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Electric Field Enhanced Processing of Advanced Materials II: Complexities and Opportunities

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Field assisted sintering of larger scaled ceramic parts using adapted tool design and hybrid heating

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Field Enhanced Processing of Advanced Materials II: Complexities and Opportunities
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Forschungszentrum Jülich GmbH
Institute of Energy and Climate Research
IEK-1: Materials Synthesis and Processing

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Motivation of field assisted sintering technologies

Attractive due to:

- Direct heat transfer
- High heating rates
- Pressure assisted sintering

→ Rapid densification
→ New material concepts
→ Efficiency

Challenges of scaling up:

- Thermal gradients
- Tool design and tool materials
- Taking advantage of new heating concepts (e.g. Hybrid heating, Flash)
- Net-shaping
- Startegies for automated production

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FAST/SPS equipment at our institute

Lab-scale: HP-D 5

50 kN max. force
37 kW power source
Max. sample size ∅ 30 mm
Atmosphere control

Scale up: H-HP-D25 SD/FL/MoSi

Max. load 250 kN
Max. sample size ∅ 100 mm
60 kW for FAST/SPS heating
80 kW for hybrid heating
1.000 V voltage source (AC/DC)

FCT Systeme GmbH FCT Group

FAST/SPS equipment at our institute

Lab-scale: HP-D 5

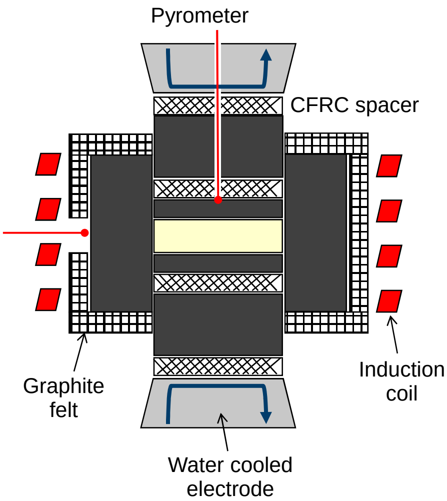
Modes of operation:
- FAST/SPS

Scale up: H-HP-D25 SD/FL/MoSi

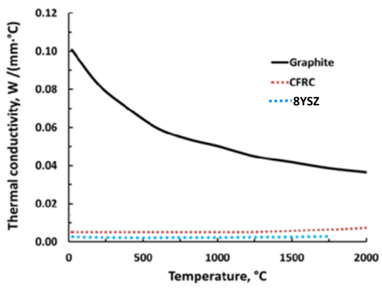
Modes of operation:
- FAST/SPS
- Hybrid FAST/SPS (+ induction coil)
- Flash (AC/DC source + induction coil)
- Hot press (+ $MoSi_2$ heater)

Concepts of reducing thermal gradients

Graphite tool
sample ∅100 mm



CFRC = Carbon fiber reinforced carbon



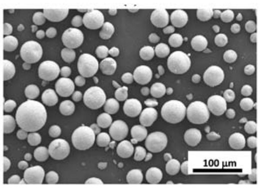
Well-balanced combination of all influence factors

Importance of thermal insulation (HP-D5, Tool for sample size ∅17 mm)

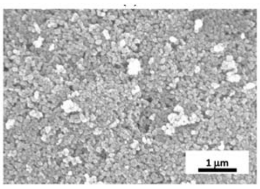
A.M. Laptev et al. J. Mat. Proc. Techn. 262 (2018) 326 - 339.

