„FAST/SPS of hBN/TiB_2 Composites“

J. Räthel, M. Herrmann

Fraunhofer Institute for Ceramic Technologies
and Systems, Dresden, Germany

ICS IX

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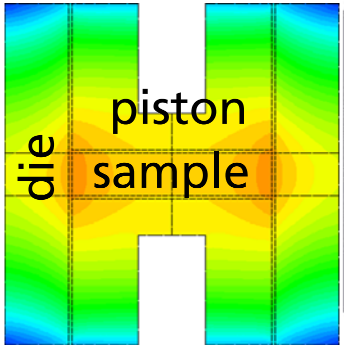
Outline

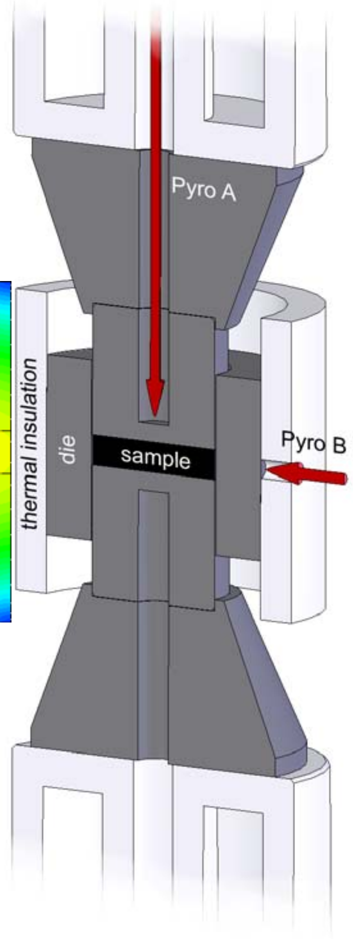
- Motivation
- Experimental/ Results
- Conclusion

Motivation

Temperature measurements during FAST/SPS:

- Possibilities
  - TC, Pyrometer, melting point calibration, IR-camera
- Influenced by
  - Place of measurement, Temperature
  - Tool design (die thickness, thermal insulation,…)
  - Electrical resistivity of sample
- Effects
  - “low temperature sintering” by FAST/SPS in literature





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Motivation

Electrically conductive composites

- Composition close to percolation threshold
  - Small change in composition → large change in resistivity
  - “Sintering behavior”, thermal conductivity, heat capacity nearly constant
- Advantages
  - Observation of temperature differences only dependent on volume change (sample consolidation) and/or electrical resistivity

→ **Observation of temperature differences between two pyrometers**

Outline

- Motivation
- Experimental/ Results
  - Temperature measurements
  - Resistivity measurements
  - FEM Simulation
- Conclusion

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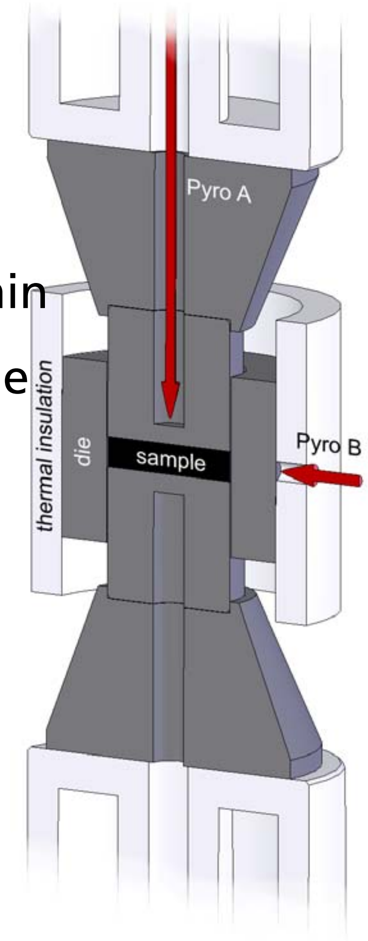
Experimental/ Results

