FAST/FLASH: A COMBINATION OF FAST/SPS AND FLASH SINTERING

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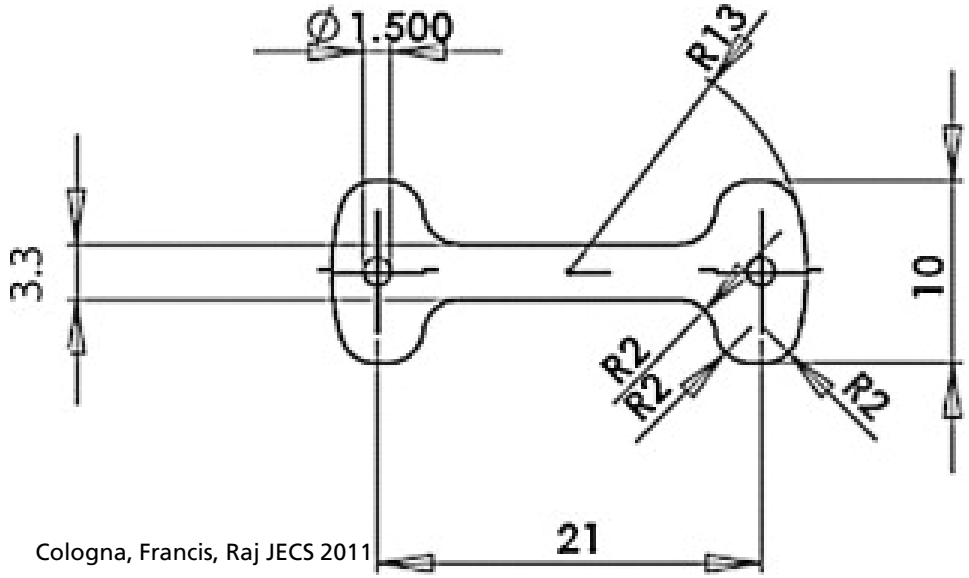
OUTLINE

- Introduction
- Experimental setup
- Results
- Discussion & conclusion

Introduction – fast sintering processes

- Flash Sintering
  - Ultra fast process
  - Air sintering process
  - $\vec{E}$ <2000 V/cm
  - Pre-shaped oxide ceramics
  - One sample geometry



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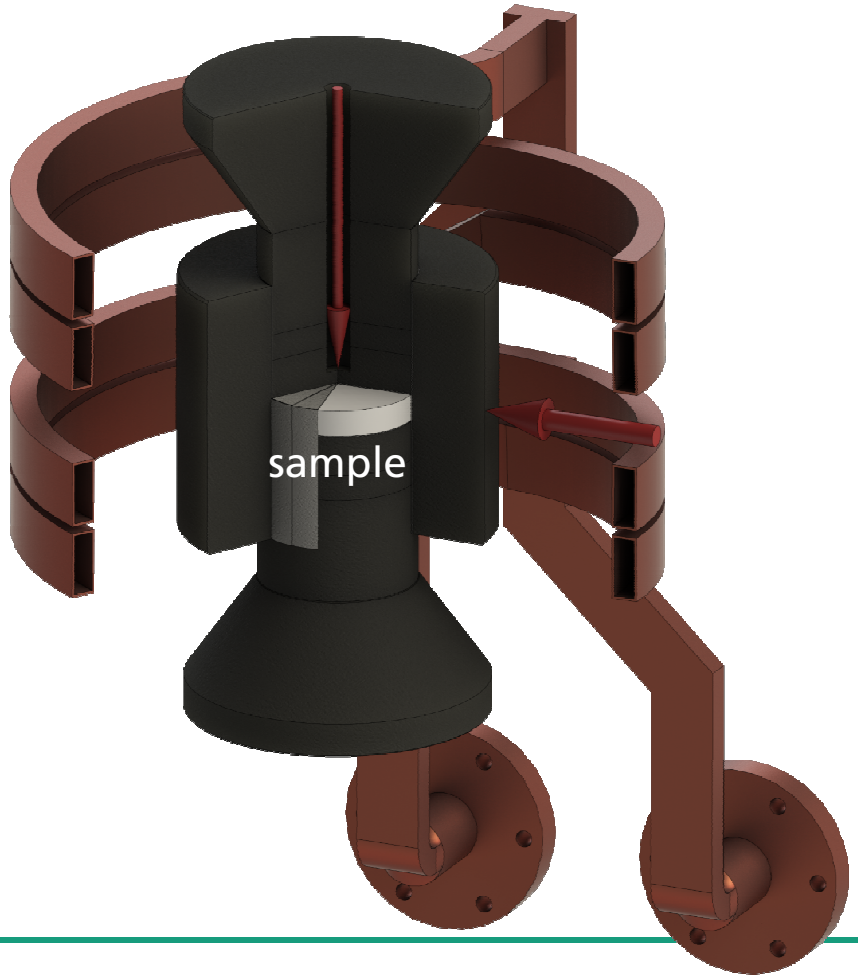
Introduction – fast sintering processes



- Hybrid/FAST
  - Fast sintering
  - Pressure-assisited
  - $\vec{E}$ <10 V/cm
  - Various powder materials
  - Various large geometries
  - Vacuum or inert gas process

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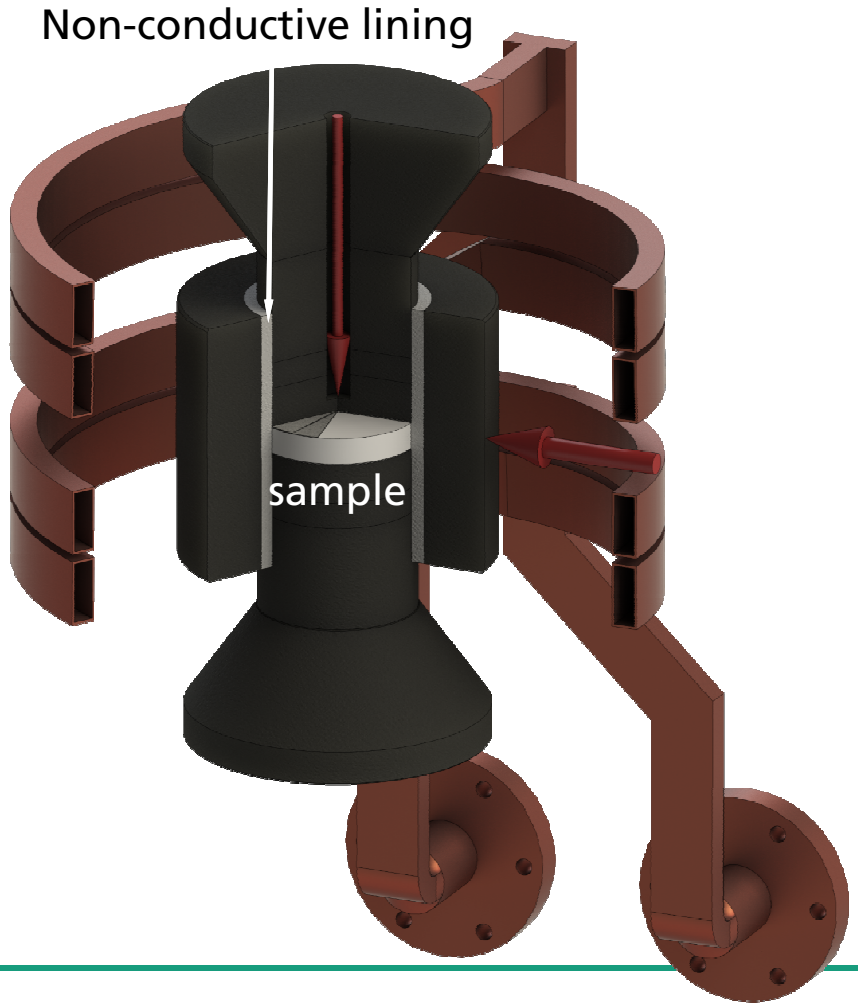
Introduction – fast sintering processes