Starting materials and FAST/SPS parameters

8YSZ powder



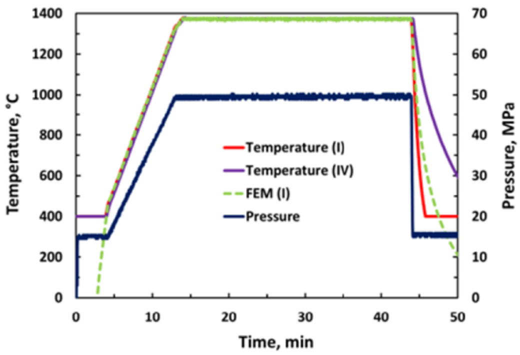
soft agglomerates



small primary particles

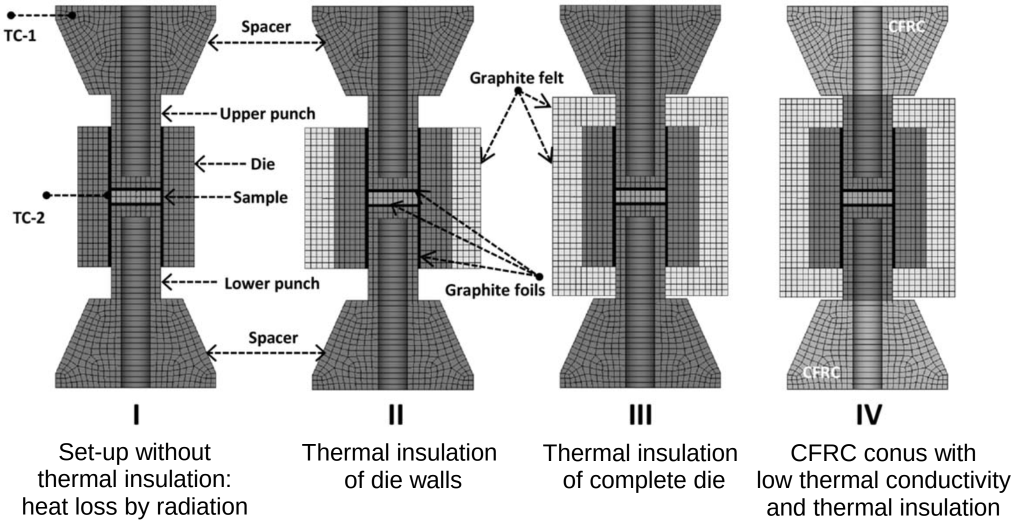
Precipitation, spray drying (TOSOH)
Agglomerate size 10 - 50 µm
Primary particle size < 100 nm (16 ± 3 m^2/g)
8YSZ = ZrO_2 doped with 8 mol.% Y_2O_3

Established for SOFC/SOEC electrolytes



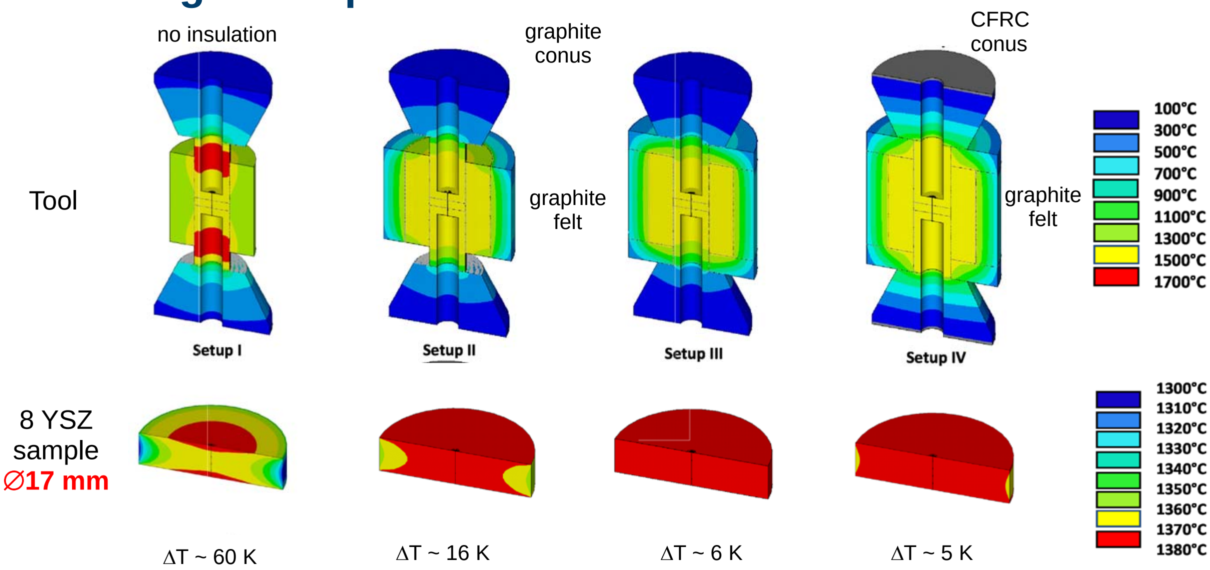
FAST/SPS parameters:
Heating: 100 K/min, 50 K/min
T = 1375°C, t = 30 min
p = 50 MPa
Sintering density > 94%