Investigation on hBN/TiB_2 composites

- FAST/SPS FCT HPD 25/1, two pyrometers
  - Sample ∅40mm,sample height 5mm
  - 1900°C,50MPa,5min isothermal dwell time,100K/min
  - 1st run sample,1st run and 2nd run on same sample
- Measurements
  - Density,electrical resistivity
- Three compositions of hBN/TiB_2 composites [vol%]

	high ohmic	medium ohmic	low ohmic
hBN	32	34	50
TiB_2	68	66	50



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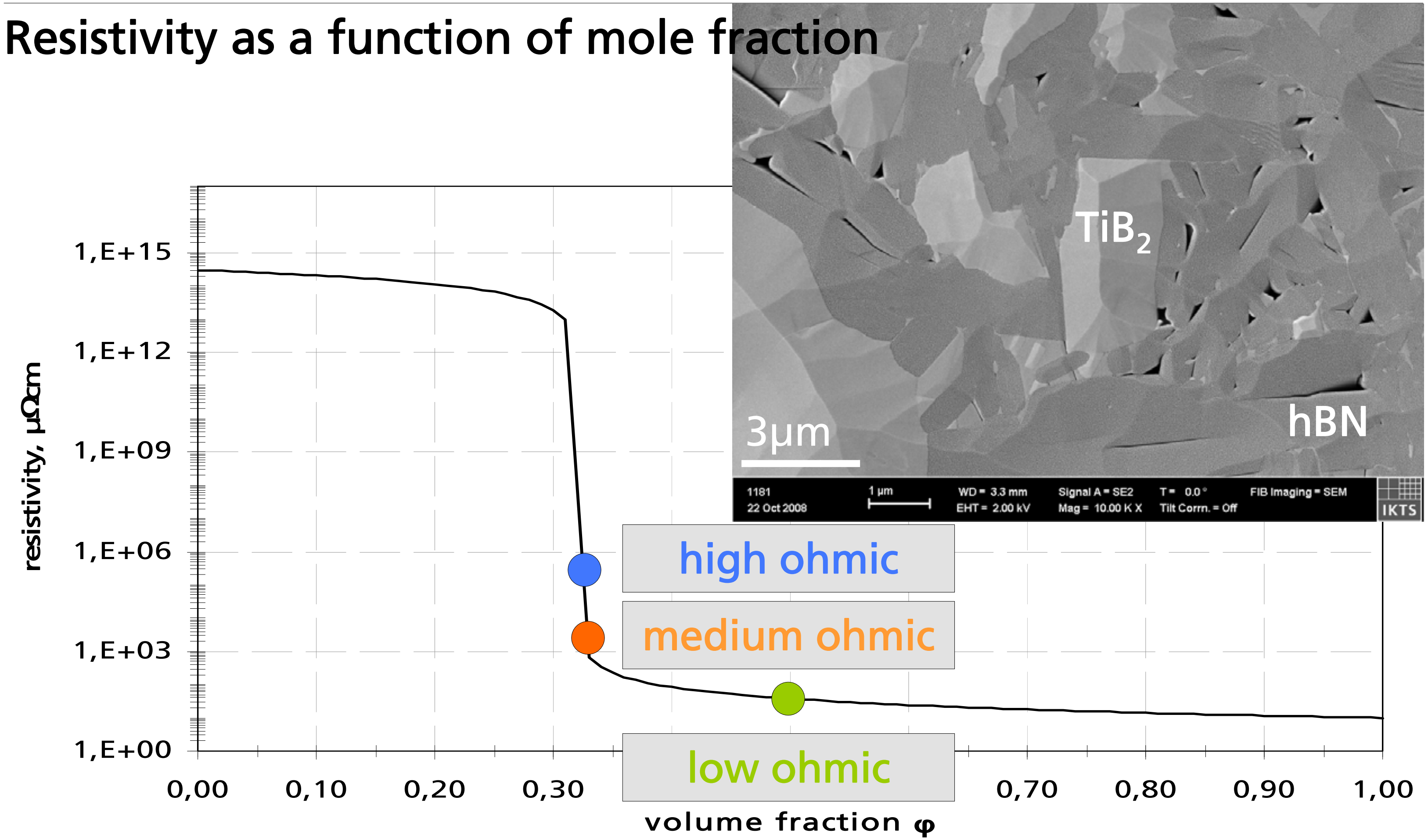
Experimental/ Results