Hybrid/FAST → FAST/Flash

- Additional voltage support (180V, 4kA)
- Application of $\vec{E}$ < 400V/cm

→ Ultra rapid, pressure-assisted sintering technology starting from various powder materials at electrical field < 400 V/cm for the production of industry-relevant components with new/superior properties

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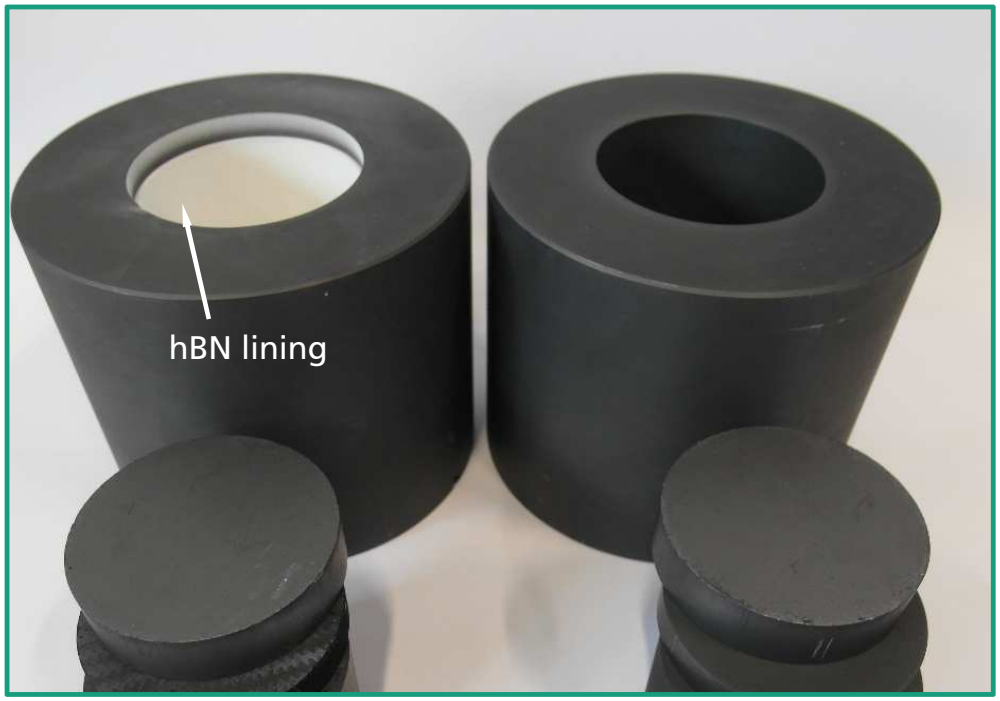
Introduction – fast sintering processes

- HHPD25 (IKTS)
  - FAST/SPS + Induction + Flash@<180V
  - Resistance measurements ∅20mm
  - Sample diameter ∅30-100mm

FAST/Flash tool Standard tool



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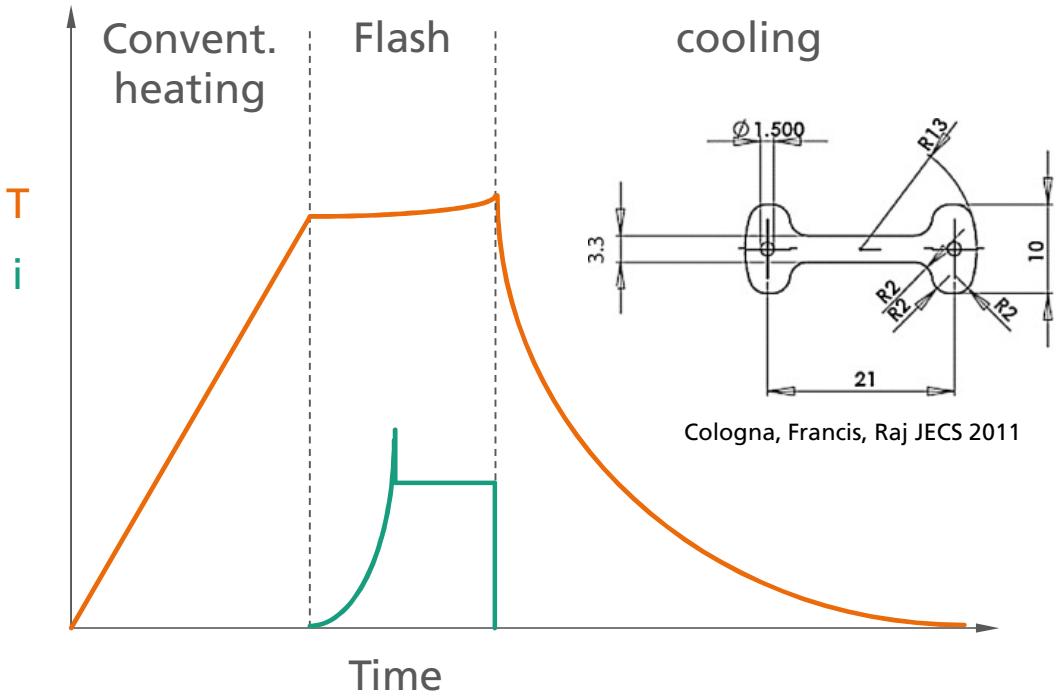
Experimental setup

