SFA-AM: Precision Free Form Manufacturing (PFFM) - TFA 1

Ceramic X.0

High-precision micro-manufacturing of ceramics

Period 2017-2020/21

Annual Review Meeting 2022

18th March 2022

Juergen Brugger, by ZOOM

The project idea

Precursor preparation

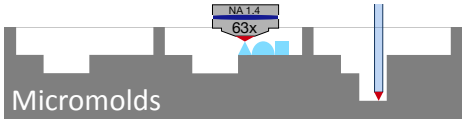
Preceramic Polymers

Nanofillers, catalysts, initiators

Photolitho-graphy

2PP micro 3D printing

Laser ablation

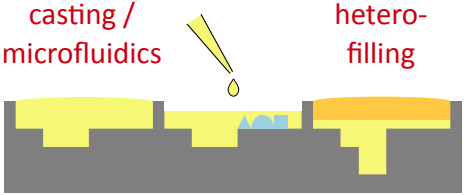


Mold fabrication

Mold filling

casting / microfluidics

hetero-filling



curing, mold release, & pyrolysis

in situ analysis

Polymer derived ceramics

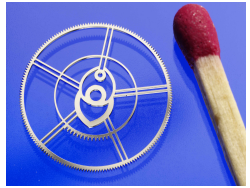
hetero-ceramics

Up-scaling manufacturing

Applications

non-conducting

Biomedical implants



High precision watch parts

conducting

Transducers for harsh settings

Micromechanics

Microreactors

...

Testing

EPFL Microsystems Laboratory

- *Lorenz Hagelüken (PhD Student)*
- *Pierrick Clement, Jongmoon Jang (Postdoc Scientists)*
- *Jürgen Brugger*

EPFL Optics Laboratory

- *Eirini Kakkava (PhD Student)*
- *Gulia Panusa (PhD Student)*
- *Jieping Wang (Postdoc Scientist)*
- *Demetri Psaltis*

EPFL Laboratory of Applied Photonics Devices

- *Georgia Konstantinou (PhD Student)*
- *Christophe Moser*

Empa Laboratory for High Performance Ceramics

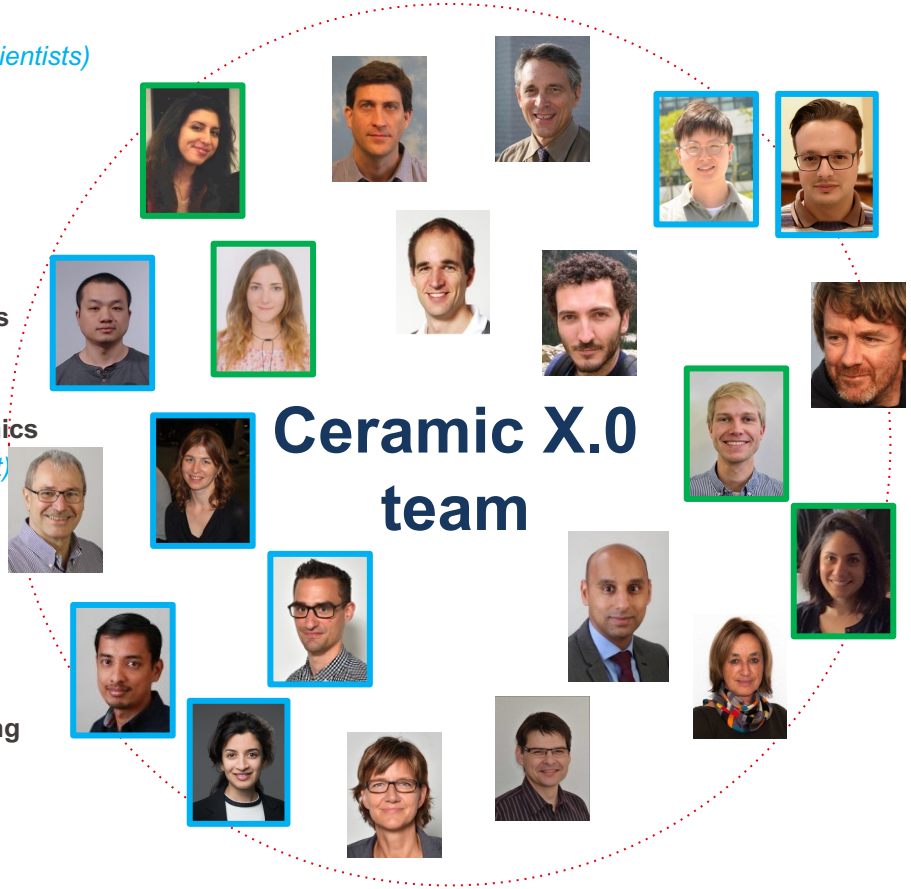
- *Pradeep* Vallachira Warriam *(Postdoc Scientist)*
- *Gurdial Blugan*
- *Jakob Kübler*

EMPA Laboratory for Biointerfaces

- *Yashoda Chandorkar (Postdoc Scientist)*
- *Markus Rottmar*
- *Katharina Maniura*

PSI Photons for Engineering and Manufacturing

- *Malgorzata Makowska (Postdoc Scientist)*
- *Nicola Casati*
- *Daniel Grolimund*
- *Helena van Swygenhoven*



SwissLitho AG
Innovative Nanofabrication Tools

Medtronic

straumann

Mimotec
Micropièces pour grandes idées
Groupe Acrotec

ceramaret
Switzerland

LITHOZ

Overview of key results (1)

Materials	
Polysiloxane → SiOC	*Konstantinou, Add. Man., 2020* *Kollep, Adv. Eng. Mat., 2022*
Polysiloxane + DVB → SiOC	*Sasikumar, Ceram. Int., 2018* *Jang, Ceram. Int., 2021*
Polysiloxane + DVB +Ti → SiTiOC	*Sasikumar, Appl. Mat. Int., 2020* *Jang, Ceram. Int., 2021* *Makowska, Acta Mat., 2020*
Polysilazane + DVB → SiCN	*Hagelüken, J. Eur. Cer. Soc., 2022*

Overview of key results (2)

Shaping techniques:	
Two-photon polymerization	*Konstantinou, Add. Man., 2020*
Direct laser ablation	*on green body and mould*
Macro-moulding	*Sasikumar, Ceram. Int., 2018* *Sasikumar, Appl. Mat. Int., 2020* *Makowska, Acta Mat., 2020* *Jang, Ceram. Int., 2021* *Hagelüken, J. Eur. Cer. Soc., 2022*
Micro-moulding	*Hagelüken, J. Eur. Cer. Soc., 2022*
Volumetric printing	*Kollep, Adv. Eng. Mat., 2022*

Overview of key results (3)

Characterization:	
Strength	*Sasikumar, Ceram. Int., 2018* *Sasikumar, Appl. Mat. Int., 2020* *Hagelüken, J. Eur. Cer. Soc., 2022*
X-ray tomography	*Hagelüken, J. Eur. Cer. Soc., 2022* *Makowska, Acta Mat., 2020* *Konstantinou, Add. Man., 2020* *Kollep, Adv. Eng. Mat., 2022*
Conductivity	*Sasikumar, Ceram. Int., 2018* *Sasikumar, Appl. Mat. Int., 2020* *Hagelüken, J. Eur. Cer. Soc., 2022*
Electro-chemical properties	*Sasikumar, Appl. Mat. Int., 2020* *Jang, Ceram. Int., 2021*
Cytocompatibility	*Sasikumar, Appl. Mat. Int., 2020* *Jang, Ceram. Int., 2021* *Hagelüken, J. Eur. Cer. Soc., 2022*
Microstructure	*Sasikumar, Ceram. Int., 2018* *Sasikumar, Appl. Mat. Int., 2020* *Makowska, Acta Mat., 2020* *Konstantinou, Add. Man., 2020* *Hagelüken, J. Eur. Cer. Soc., 2022* *Kollep, Adv. Eng. Mat., 2022*
Composition	*Makowska, Acta Mat., 2020* *Konstantinou, Add. Man., 2020* *Hagelüken, J. Eur. Cer. Soc., 2022*