Different set-ups of thermal insulation



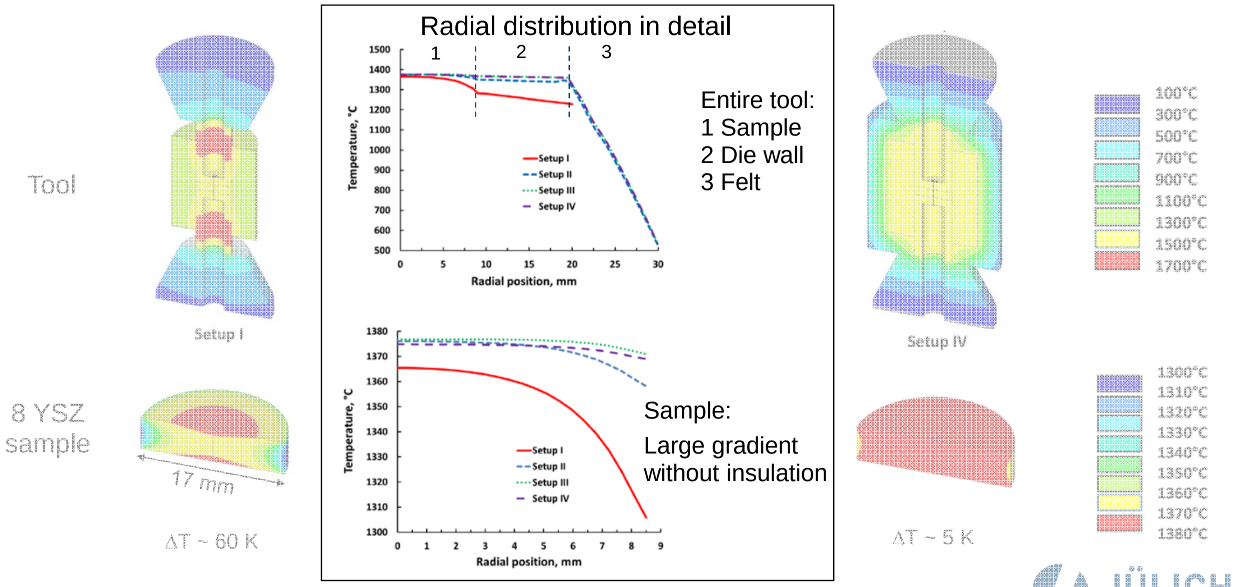
Sample diameter: 17 mm
Die wall thickness: 11.5 mm
Graphite felt: 11.5 mm

Modelling of temperature distribution



A.M. Laptev et al. J. Mat. Proc. Techn. 262 (2018) 326 - 339.

