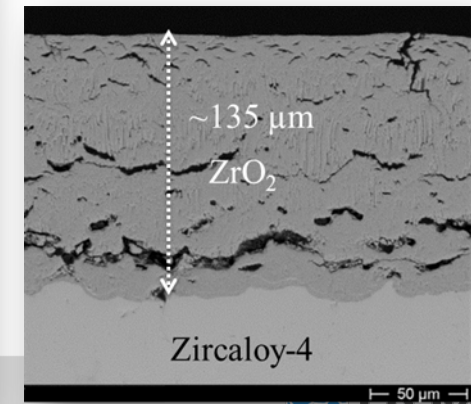
Annealing



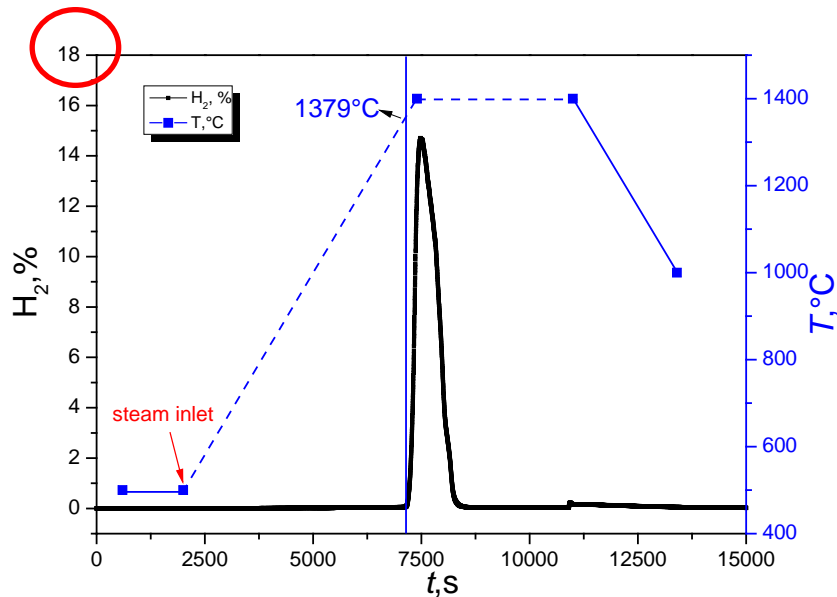
## Magnetron sputtered Cr/C/Al nano layers