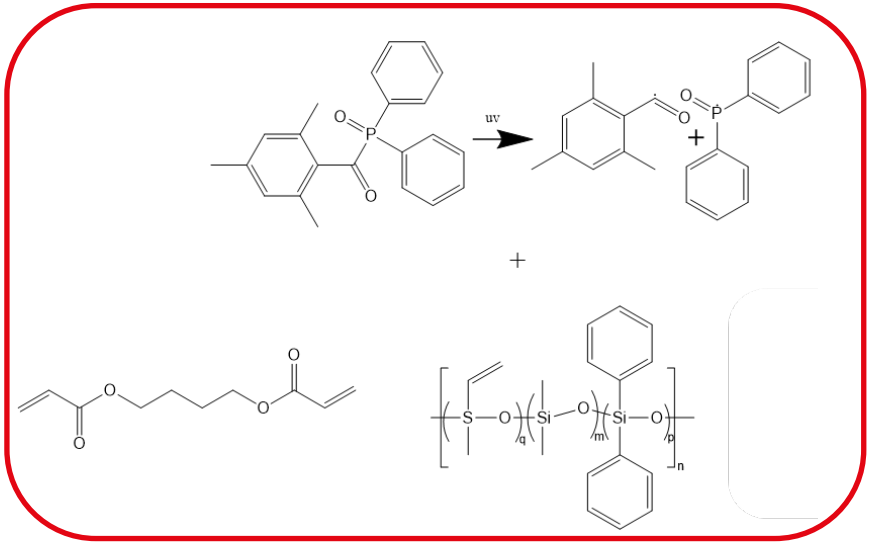
Overview of key results (4)

Application targets confirmed:	
Pacemaker electrode	*Sasikumar, Appl. Mat. Int., 2020* *Jang, Ceram. Int., 2021*
Micronozzle	*Konstantinou, Add. Man., 2020*
Harsh environment compatibility	*Kollep, Adv. Eng. Mat., 2022*
Ceramic QR micro-code	*Hagelüken, J. Eur. Cer. Soc., 2022*
Heterogenous ceramics	*Hagelüken et al. (in prep)*

Fabrication of PDC parts by 3D printing (DLP)

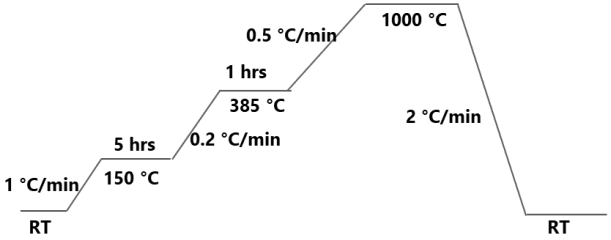
Precursors:
Polyramics SPR-684 from Starfire (SPR)+ 1,4-Butanediol Diacrylate (BDDA) + Genocure LTM

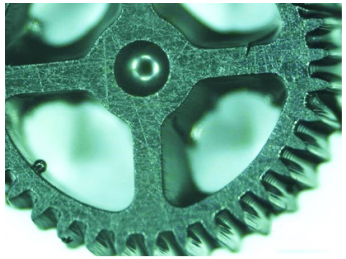
SPR- Methylvinylphenylpolysiloxane



Polymer to acrylate ratio

- ✗ 70:30- High swelling
- ✗ 80:20 - Swelling
- ✓ 85:15
- ✓ 90:10

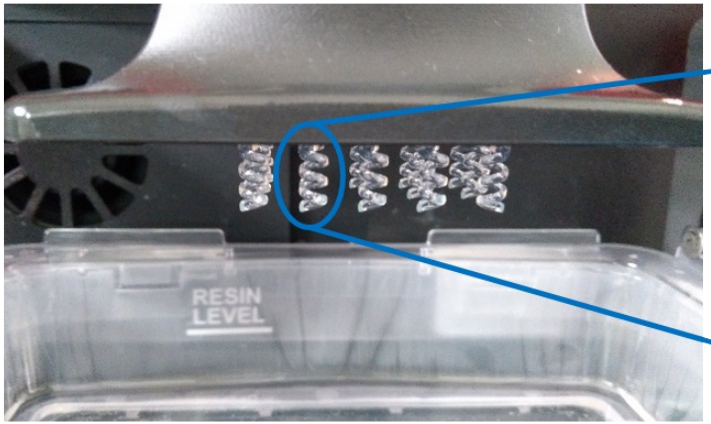




SiOC parts after pyrolysis

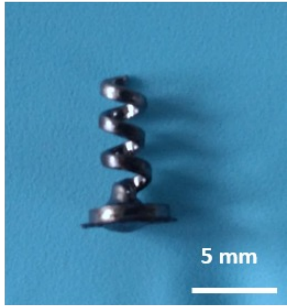
Fabrication of PDC electrodes by 3D printing