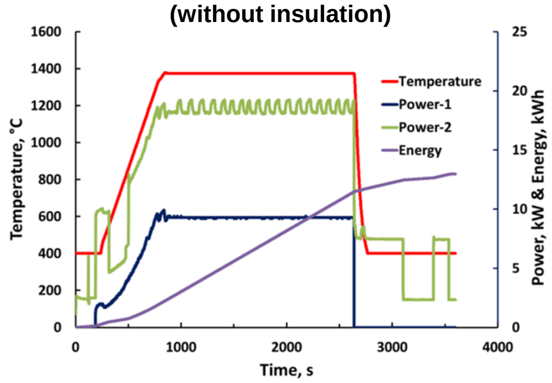
Modelling of temperature distribution



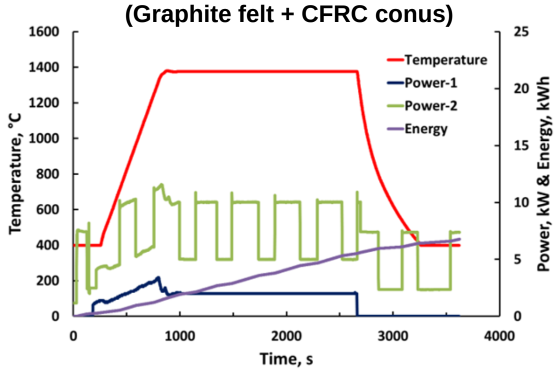
A.M. Laptev et al. J. Mat. Proc. Techn. 262 (2018) 326 - 339.

Total energy consumption

Setup I (without insulation)



Setup IV (Graphite felt + CFRC conus)



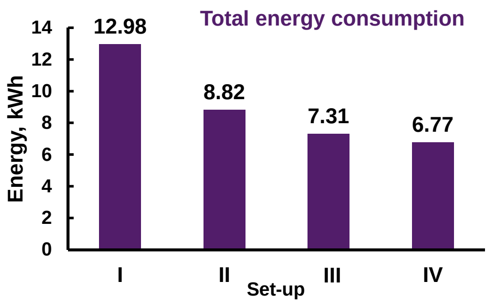
Power 1 = Data logger HP-D5

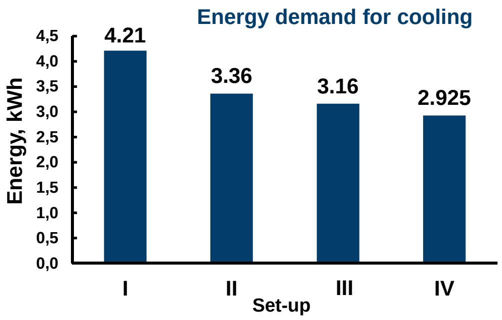
Power 2 = Total power demand

- **FAST/SPS heating**
- **Cooling water**
- **Hydraulic press**
- **System control**

External power & energy data logger Chauvin Arnoux PEL 102/103

Measurement of total energy consumption





Total energy consumption:
Reduced by factor 2 due to thermal insulation and CFRC spacer (set-up IV)

To consider:
Large contribution of cooling water system

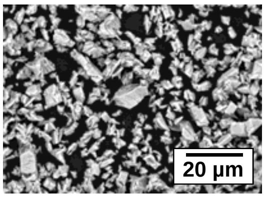
Scaling up of technology (H-HP-D25, Tool for sample size ∅100 mm)

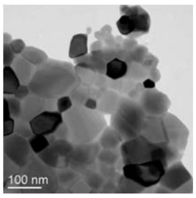
Preliminary results

PhD thesis M. Kindelmann

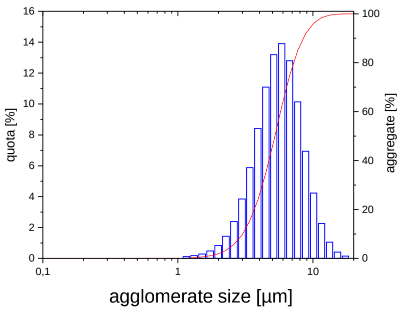
Starting materials and FAST/SPS parameters