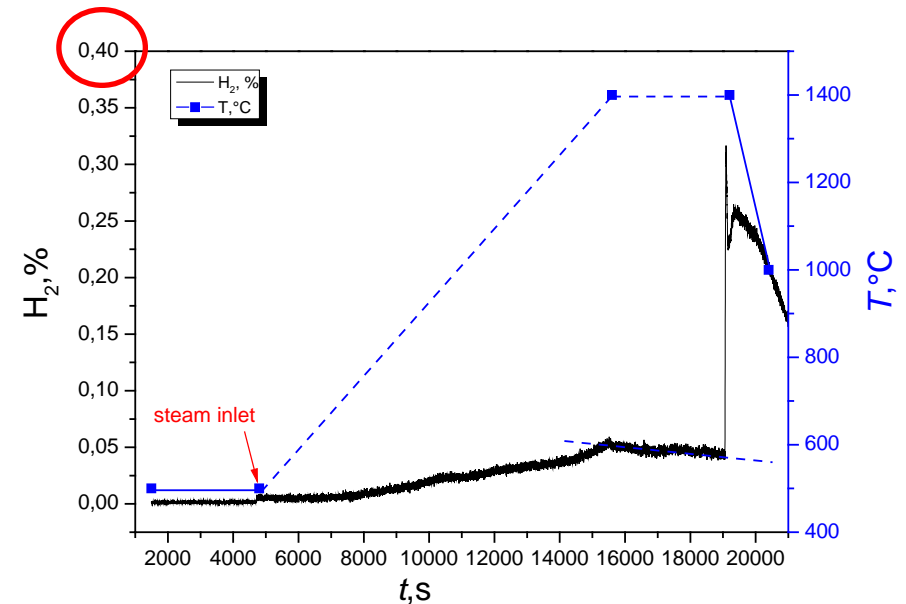
## Pure MAX phase



# Oxidation of Kanthal-APM (FeCrAl) in steam



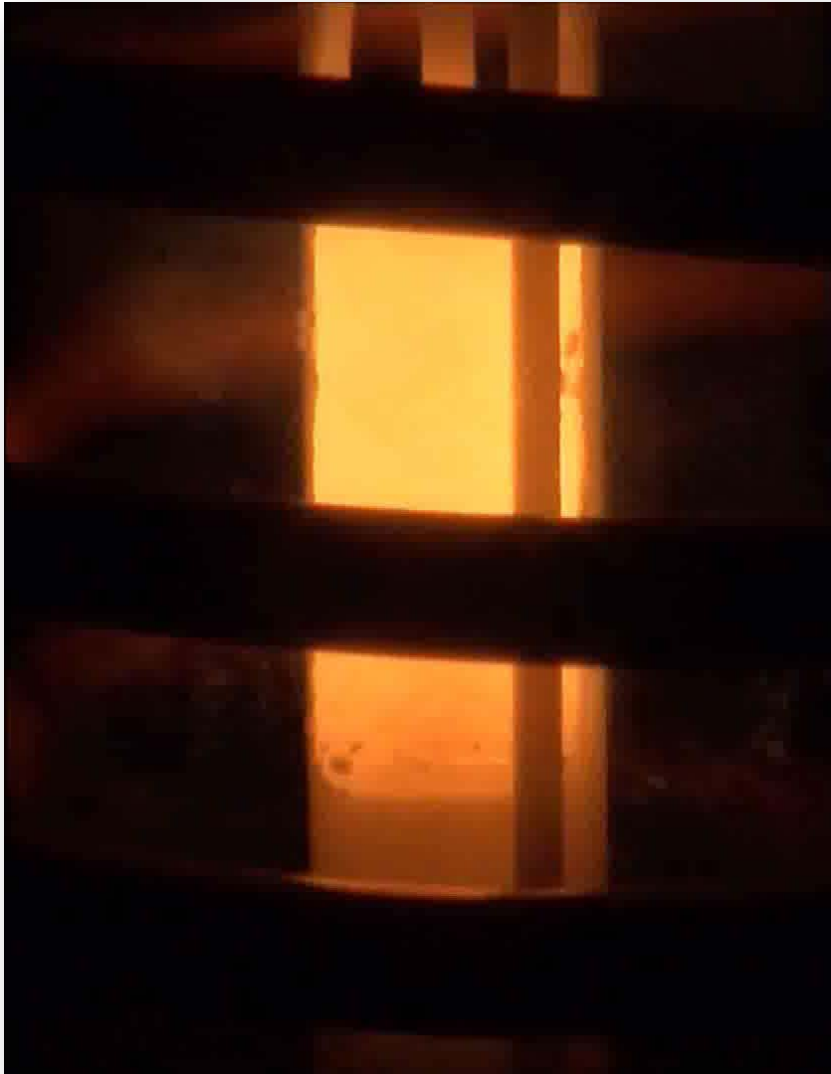
Heating with 10 K/min to 1400°C and subsequent isothermal annealing for 1 h in steam



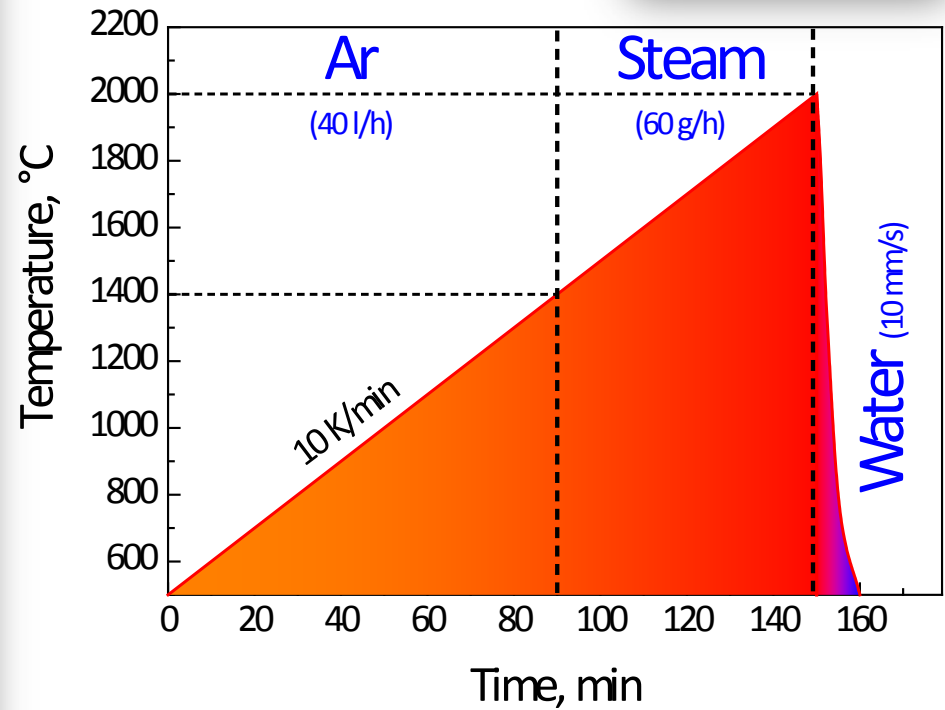
Heating with 5 K/min to 1400°C and subsequent isothermal annealing for 1 h in steam