- Flash Sintering vs. FAST/Flash



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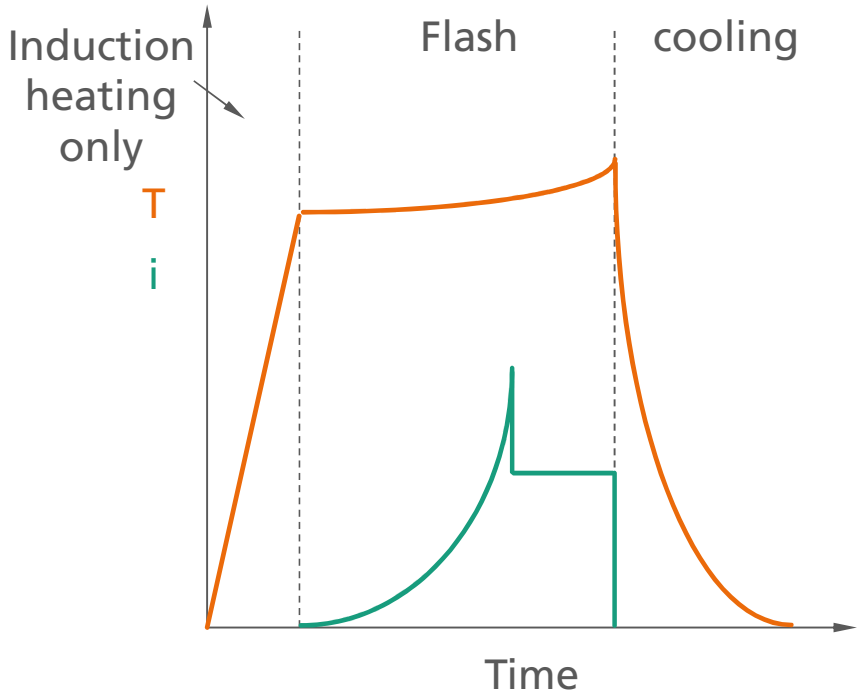

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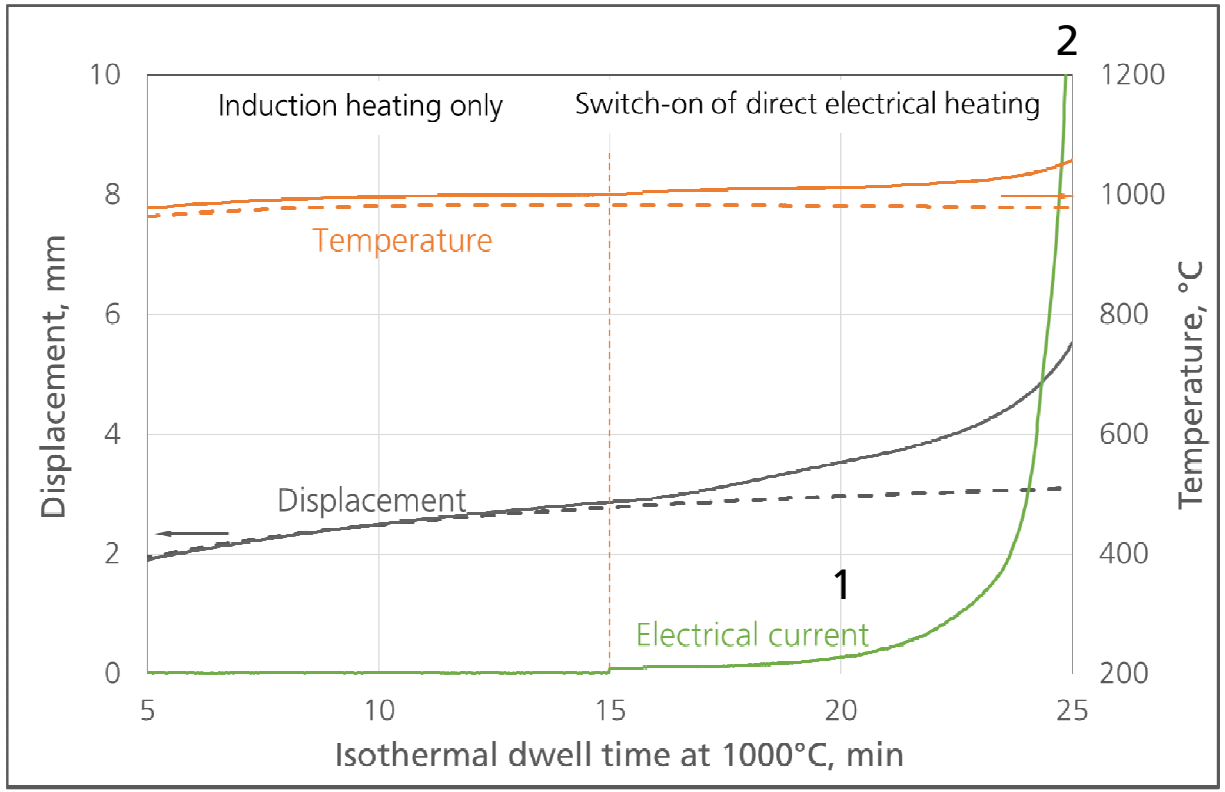
Results – FAST/Flash ZrO_2 (3YSZ)

FAST/Flash tool

- I. Induction heating only
- II. FAST/Flash
  - Electrical current rises
  - Densification rises
  - Temperature rises slowly