DLP Printer



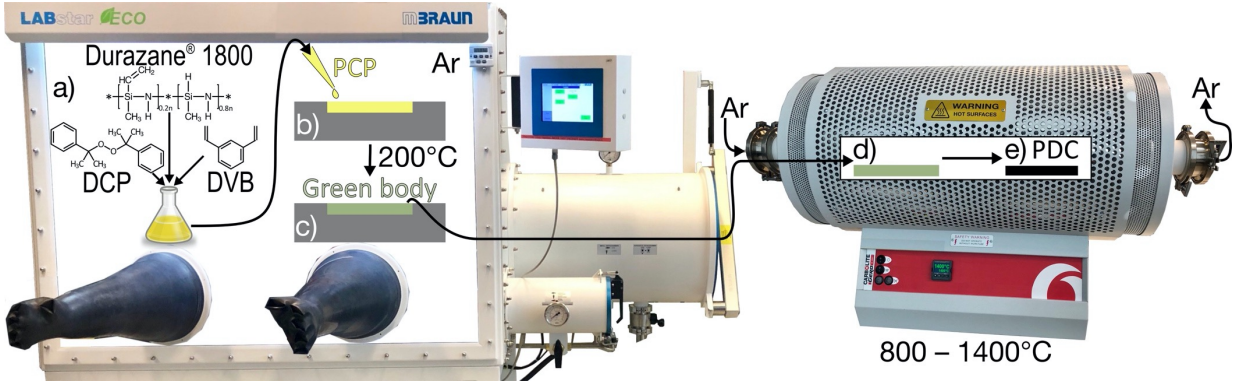
Preceramic part

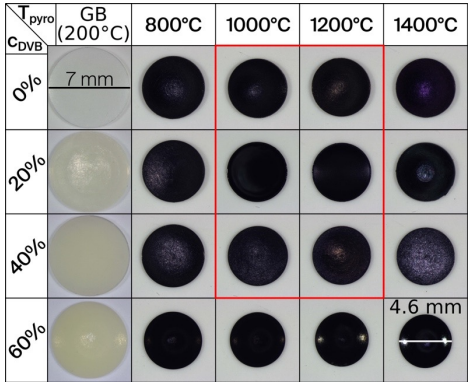
1000 °C

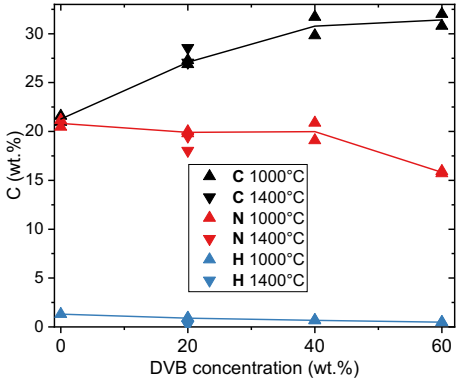


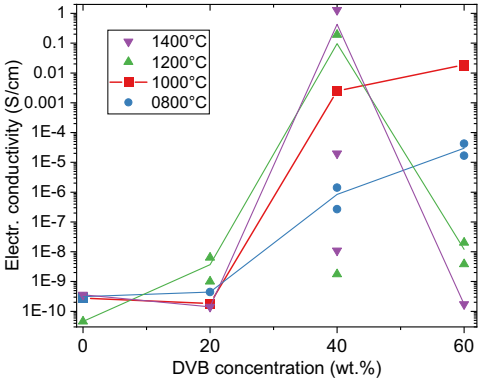
SiOC glass ceramics

Silicon Carbonitride Property Tuning



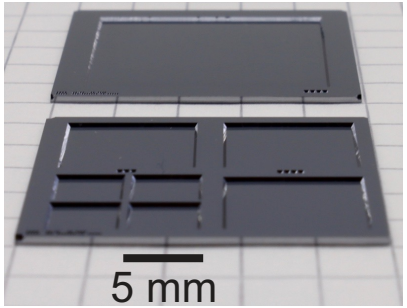




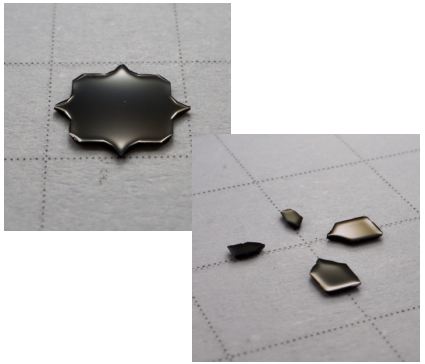


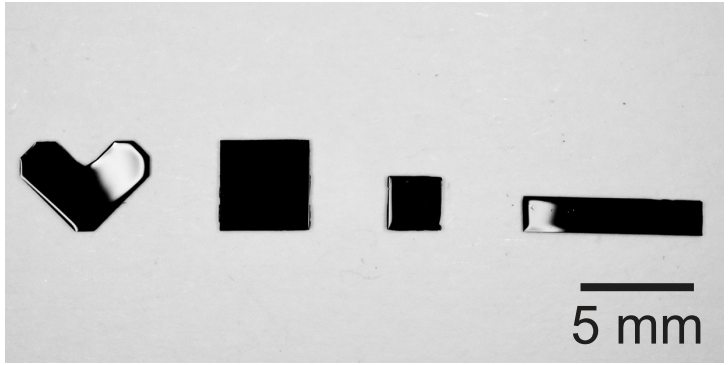
- Variation of precursor composition and pyrolysis temperature
- Pressureless thermal processing in Ar
- Addition of organic filler allows increase of PDCs' carbon content
- Flexural strength up to 1700 MPa
- Jump in electrical conductivity from 20 to 40% filler concentration