Resistivity as a function of mole fraction



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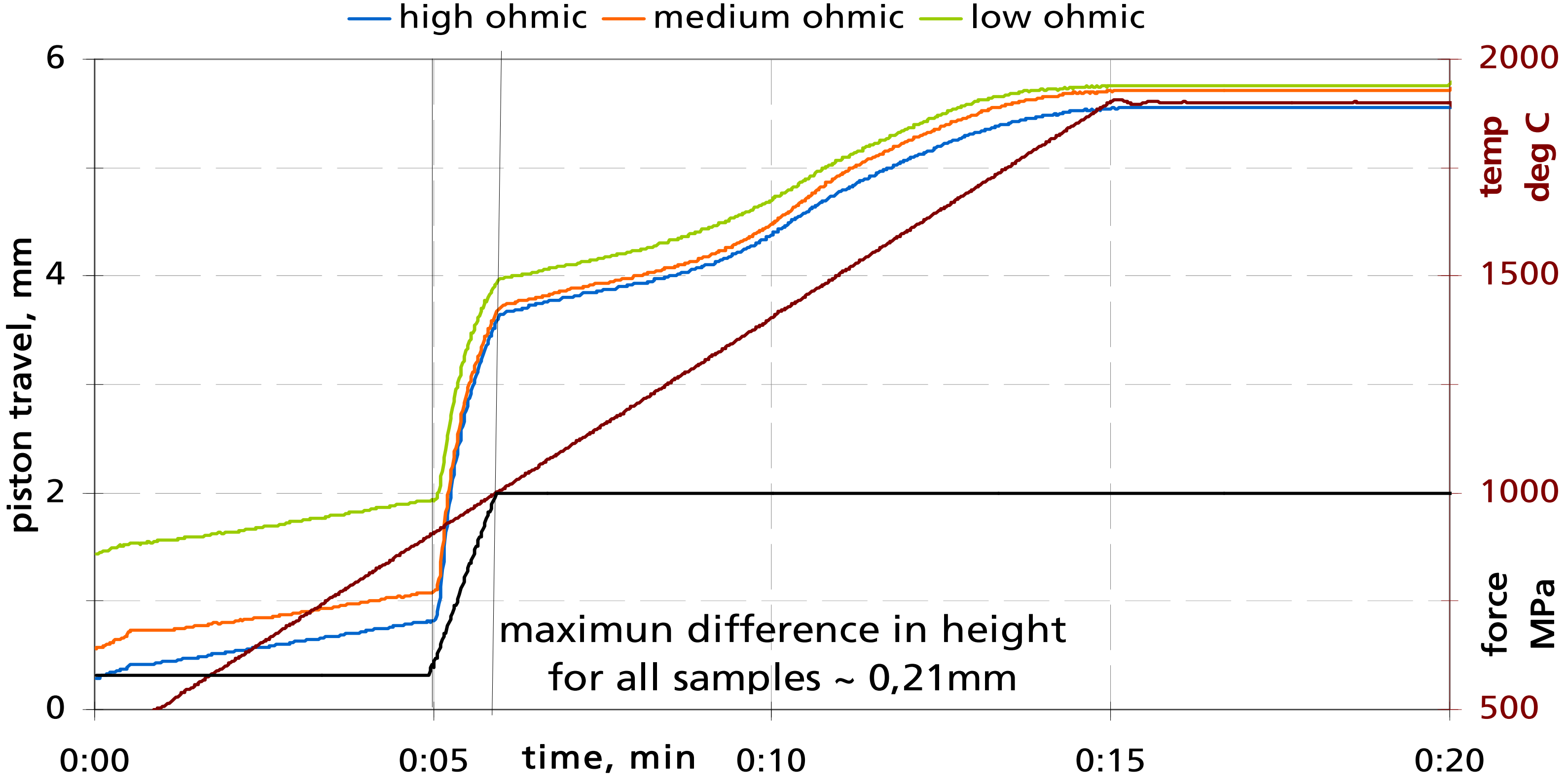
Corrected piston travel as a function of time (1st run)