Y_2O_3 powder





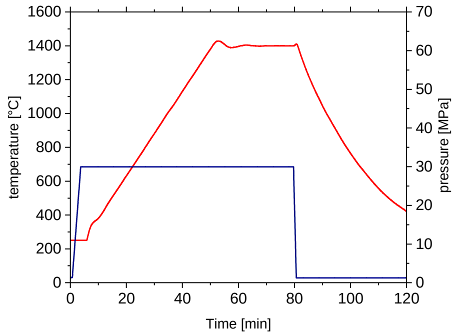
primary particle size ~ 60 nm



agglomerate size

d_{10} = 3.0 µm
d_{50} = 5.2 µm
d_{90} = 8.5 µm

Commercial Y_2O_3 powder with non-optimized powder morphology



FAST/SPS parameters:
Heating rate: 25 K/min
T = 1400°C, t = 30 min
p = 30 MPa
Sintering density > 95%

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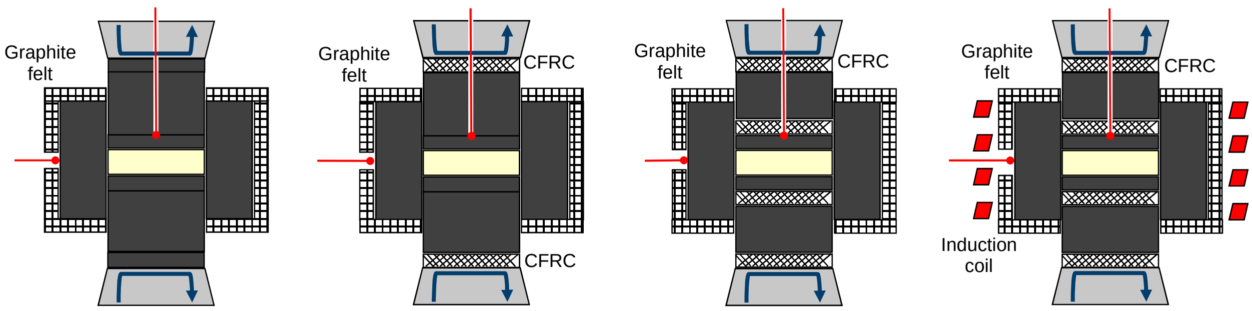
Different experimental set-ups



I	II	III	IV
Graphite felt no CFRC spacer	Graphite felt 1 set of CFRC spacer	Graphite felt 2 sets of CFRC spacers	Graphite felt 2 sets of CFRC spacers Induction coil

= Hybrid FAST/SPS

Sample diameter: 100 mm
Die wall thickness: 35 mm
Graphite felt: 11.5 mm

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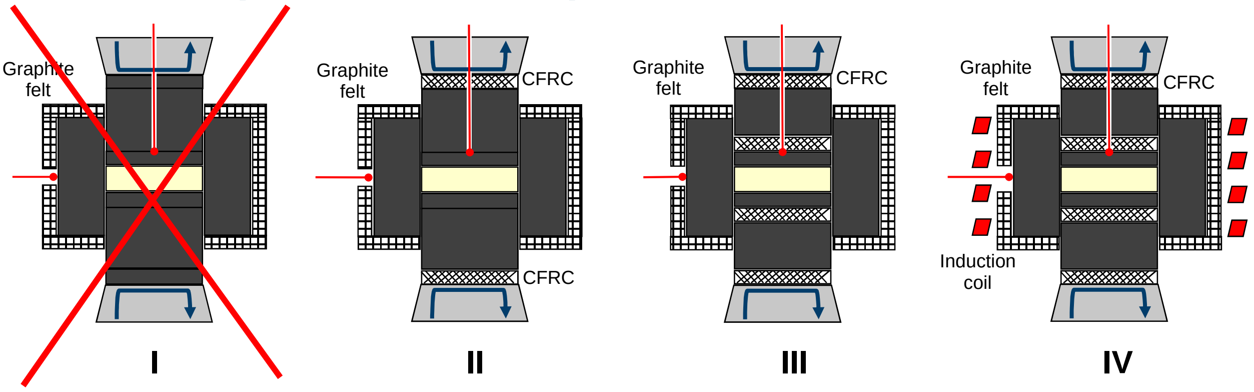
Different experimental set-ups