Multi-scale micromoulding

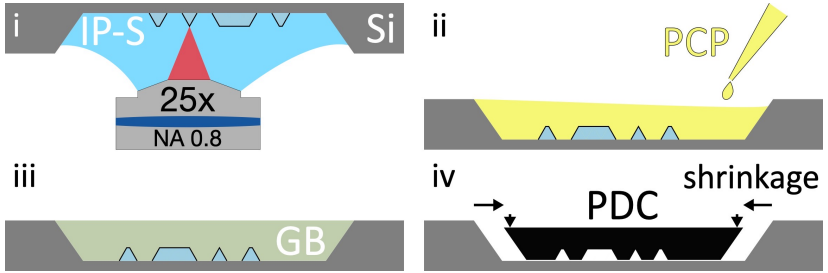


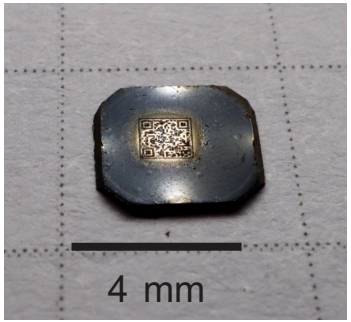
KOH-etched Si mold chips

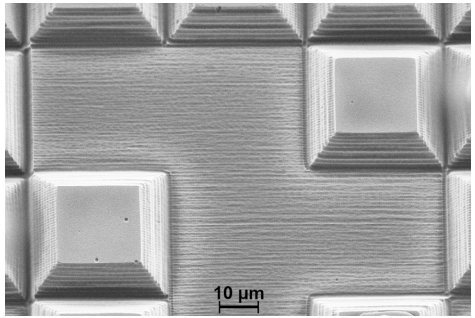




Examples of 250 µm thick SiCN structures



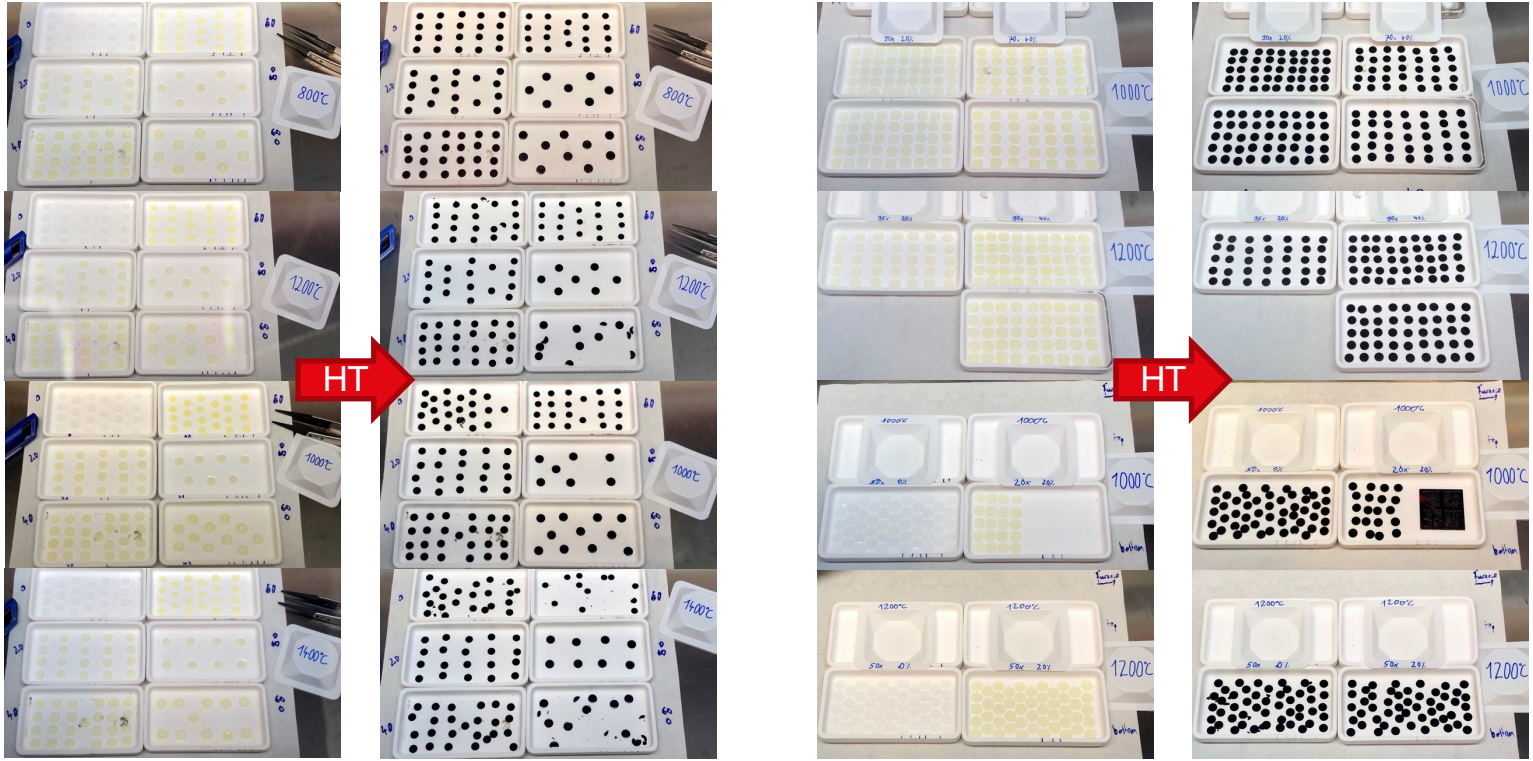




Imprinting of micro QR code in PDC achieving submicrometer features

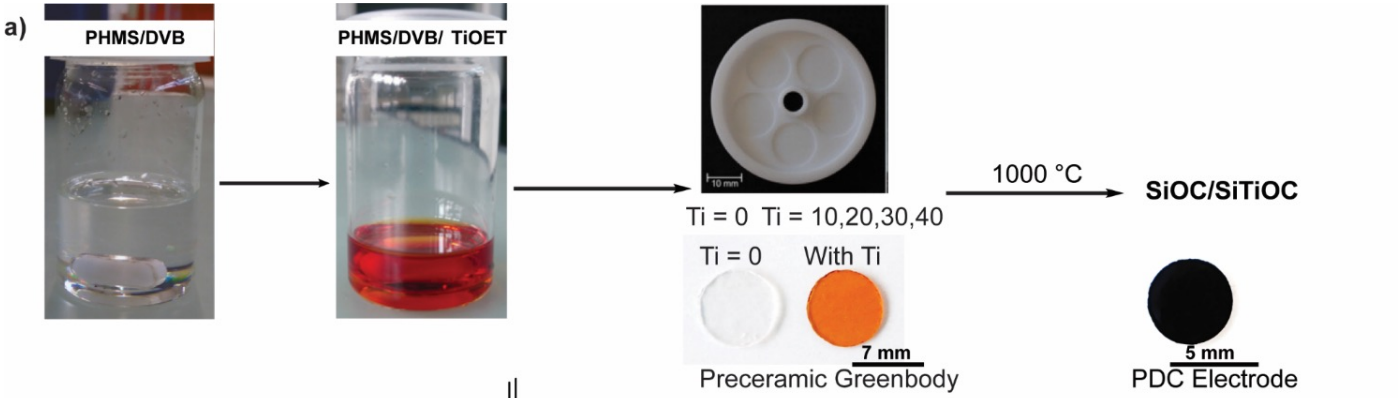
Systematic parameter screening for SiCN micropart fabrication

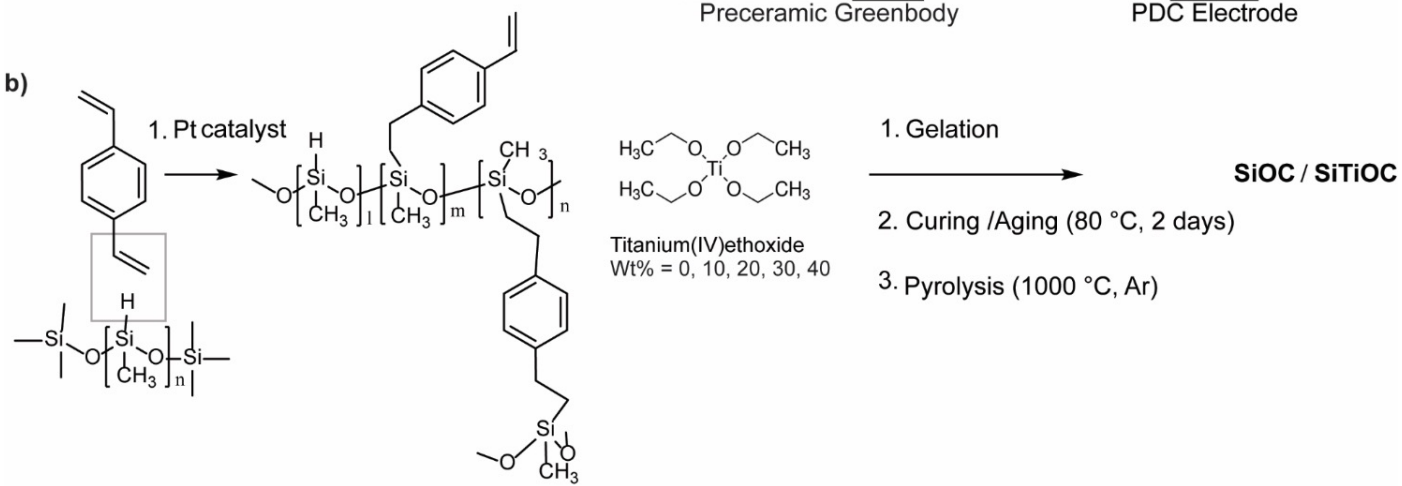
>> 1000 SiCN samples fabricated to this day