(subtracting piston travel of second run from first run's piston travel)



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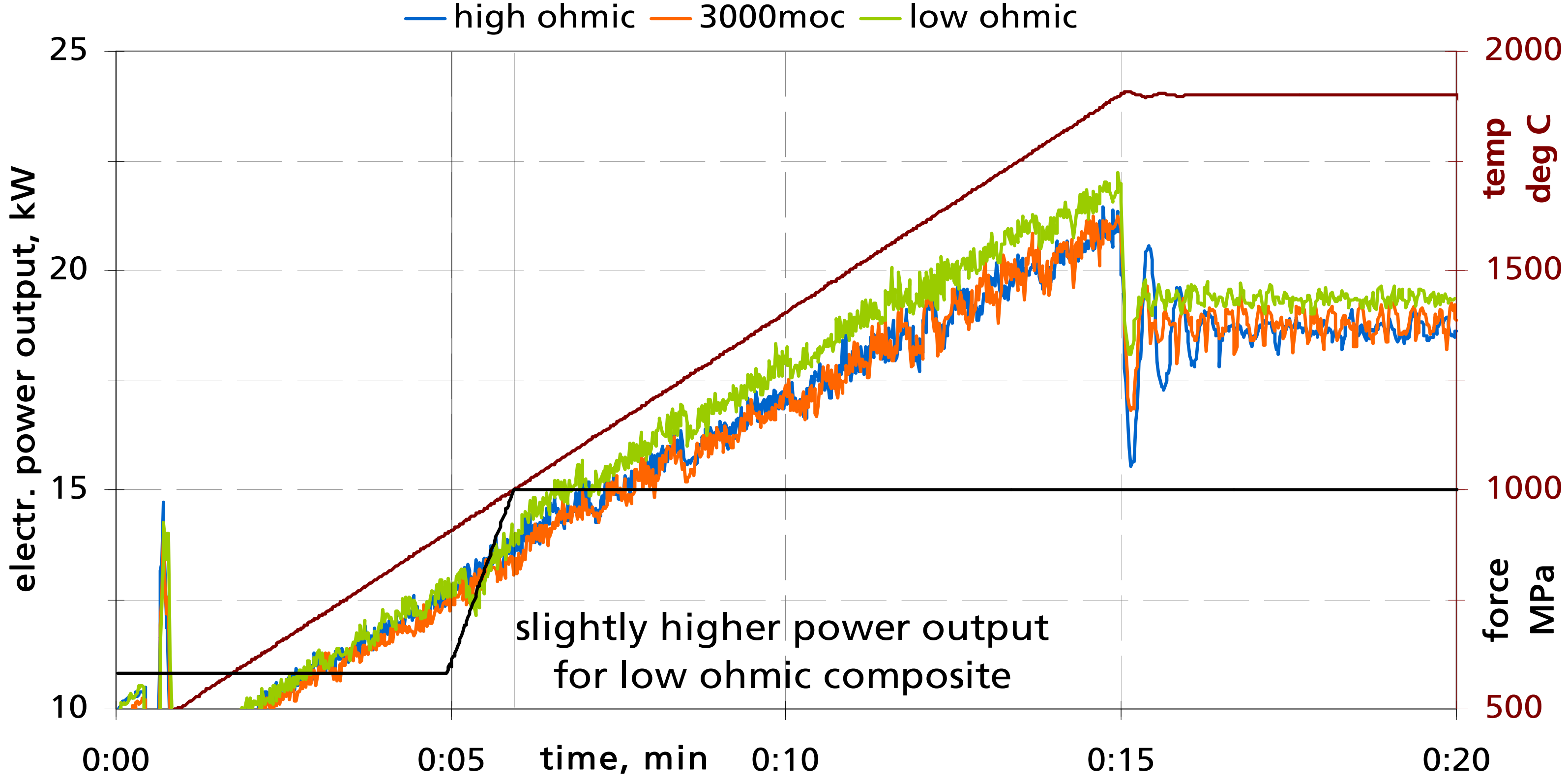
Experimental – temperature measurements