- ➡ Formation of a protective alumina scale during slow heatup or pre-oxidation at lower temperatures
- ➡ Otherwise, rapid and complete oxidation of the FeCrAl alloy

# Steam oxidation of SiC and quench from 2000°C



Post-test appearance of SiC-SiC sample



Test conduct

- Zirconium oxidation at high temperatures is a source of significant release of hydrogen and heat affecting nuclear accident progression.
- Oxidation is not always of parabolic kinetics and may be strongly dependent on experimental boundary conditions.
- Eutectic interactions may lead to melt formation far below the melting points of the individual materials. These melts may slowly relocate and severely oxidize.
- ATF claddings could strongly decrease the risk of temperature escalation and hydrogen detonation during BDB accidents as well as significantly increase the coping time for AMMs.
- Most experimental results were used to improve models, especially in cooperation with IBRAE, GRS, and IRSN as well as internally by H. Steiner and M. Große.

# Acknowledgement

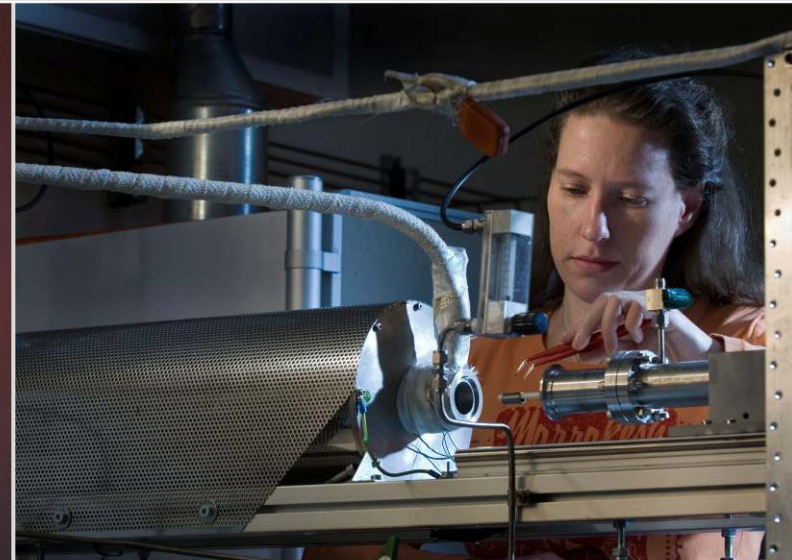
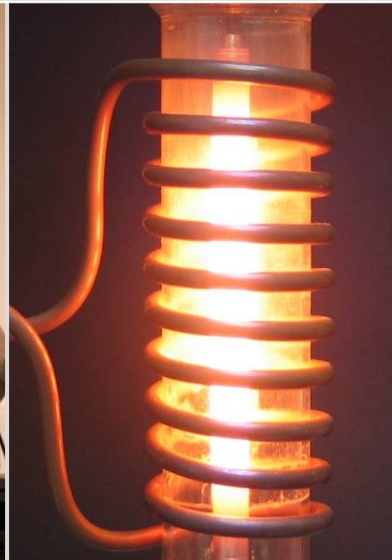
- The QUENCH group at KIT for their excellent work and support
- Program NUSAFE at KIT
- Various projects in the 3<sup>rd</sup> to 7<sup>th</sup> EU Framework Programs for Research and Innovation
- National and international partners
  
- You, for your kind attention

# 25 years QUENCH program Highlights of separate-effects tests

M. Steinbrück, J. Stuckert, M. Große et al.

*25th International QUENCH Workshop, Karlsruhe, 22-24 October 2019*

Institute for Applied Materials IAM-AWP & Program NUSAFE



# Key papers: Oxidation of Zr alloys and hydrogen behavior

Grosse, M., Pulvermacher, S., Steinbrück, M., Schillinger, B.