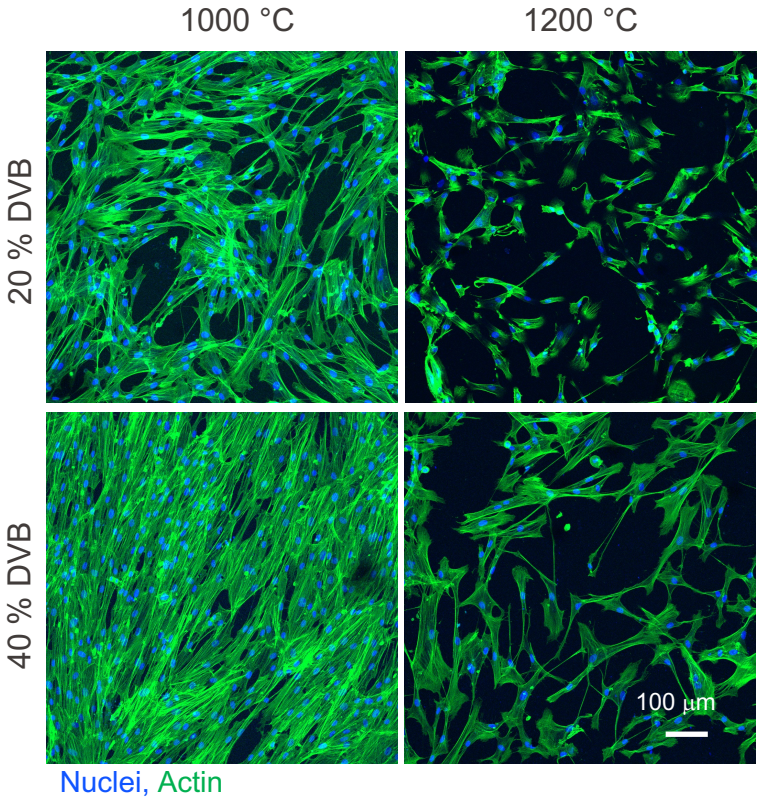
Green body | PDC | Green body | PDC

Fabrication of PDC electrodes for pacemaker studies

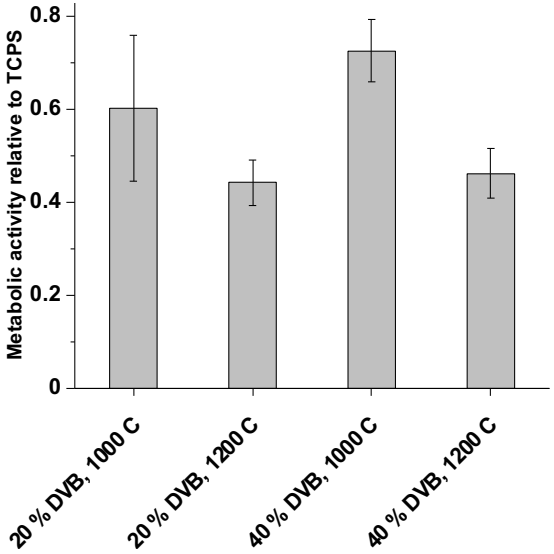




Cytocompatibility of PDCs doped with DVB

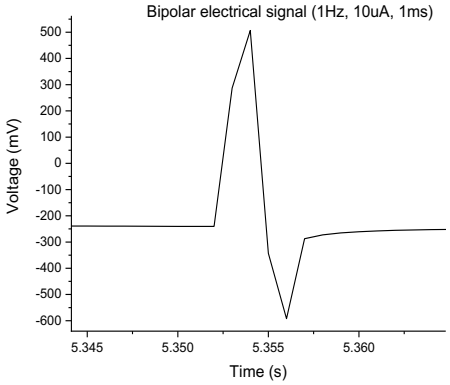


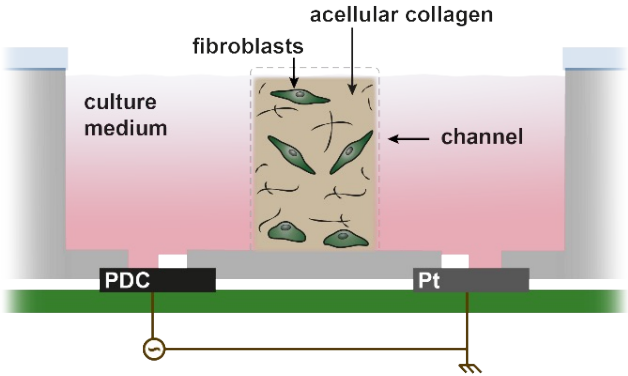
Fibroblasts cultivated on the surface of PDCs



→ **Higher pyrolysis temperature is detrimental to cell growth at day 4**

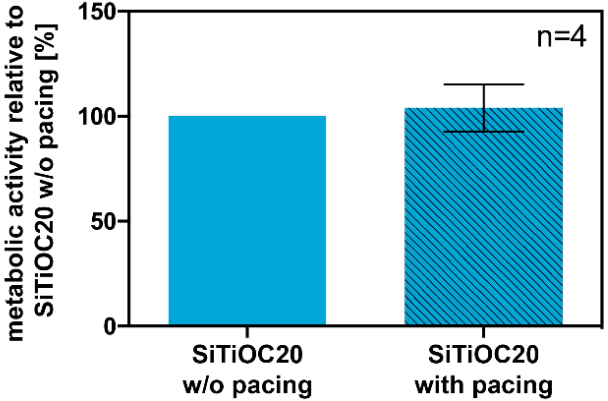
PDCs as pacemaker electrodes

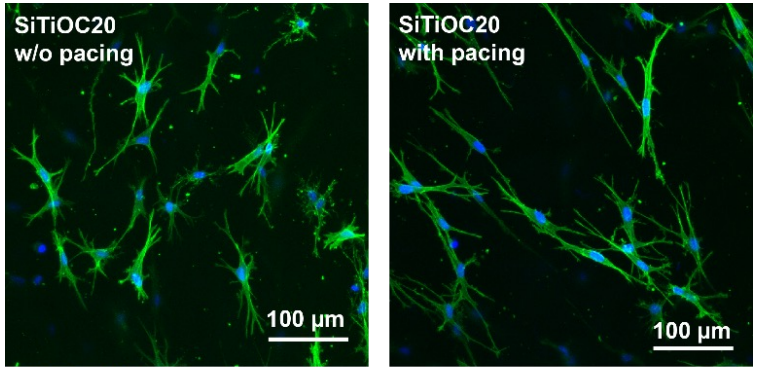




Setup: fibroblasts encapsulated in collagen hydrogels placed in between electrodes

Pacing conditions: Within 48 hours, four times pacing for 30min with symmetric bipolar electrical pulses (10µA, 1ms, 1Hz)





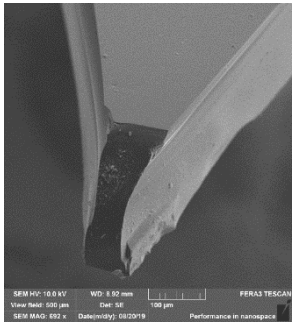
nuclei actin

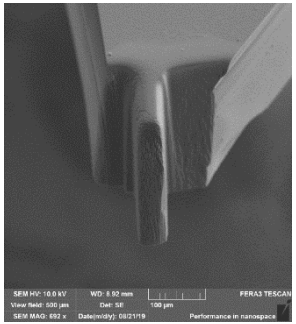
→ **no differences were observed between paced and non-paced samples**

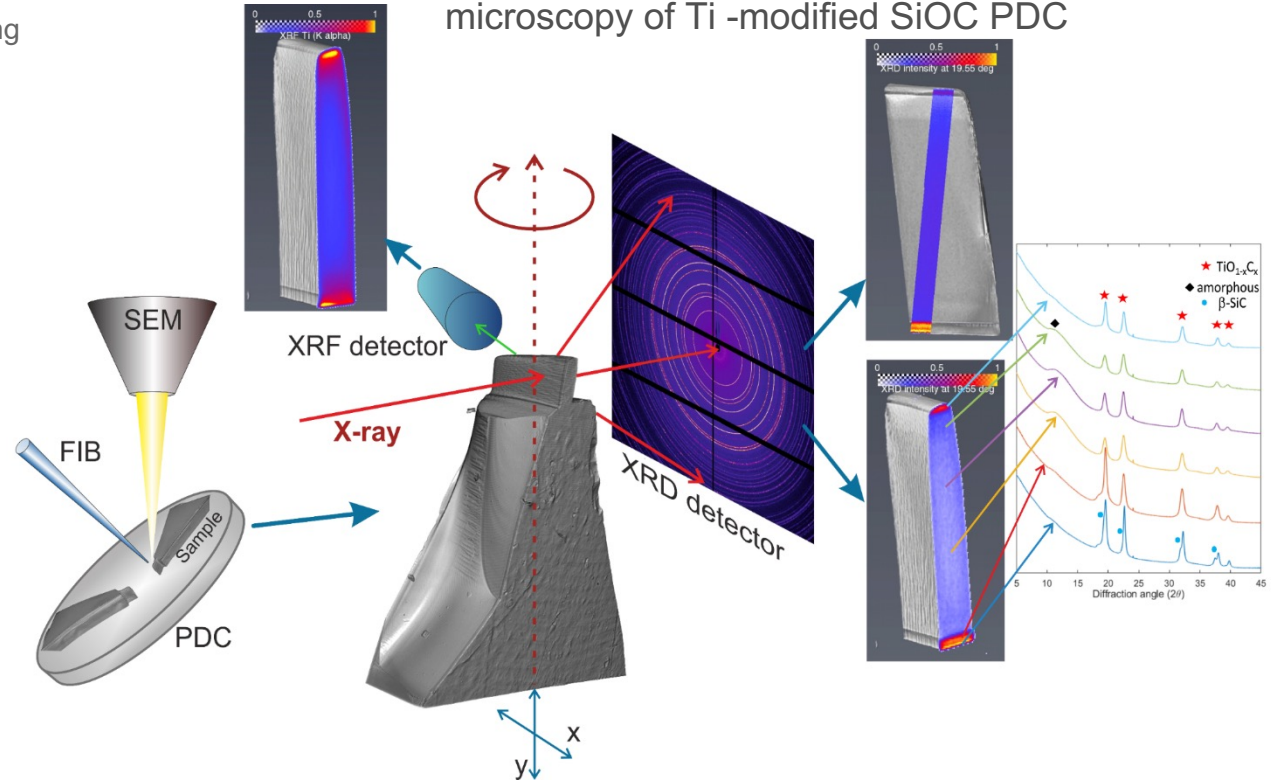
Structural studies at PSI (SLS synchronoton)