Overall electrical power output as a function of time

(no difference between 1st and 2nd run)



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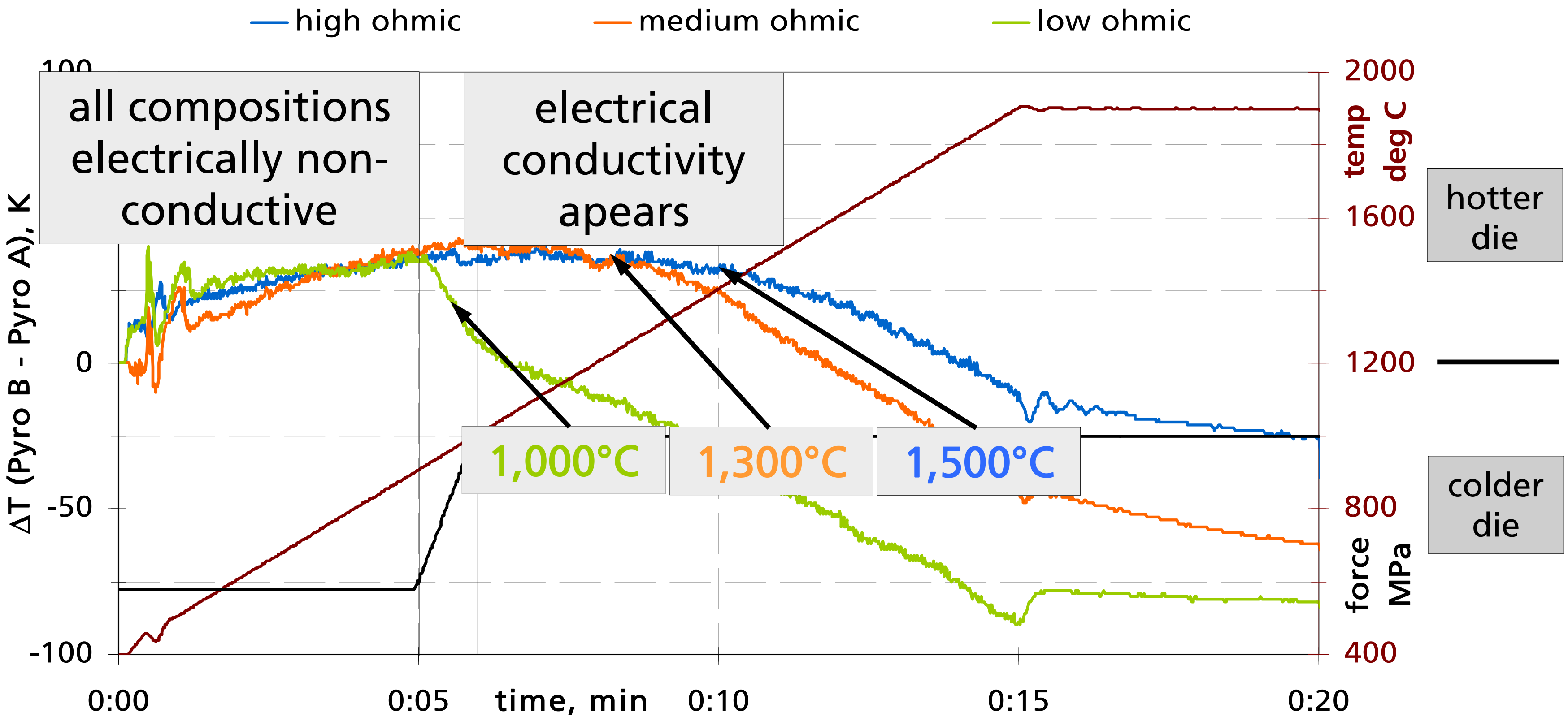
Experimental – temperature measurements