In-situ neutron radiography investigations of the reaction of Zircaloy-4 in steam, nitrogen/steam and air/steam atmospheres (2018) *Physica B: Condensed Matter*, 551, pp. 244-248. Cited 2 times.

DOI: 10.1016/j.physb.2018.03.030

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In situ investigations of the hydrogen uptake of zirconium alloys during steam oxidation (2018) *ASTM Special Technical Publication, STP 1597*, pp. 1114-1135. Cited 3 times.

DOI: 10.1520/STP159720160041

Steinbrück, M., da Silva, F.O., Grosse, M.

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DOI: 10.1016/j.jnucmat.2017.04.034

Stuckert, J., Hózer, Z., Kiselev, A., Steinbrück, M.

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DOI: 10.1016/j.anucene.2015.12.034

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DOI: 10.1007/s11085-015-9572-1

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# Key papers: Oxidation of Zr alloys and hydrogen behavior

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DOI: 10.1016/j.nucengdes.2014.06.022

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DOI: 10.1016/j.jnucmat.2013.12.024

Große, M., Steinbrück, M., Stuckert, J., Kastner, A., Schillinger, B.

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DOI: 10.1007/s10853-011-5553-1

Steinbrück, M., Vér, N., Grosse, M.

Oxidation of advanced zirconium cladding alloys in steam at temperatures in the range of 600-1200 °C (2011) Oxidation of Metals, 76 (3-4), pp. 215-232. Cited 58 times.

DOI: 10.1007/s11085-011-9249-3

Steinbrück, M., Böttcher, M.

Air oxidation of Zircaloy-4, M5<sup>®</sup> and ZIRLO<sup>™</sup> cladding alloys at high temperatures

(2011) Journal of Nuclear Materials, 414 (2), pp. 276-285. Cited 72 times.

DOI: 10.1016/j.jnucmat.2011.04.012

Steinbrück, M., Große, M., Sepold, L., Stuckert, J.

Synopsis and outcome of the QUENCH experimental program

(2010) Nuclear Engineering and Design, 240 (7), pp. 1714-1727. Cited 84 times.

DOI: 10.1016/j.nucengdes.2010.03.021



# Key papers: Oxidation of Zr alloys and hydrogen behavior

Grosse, M.

Comparison of the high-temperature steam oxidation kinetics of advanced cladding materials  
(2010) Nuclear Technology, 170 (1), pp. 272-279. Cited 20 times.  
DOI: 10.13182/NT10-A9464

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DOI: 10.1016/j.jnucmat.2009.04.018

Große, M., Lehmann, E., Steinbrück, M., Kühne, G., Stuckert, J.

Influence of oxide layer morphology on hydrogen concentration in tin and niobium containing zirconium alloys after high temperature steam oxidation  
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(2008) Oxidation of Metals, 70 (5-6), pp. 317-329. Cited 35 times.  
DOI: 10.1007/s11085-008-9124-z

Grosse, M., Steinbrück, M., Lehmann, E., Vontobel, P.

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DOI: 10.1007/s11085-008-9113-2

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## Key papers: Control rod degradation

Kurata, M., Barrachin, M., Haste, T., Steinbrueck, M.

Phenomenology of BWR fuel assembly degradation

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DOI: 10.1016/j.anucene.2016.11.039

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Influence of boron carbide on core degradation during severe accidents in LWRs

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DOI: 10.1016/j.anucene.2013.09.027

Steinbrück, M.

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(2010) Journal of Nuclear Materials, 400 (2), pp. 138-150. Cited 32 times.

DOI: 10.1016/j.jnucmat.2010.02.022

Dubourg, R., Austregesilo, H., Bals, C., Barrachin, M., Birchley, J., Haste, T., Lamy, J.S., Lind, T., Maliverney, B., Marchetto, C., Pinter, A., Steinbrück, M., Stuckert, J., Trambauer, K., Vimi, A.

Understanding the behaviour of absorber elements in silver-indium-cadmium control rods during PWR severe accident sequences

(2010) Progress in Nuclear Energy, 52 (1), pp. 97-108. Cited 19 times.

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DOI: 10.1016/j.jnucmat.2004.09.022

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Steinbrueck, M., Angelici Avincola, V., Markel, I.J., Stegmaier, U., Gerhards, U., Seifert, H.J.