XRF-, XRD- and absorption- contrast tomographic microscopy of Ti -modified SiOC PDC

Sample preparation using FIB (@ ScopeM)

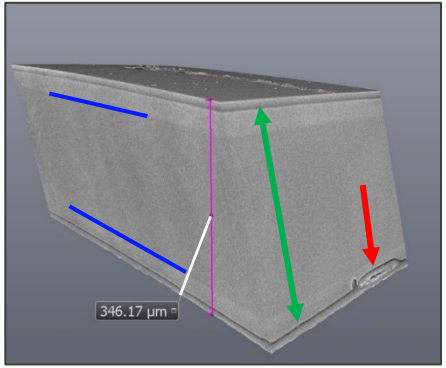


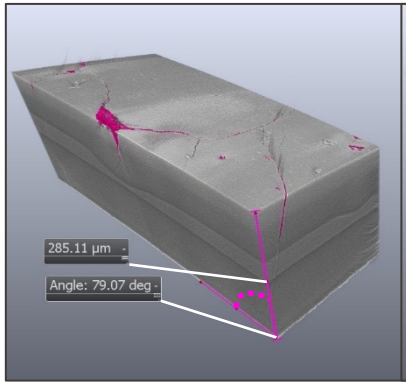


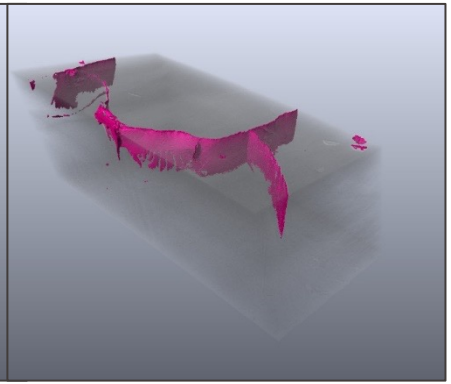


Structural studies at PSI (SLS synchrotron)

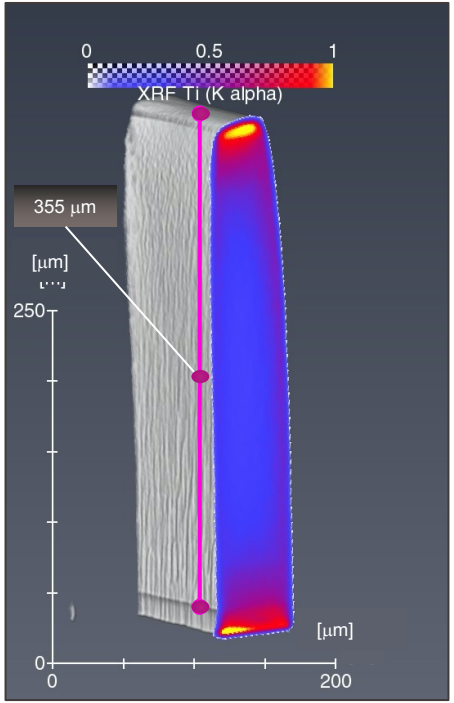
Features observed in full field tomography



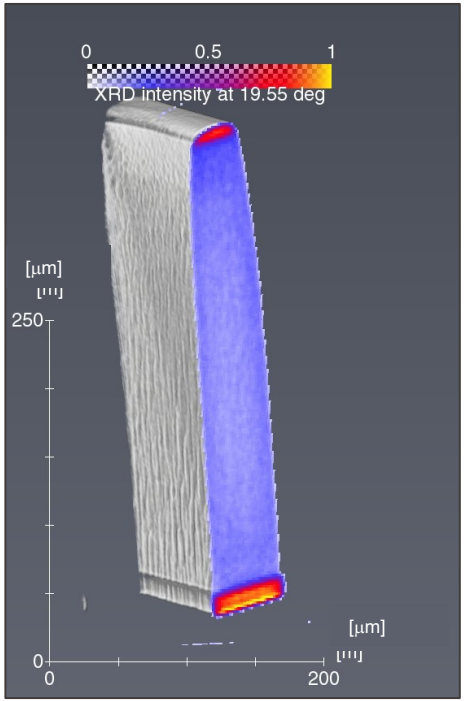




XRF mapping
(Ti Ka radiation intensitiy)



XRD mapping
($TiO_{1-x}C_x$ phase peak intensitiy)



M. Makowska, P.V.W. Sasikumar, L. Hagelüken, D.F. Sanchez, N. Casati, F. Marone, G. Blugan, J. Brugger, H. Van Swygenhoven, **Acta Mater**. 198 (**2020**) 134–144.

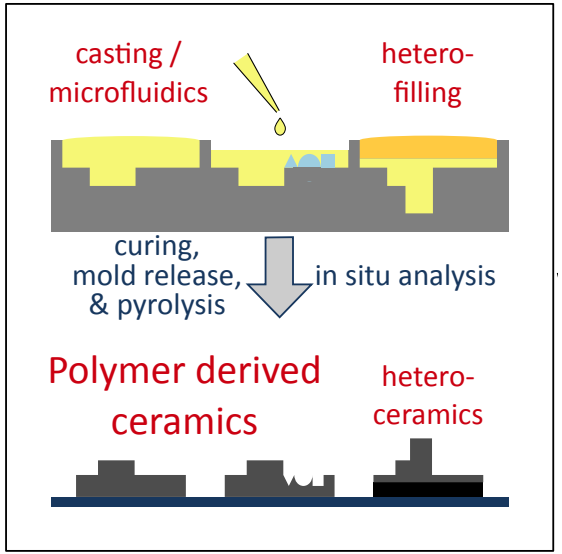
Seamless integration of heterogenous micro-ceramic parts

Conducting and non-conducting parts

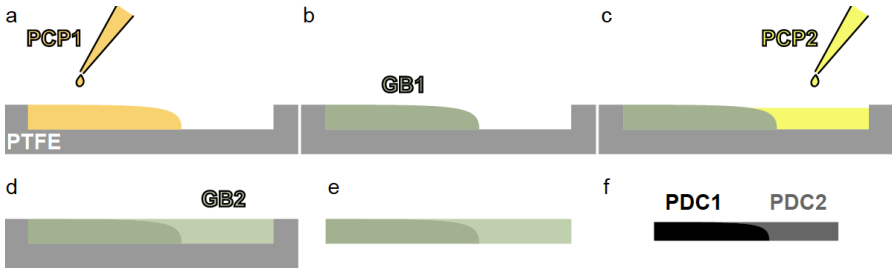
Curiosity driven: how sharp is the interface?