ΔT (Pyro B - Pyro A) of 1st run as a function of time



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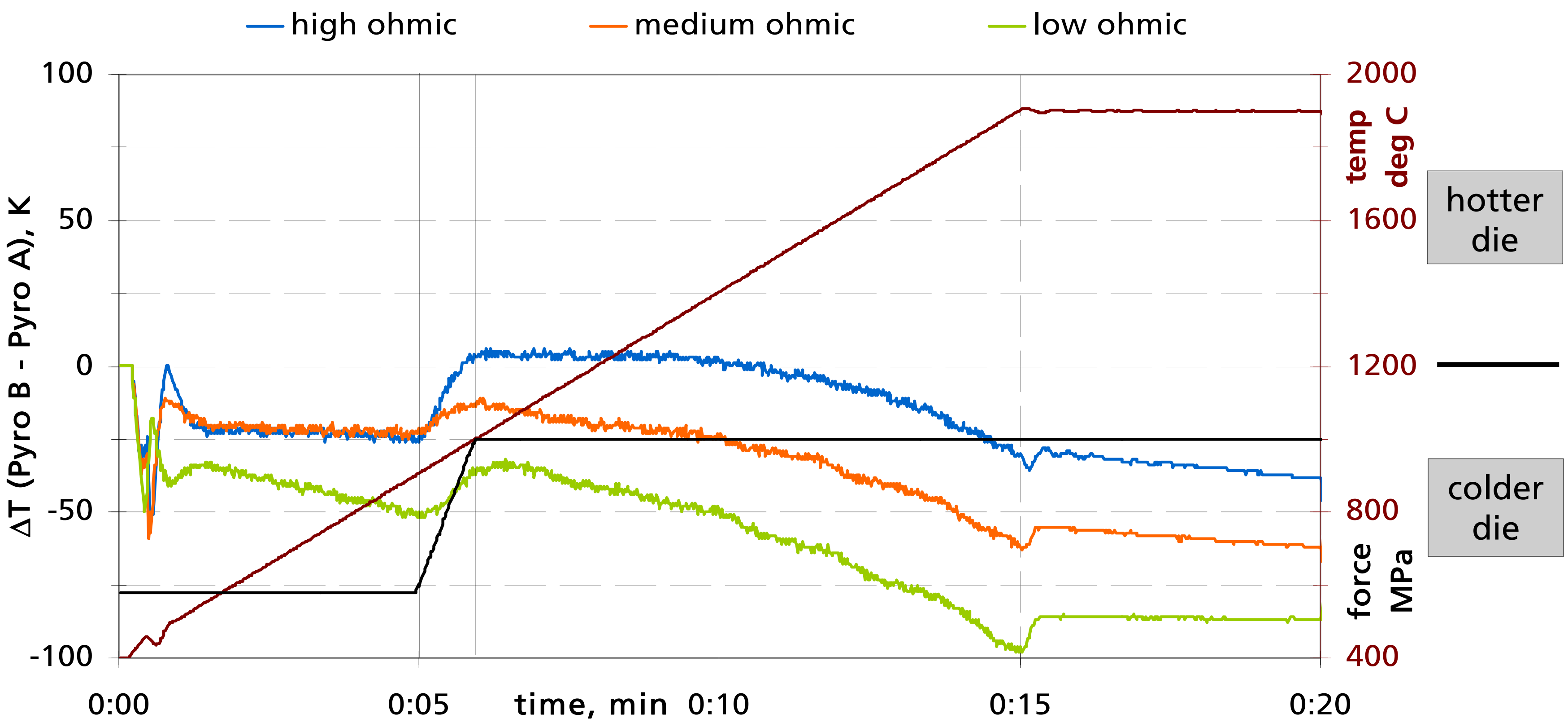
Experimental – temperature measurements