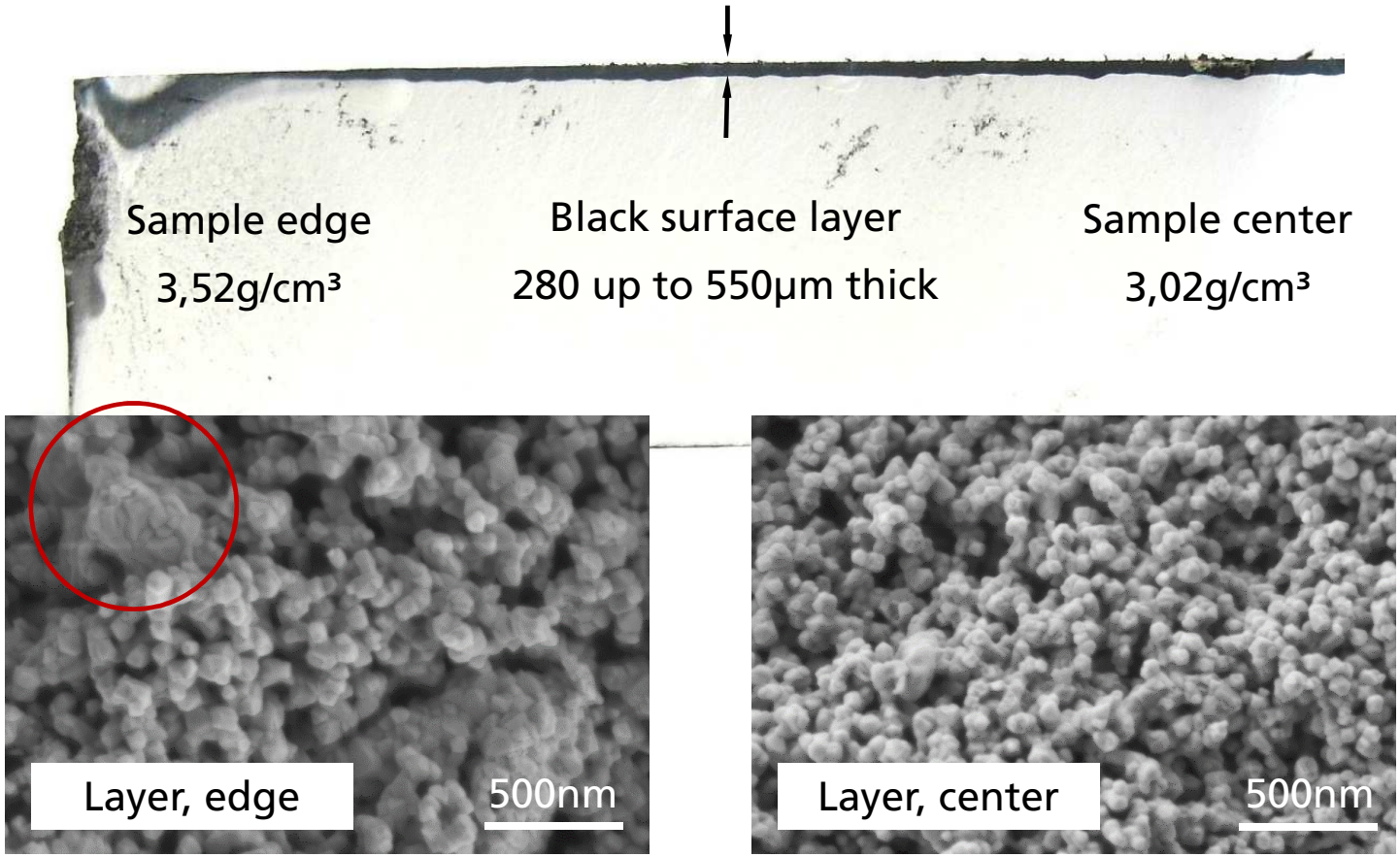
- Control of the process possible?
- Homogeneous process?



Results – FAST/Flash ZrO_2 (3YSZ)

II. FAST/Flash – stage 1

- Cathode
  - Black surface layer
  - Oxygen deficit ZrO_{2-x}
  - Interaction due to radial temperature gradient
- Anode
  - ZrO_2
  - No surface layer



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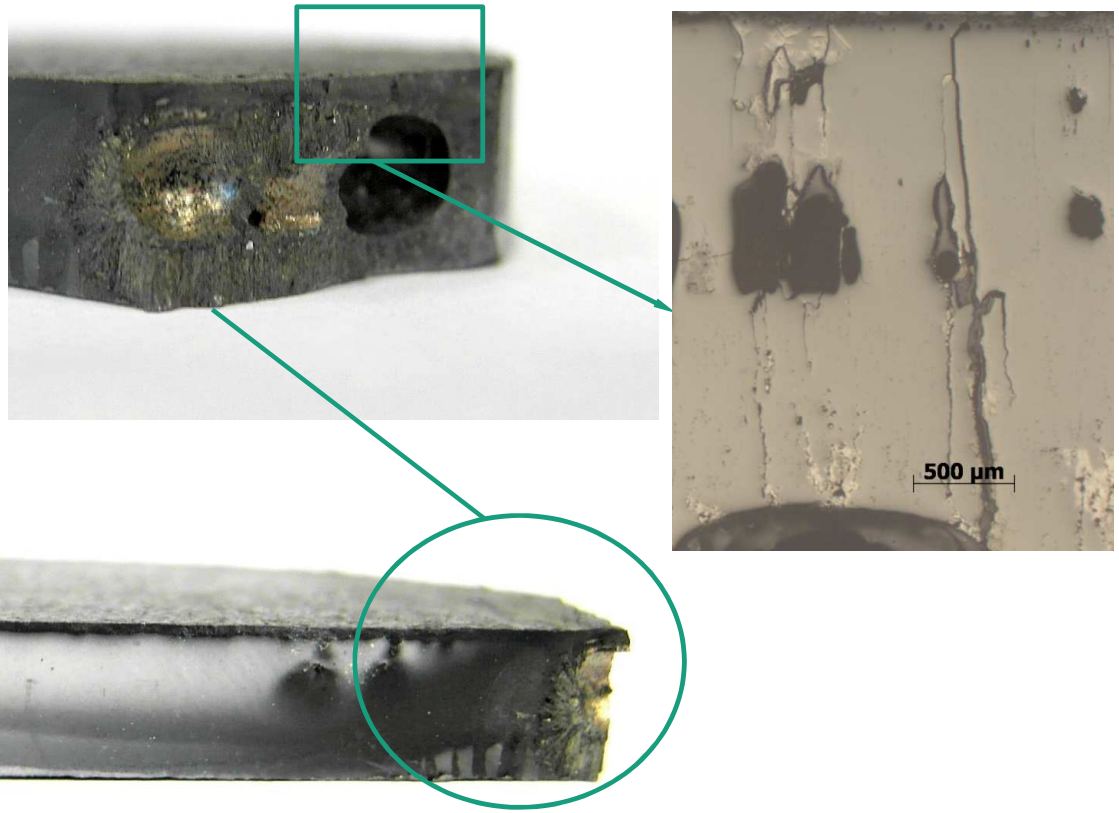
Results – FAST/Flash ZrO_2 (3YSZ)

II. FAST/Flash – stage 2

- Not limiting the electrical powder
- Cathode
  - Further reduction of ZrO_2 in direction to the anode side
- Anode
  - ZrO_2
- Non-homogeneous effect