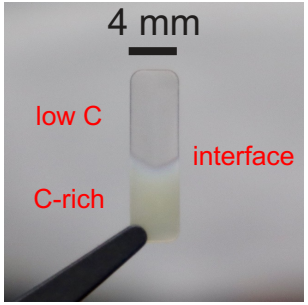
Application driven: ceramic wall electrically conducting feedthrough

Mold filling

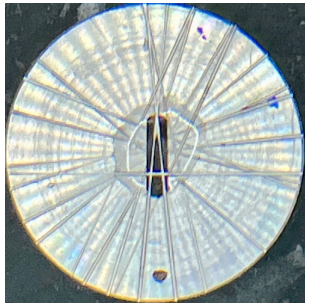


Heteroceramics

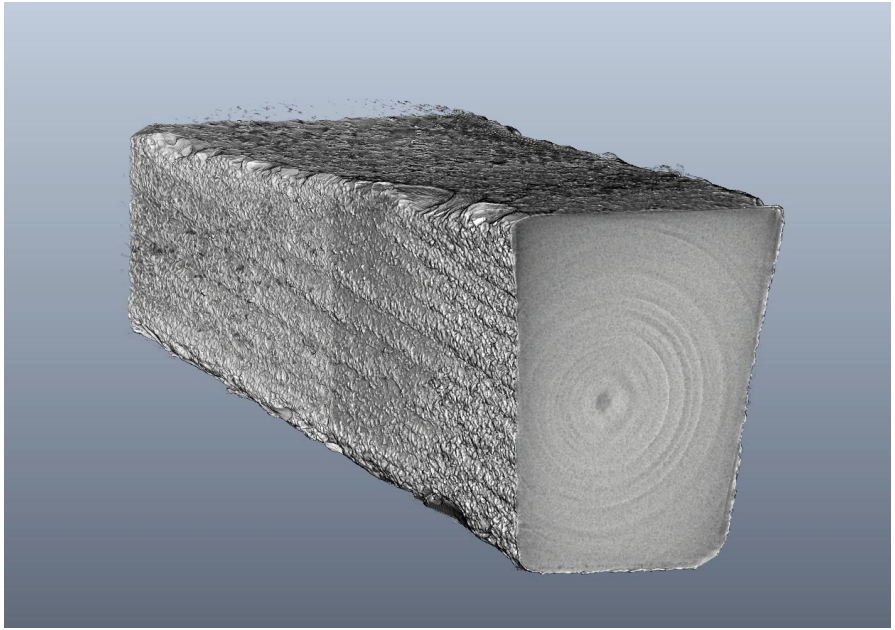




Lateral hetero-GB

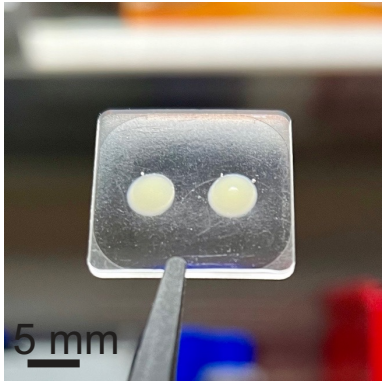


Cutting of PDC

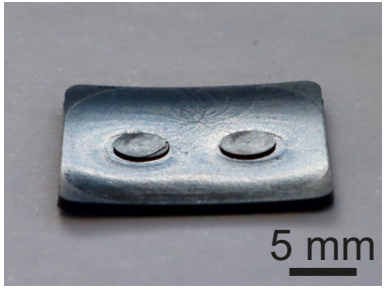


Hagelüken et al. (in prep)

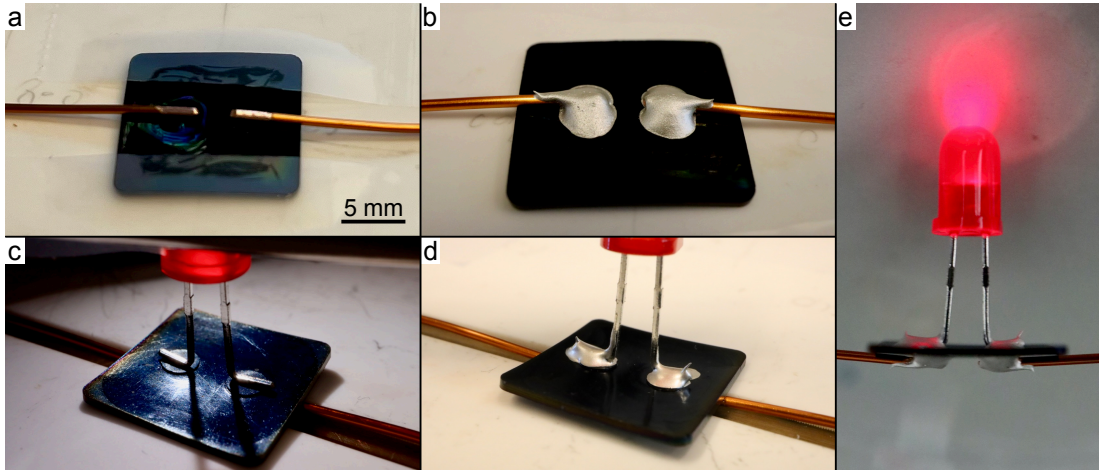
Power supply through hetero-ceramics



Bi-feedthrough GB



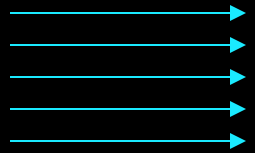
BFT hetero-ceramic



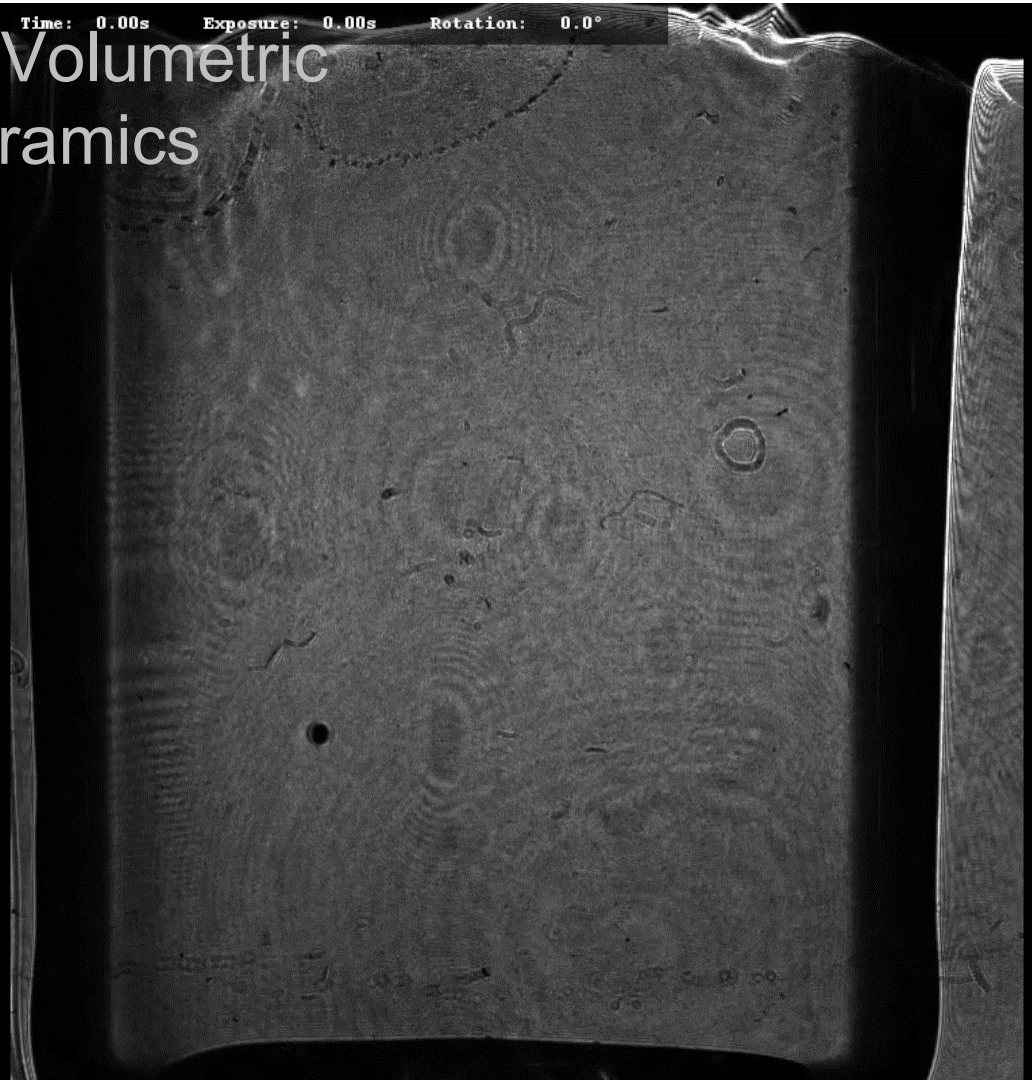
Assembly process

Hagelüken et al. (in prep)