Oxidation of SiC f -SiC CMC cladding tubes for GFR application in impure helium atmosphere and materials interactions with tantalum liner at high temperatures up to 1600°C

(2019) Journal of Nuclear Materials, 517, pp. 337-348.

DOI: 10.1016/j.jnucmat.2019.02.024

Tang, C., Jianu, A., Steinbrueck, M., Grosse, M., Weisenburger, A., Seifert, H.J.

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Tang, C., Steinbrueck, M., Stueber, M., Grosse, M., Yu, X., Ulrich, S., Seifert, H.J.

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DOI: 10.1016/j.corsci.2018.02.035

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Protective coatings on zirconium-based alloys as accident-Tolerant fuel (ATF) claddings

(2017) Corrosion Reviews, 35 (3), pp. 141-165. Cited 36 times.

DOI: 10.1515/correv-2017-0010

Tang, C., Steinbrück, M., Große, M., Bergfeldt, T., Seifert, H.J.

Oxidation behavior of Ti2AlC in the temperature range of 1400 °C–1600 °C in steam

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DOI: 10.1016/j.jnucmat.2017.03.016

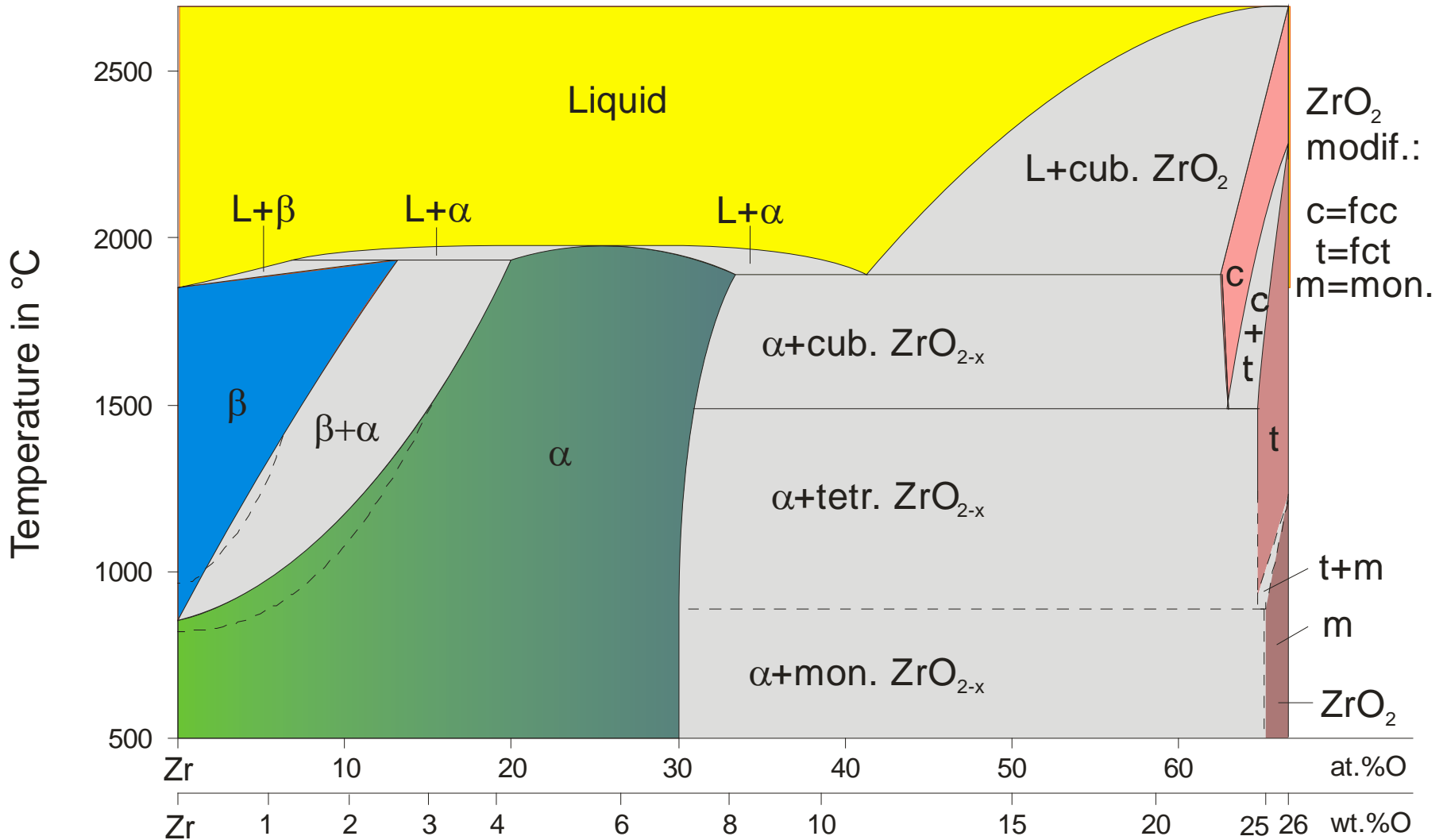
Avincola, V.A., Grosse, M., Stegmaier, U., Steinbrueck, M., Seifert, H.J.