ΔT (Pyro B - Pyro A) of 2nd run as a function of time



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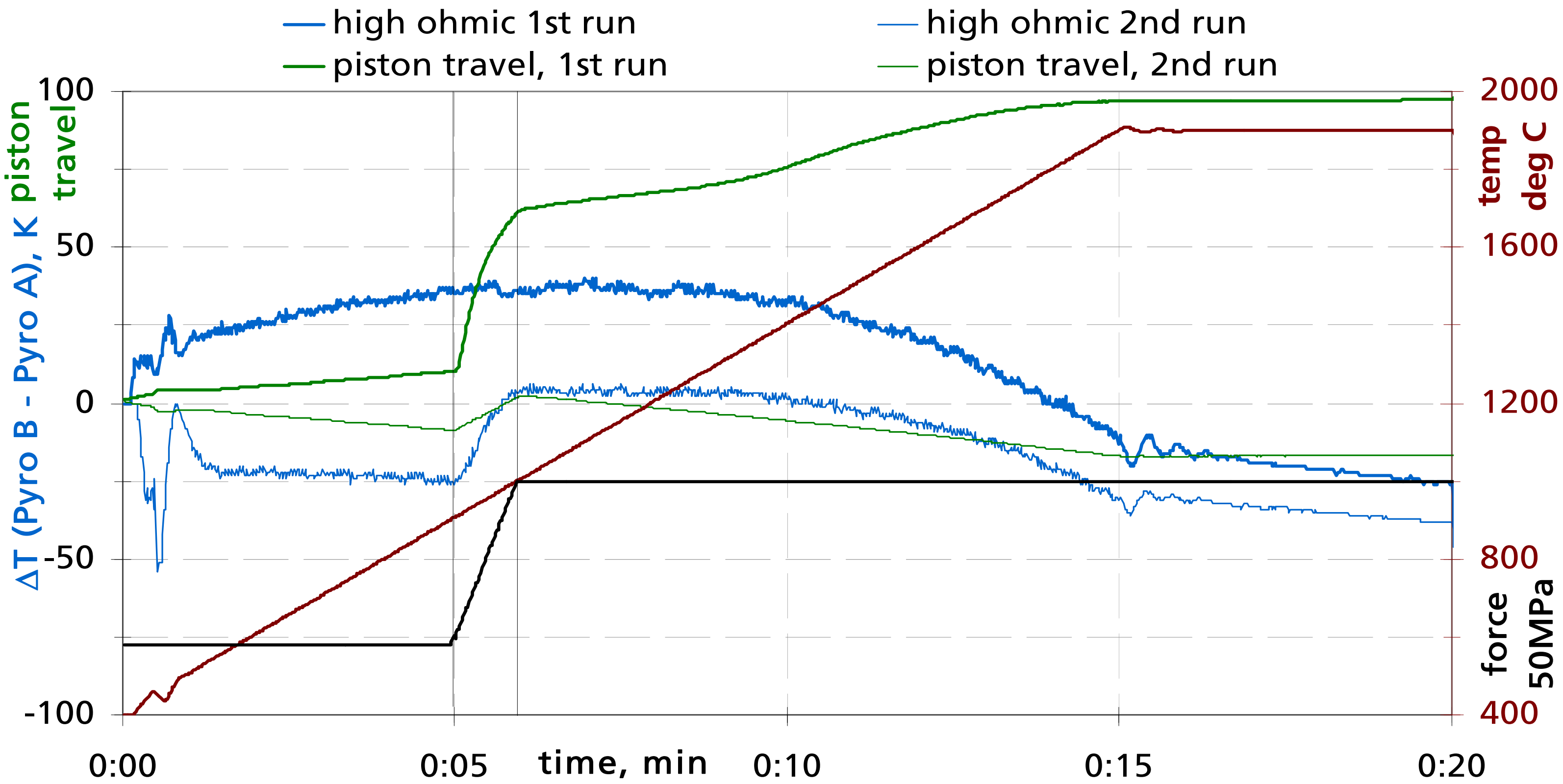
Experimental – temperature measurements