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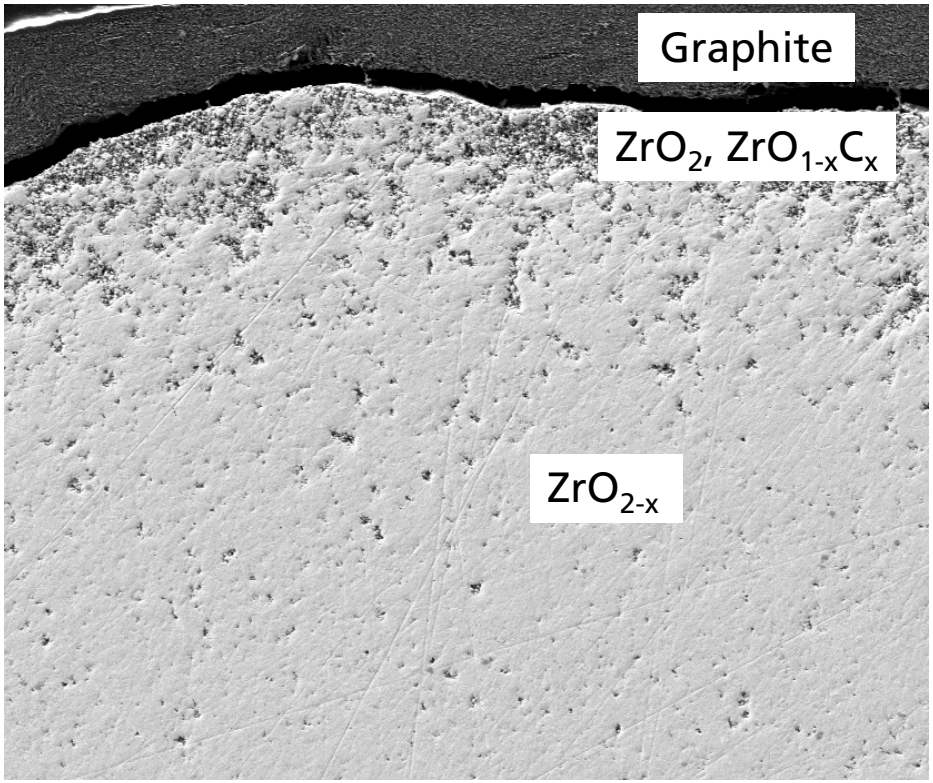
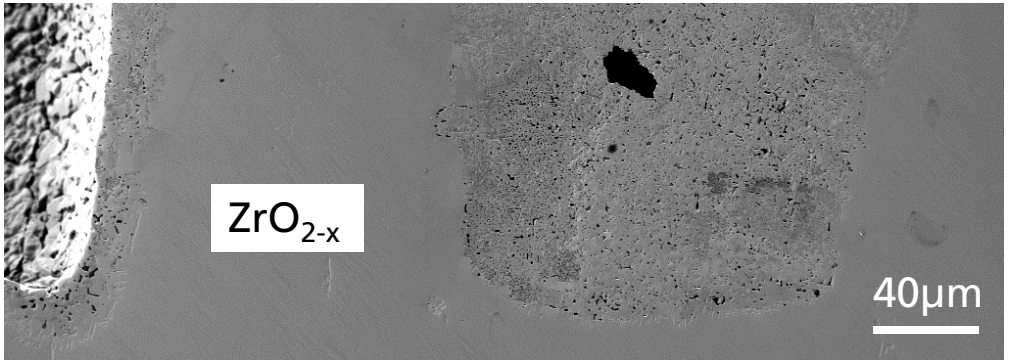
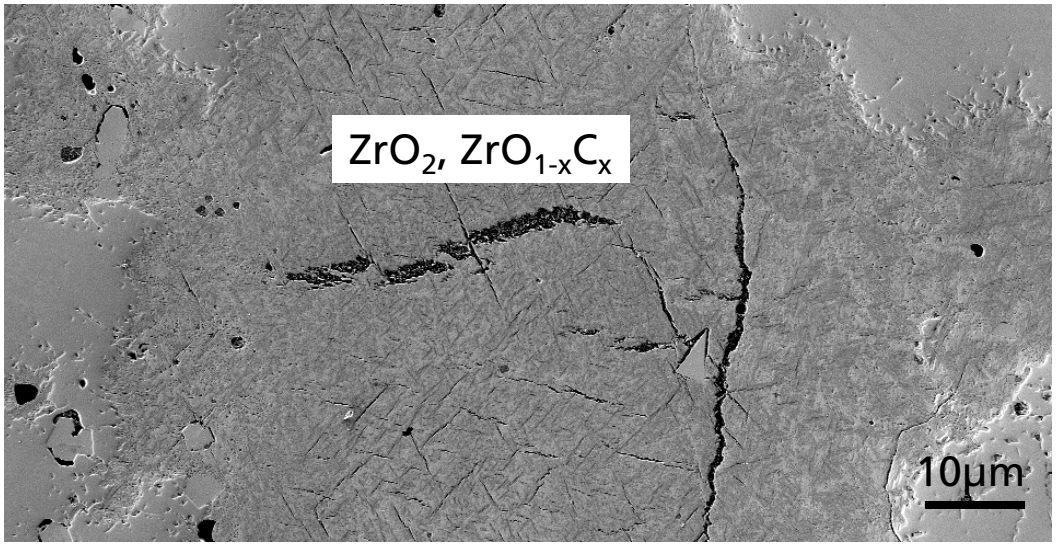
Results – FAST/Flash ZrO_2 (3YSZ)