I
Graphite felt
no CFRC spacer
not possible to achieve 1400°C

II
Graphite felt
1 set of CFRC spacer

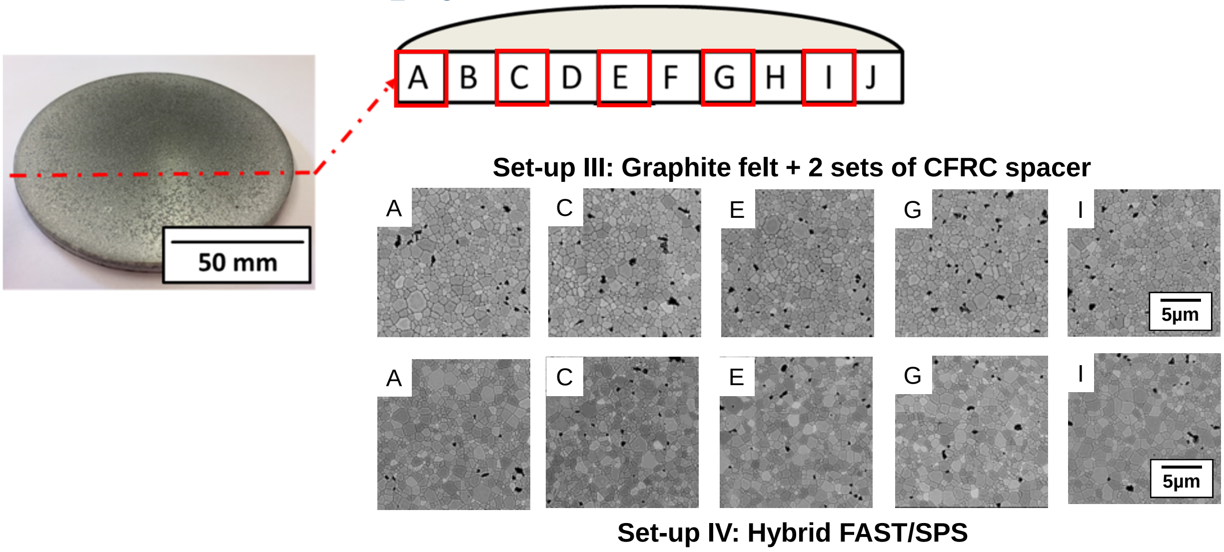
III
Graphite felt
2 sets of CFRC spacers

IV
Graphite felt
2 sets of CFRC spacers
Induction coil
= Hybrid FAST/SPS
experiments in progress

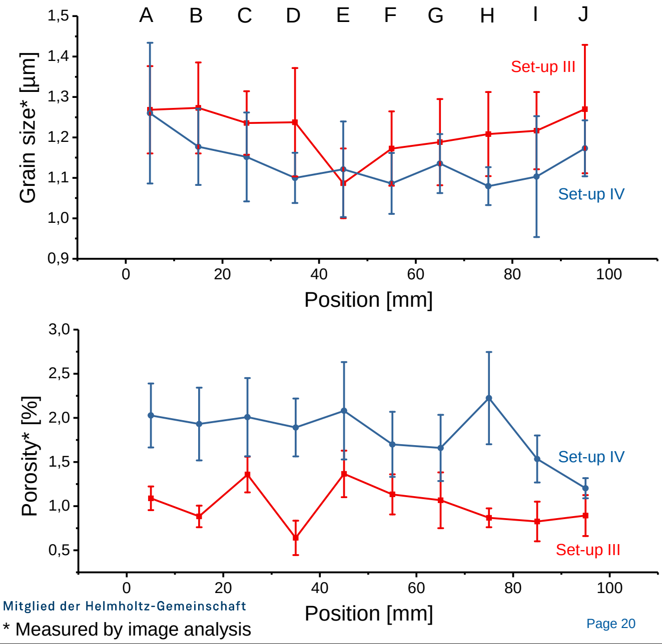
Sample diameter: 100 mm
Die wall thickness: 35 mm
Graphite felt: 11.5 mm