ΔT (Pyro B - Pyro A), piston travel as a function of time



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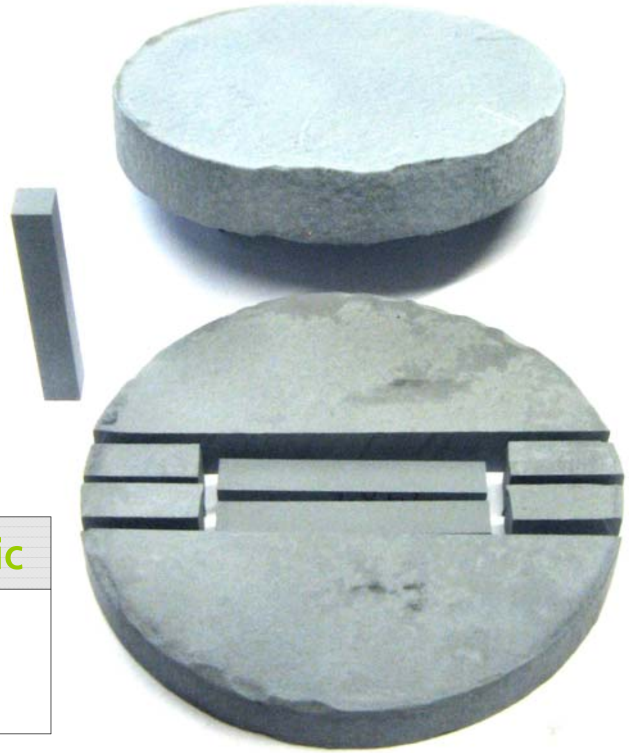
Experimental – resistivity measurements

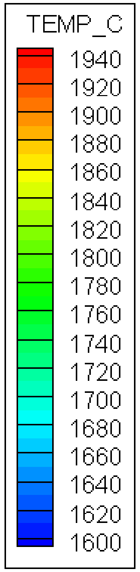
Resistivity measurements

- Method
  - 20x3x3mm samples
  - Four- point measurements
  - Room temperature only

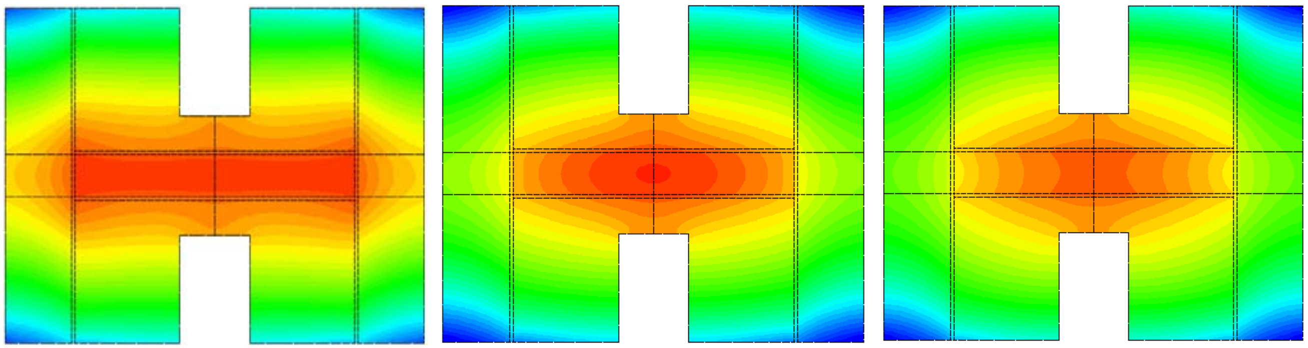


after	high ohmic	medium ohmic	low ohmic
1st run	7000±100	1100±100	32±5
2nd run	2950±100	425±50	41±5

Experimental – FEM simulation



Temperature distribution at the end of the isothermal dwell time at 1,900°C for the 1st run



ΔT (Pyro A–Pyro B)	high ohmic	medium ohmic	low ohmic
FEM	-20K	-70K	-80K
Measurement	-25K	-55K	-85K

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Conclusion

Electrically conductive composites

- Comparison of change in microstructure (REM) → resistivity measurements more sensible
- Small variation in composition → large change in electrical resistivity, but small change for all other material's properties
- Different compositions → separation of temperature effects induced by
  - sintering shrinkage and
  - occurring low electrical resistivity

FEM modeling

- Displaying well calculated temperature distribution
- Validation of the FEM model by measured data

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