Oxidation at high temperatures in steam atmosphere and quench of silicon carbide composites for nuclear application

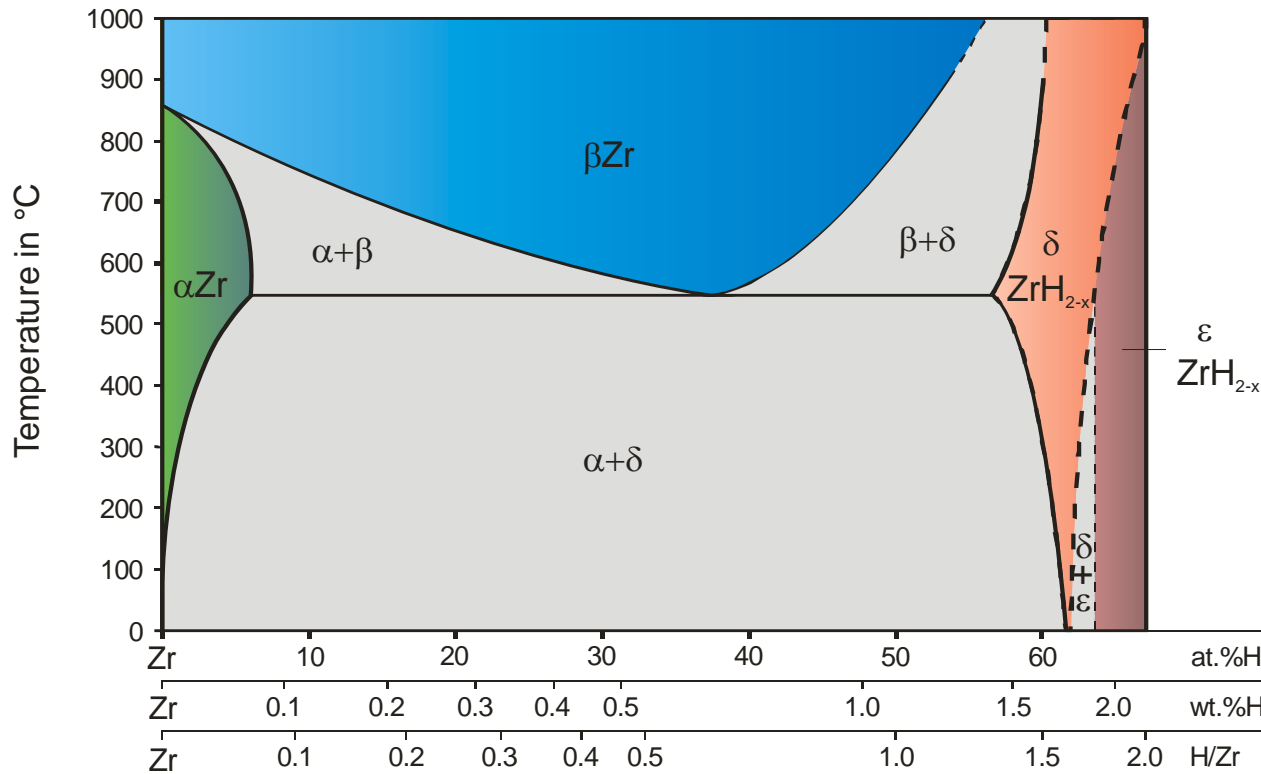
(2015) Nuclear Engineering and Design, 295, pp. 468-478. Cited 20 times.

DOI: 10.1016/j.nucengdes.2015.10.002

# Phase diagram Zr - O



# Phase diagram Zr - H



**Sieverts' law:**

$$\frac{H}{Zr} = k_S \cdot \sqrt{p_{H_2}}$$

with

$$k_S = A \cdot e^{\frac{-B}{RT}}$$