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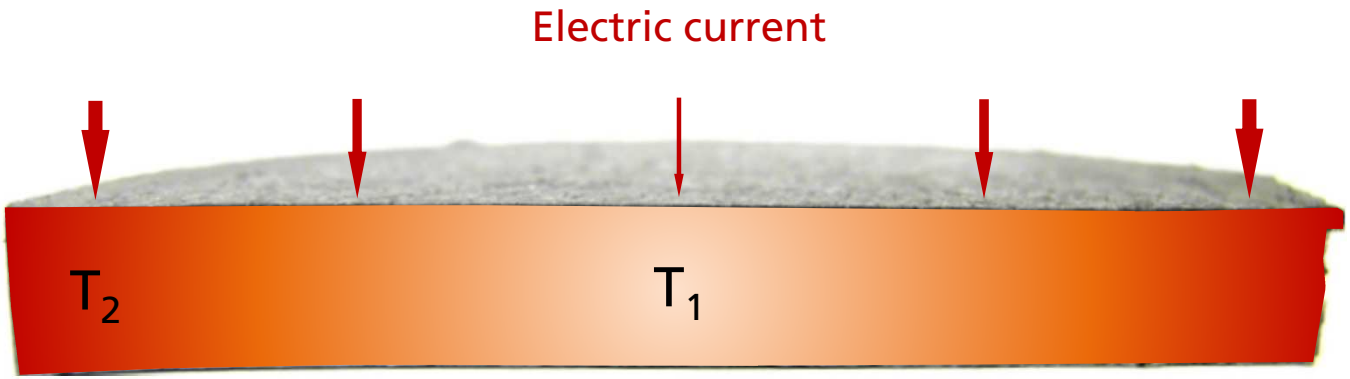
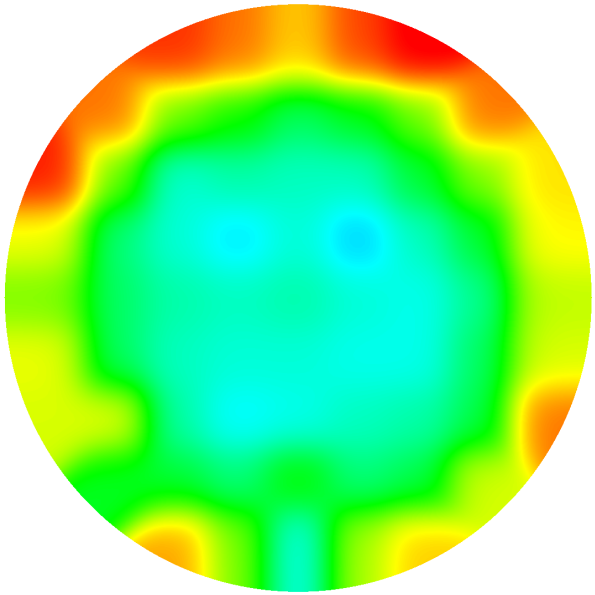
Results – FAST/Flash ZrO_2 (3YSZ)

- Induction heating only → overheating of specimens edge
- $T_2 > T_1 \rightarrow \rho_2 > \rho_1 \rightarrow \sigma_2 < \sigma_1$
- Temperature gradient further rises
- ZrO_2 is an NTC thermistor



Local density distribution



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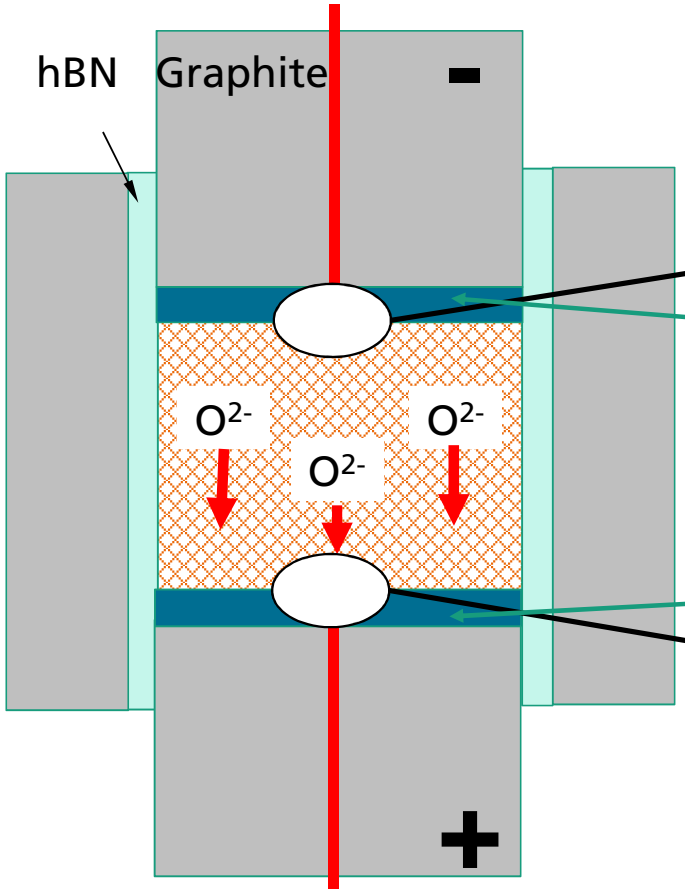
Results – FAST/Flash ZrO_2 (3YSZ)



Electronic conductivity

Reaction at the interface

$C + 4e^- \rightarrow C^{4-}$ (ZrC)

$Zr^{4+} + 4e^- \rightarrow Zr^{3+}$ (ZrO_{2-x})

What happens if cathode is non-reactive?

Molybdenum foil

$O + C \rightarrow CO$

$O^{2-} + C \rightarrow CO + 2e^-$

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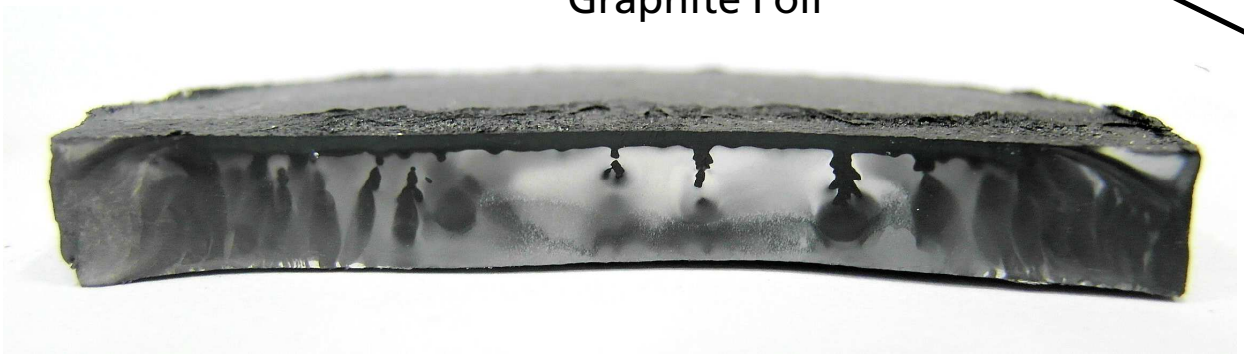
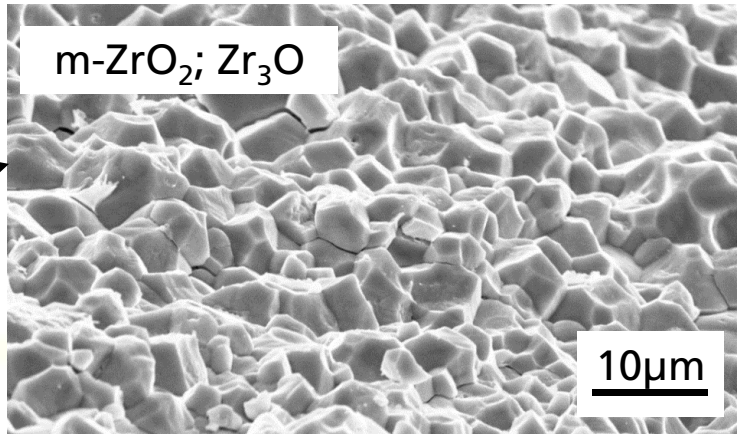
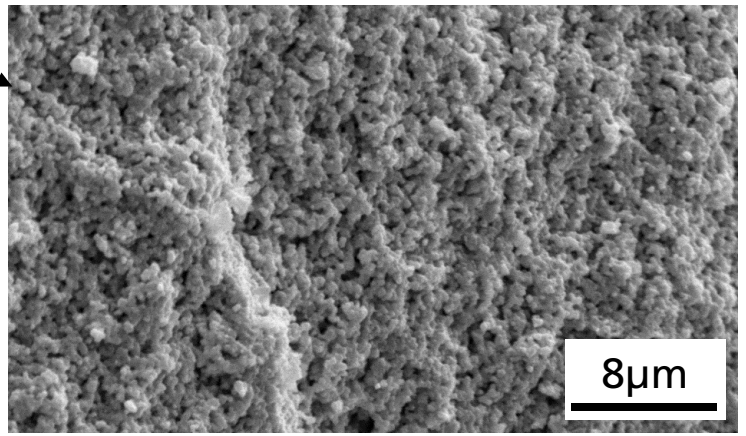
Results – FAST/Flash ZrO_2 (3YSZ)