Modelling of temperature distribution in progress…

Microstructure of Y_2O_3 samples



Grain size and porosity of Y_2O_3 samples



Set-up III: FAST/SPS + CFRC spacer

→ Slightly increased grain size

→ Slightly reduced porosity

Set-up IV: Hybrid FAST/SPS

→ No clear advantage for ∅100 mm samples and moderate heating rates (25 K/min)

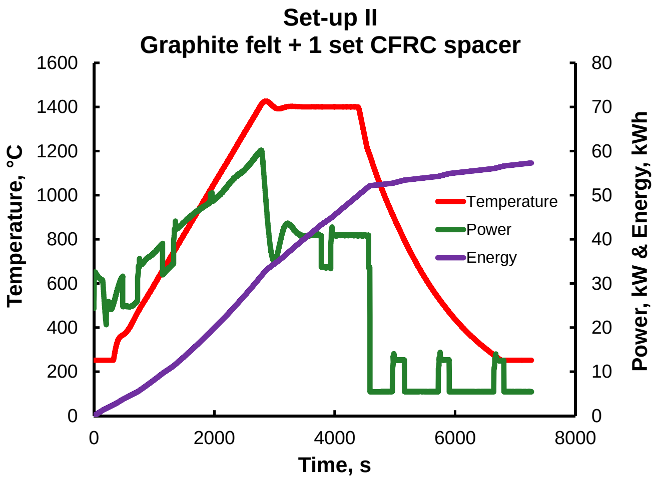
→ Required for higher heating rates

→ Recommended for larger parts > 100 mm**

**J. Hennicke et al. cfi/Ber. DKG 95 (2018) E27 - E33.

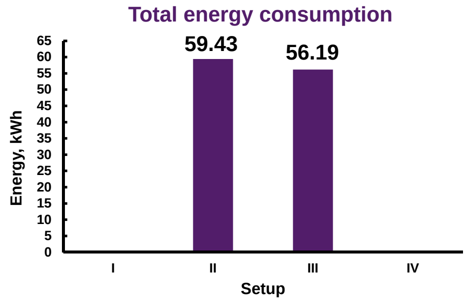
* Measured by image analysis

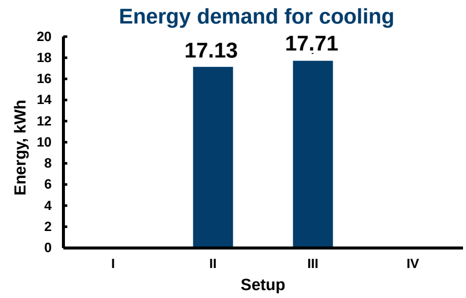
Total energy consumption



Power = Total power demand
- FAST/SPS heating
- Cooling water
- Hydraulic press
- System control

Energy = Total energy





Summary and outlook

Scaling up of FAST/SPS technology:

→ Thermal insulation highly recommended (Graphite felt + CFRC spacer)

→ Hybrid FAST/SPS recommended for higher heating rates

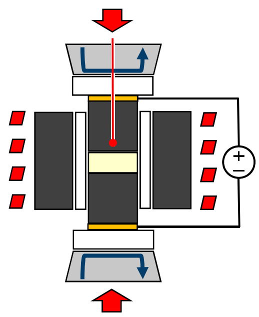
→ Large contribution of cooling water system on total energy consumption

Outlook:

→ Experimental study to be continued…

→ Modelling of temperature distribution
Understanding the role of CRFC spacer

→ Flash sintering in our H-HP-D25 device



Set-up for flash sintering:
- Special graphite/BN tool setup
- Tool ∅ 45 mm
- Pt electrodes
- AC/DC power source with U_{max} = 1000V
- Heating by induction coil

Thank you for your attention!

Our funding is highly acknowledged:

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GZ: BR 3418/1-1
„Feldunterstütztes Sintern von seltenerddotiertem Ceroxid“
im Rahmen des Schwerpunktprogramms SPP 1959