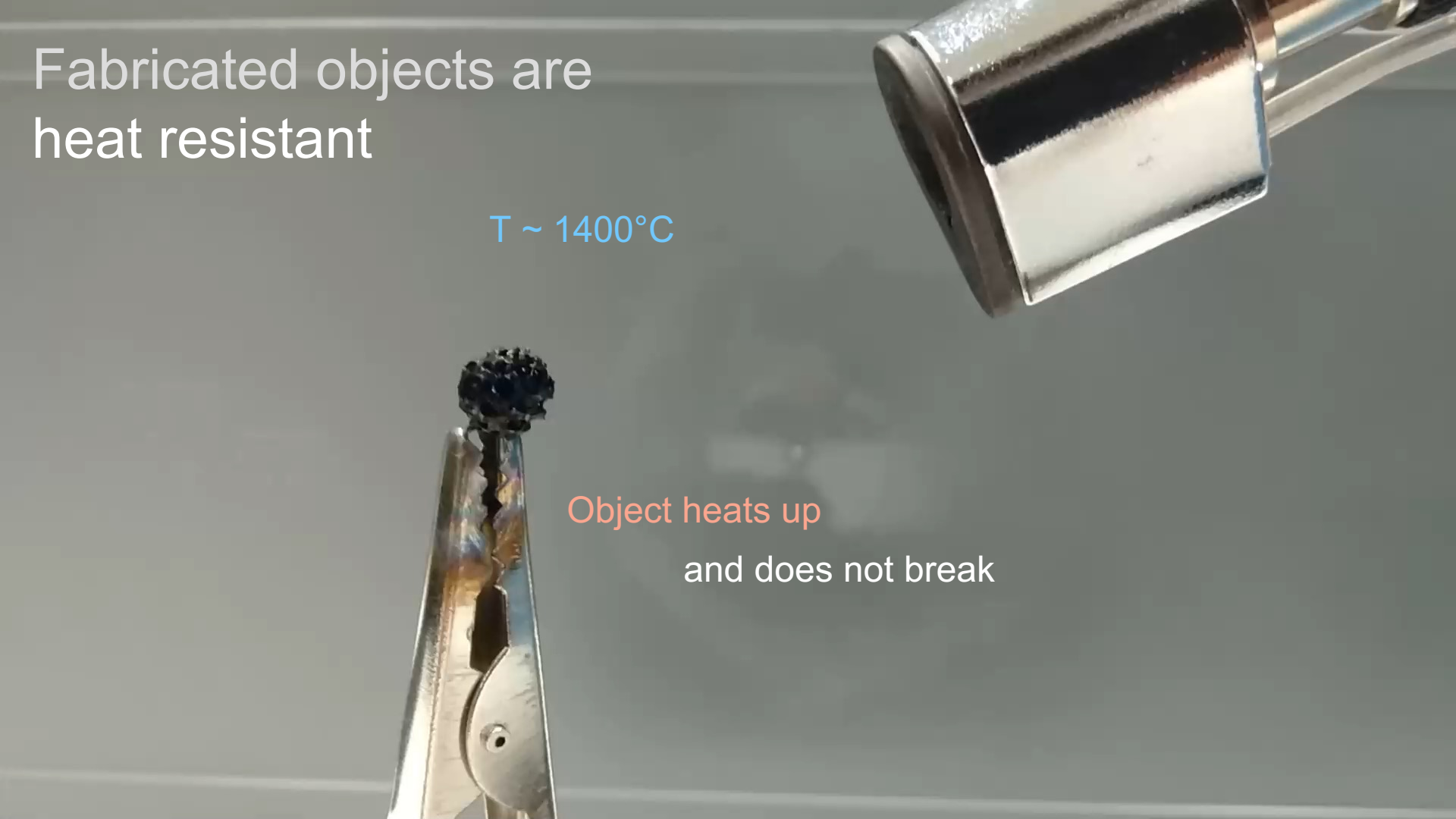
Tomographic Volumetric Printing of Ceramics



Tomographic light patterns



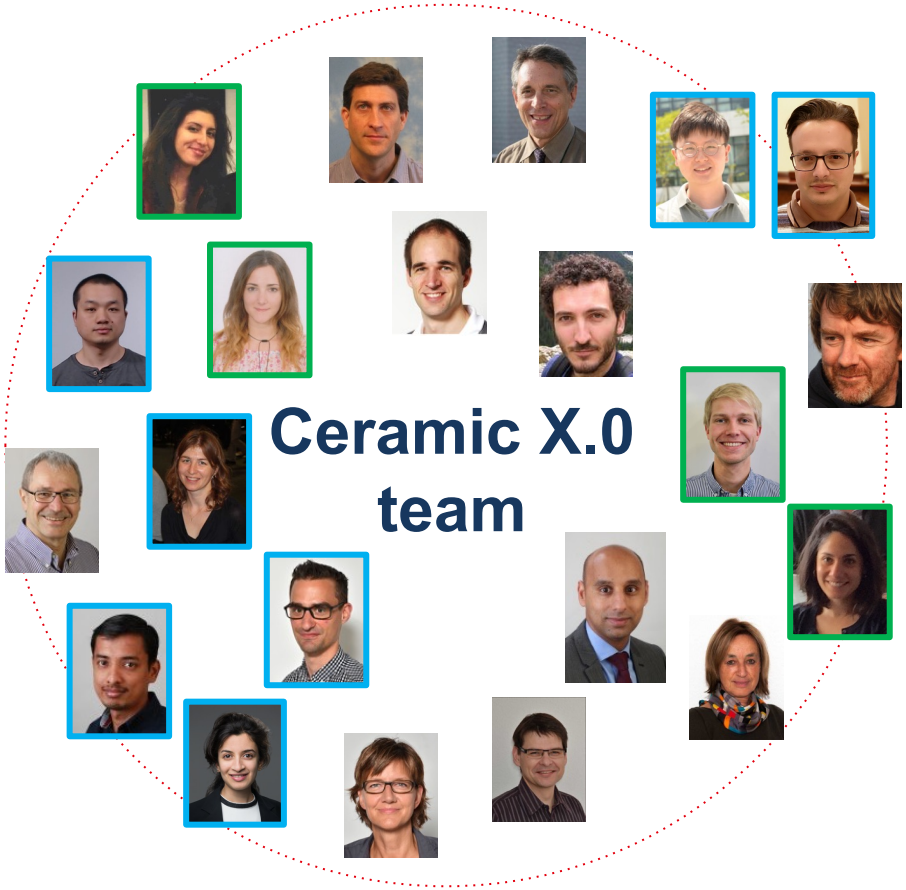
Fabricated objects are heat resistant



Key results to take away

- Liquid PCP can be modified with conducting filler
- Microshaping properties remain intact
- Various shaping methods benchmarked (3D printing, moulding…)
- PDC properties systematically characterized (tomography,…)
- Cyto-compatibility validated for possible implant application
- Electrical pacing of cells confirmed
- New volumetric 3D printing developed
- New heterogenous PDC for seamless integration prototyped

- Consortium is ready for Ceramic X.1 ☺



Thank you…

…to all team members

… to SFA for funding

… to you for listening