Molybdenum foil

Graphite Foil







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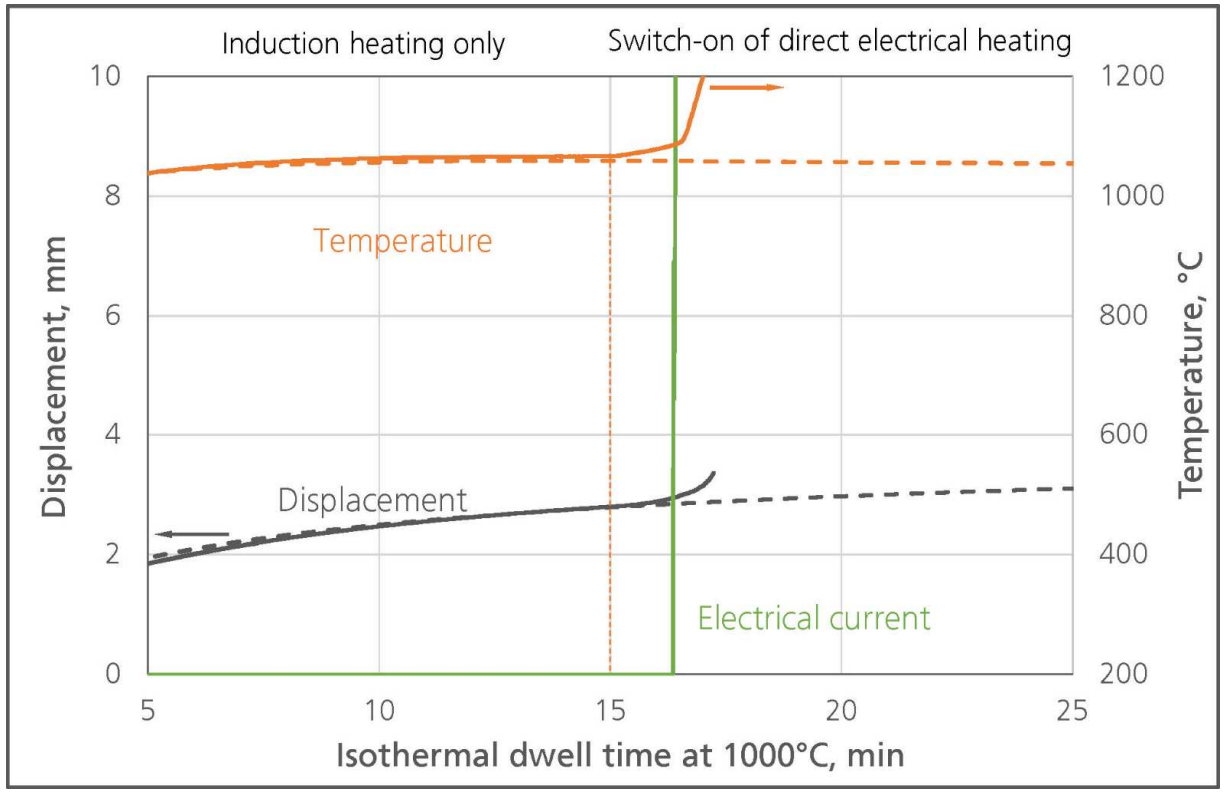
Results – FAST/Flash TiN-ZrO_2

- Stage 1 (voltage control)
  - ~ 1,5min
- Stage 2 (non-linear increase of σ)
  - immediate increase of electrical current
- Stage 3 ?



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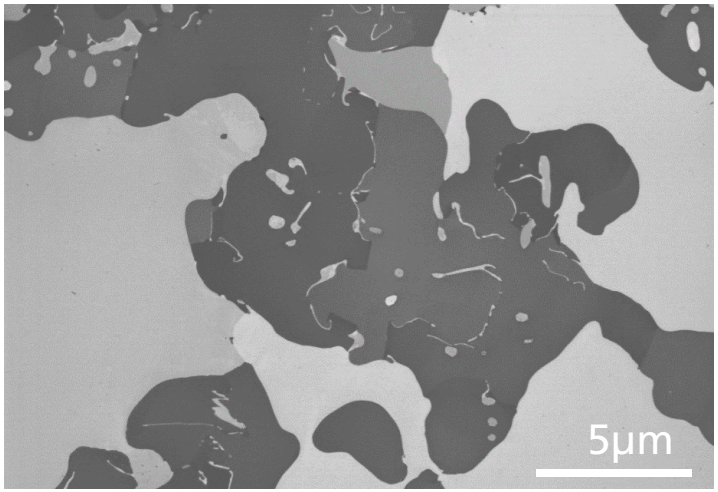
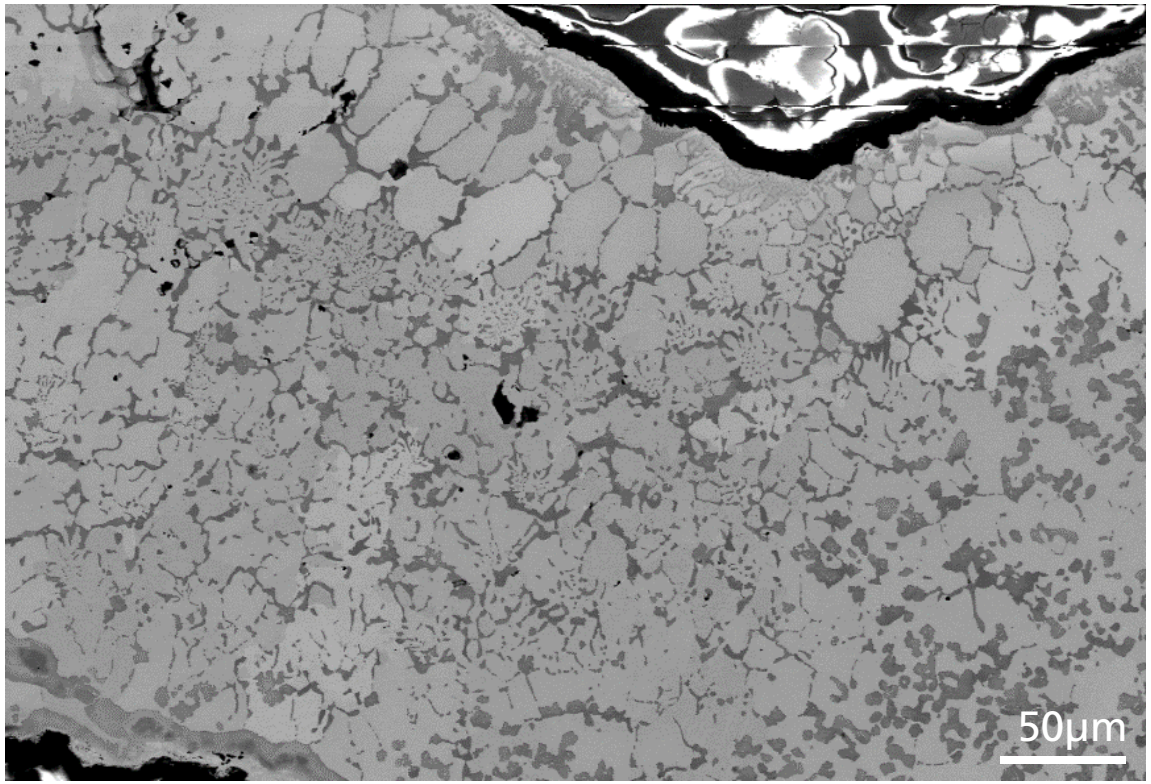
Results – FAST/Flash TiN-ZrO_2

- Microstructure of local molten area
  - Strongly distorted grains
  - Solid solution of Ti-Zr-O-N rich of Zr or Ti?





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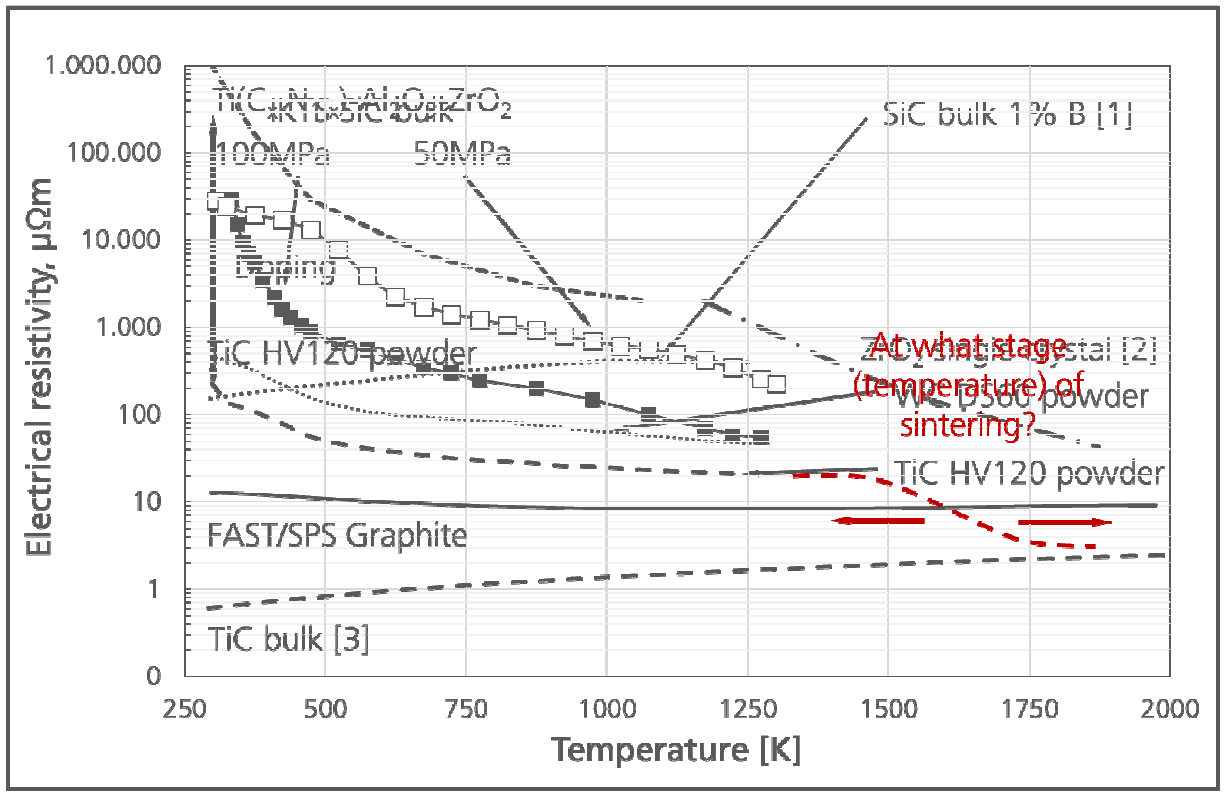
Discussion – Electrical resistivity of bulk materials vs. powders

- Bulk materials NTC – PTC
  - Semiconductor (doping level)
  - Metallic conductor
  - Ion conductor
- Composite materials
  - Resistivity close to percolation threshold
  - Combination of ion and metallic conductor
- Powder morphology, applied force
- Powder which sinter: NTC thermistor



[1] Gmelin 1984; [2] Ikeda 1983; [3] Kosolapova 1990

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Conclusion

- FAST/Flash advantages
  - Larger parts apart from dog bone
  - Starting from powder not pre-sintered parts
  - For non-oxide ceramics: optimization of process (SiC-based)
- FAST/Flash – actual area of work
  - Electrode (tool) material
  - Low oxygen partial pressure
  - Temperature homogeneity
  - NTC behavior of powders or pre-sintered samples
  - (Reproducible) process control?

→ **Enhanced high temperature electrochemistry and impact on parts (new) properties**

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