

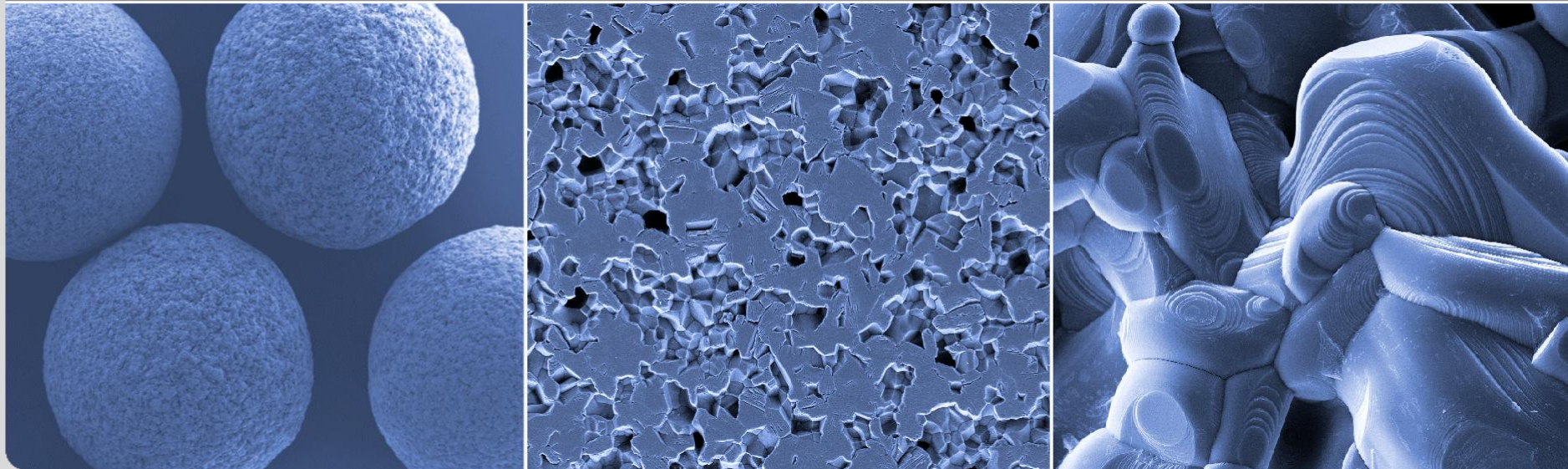
# Long-term annealing of advanced lithium metatitanate breeder pebbles

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# Broader Approach DEMO



Agreement on a privileged partnership between Japan and EURATOM in support of ITER and an early realization of fusion energy

3 projects

IFMIF/EVEDA

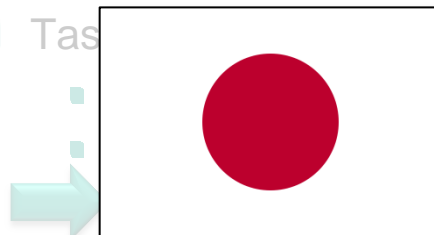
STP (JT60/SA)

T5-3: Characterization of pebbles

- Study on the stability of advanced  $\text{Li}_2\text{TiO}_3$  (JA) and  $\text{Li}_4\text{SiO}_4$  (EU) pebbles during exposure to purge gas atmospheres
- Chemical and micro-structural characterization of exposed pebbles

Task 1..4

Task



T5-4: Reprocessing of breeder material

# Contents



## Experimental

- Annealing setup
- Experimental conditions

## Results

- Porosity & density
- Microstructure
- Chemical composition & impurities
- Appearance
- Mechanical strength

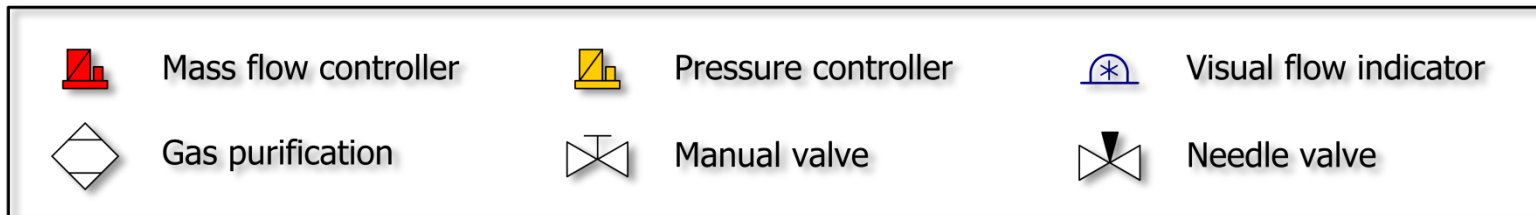
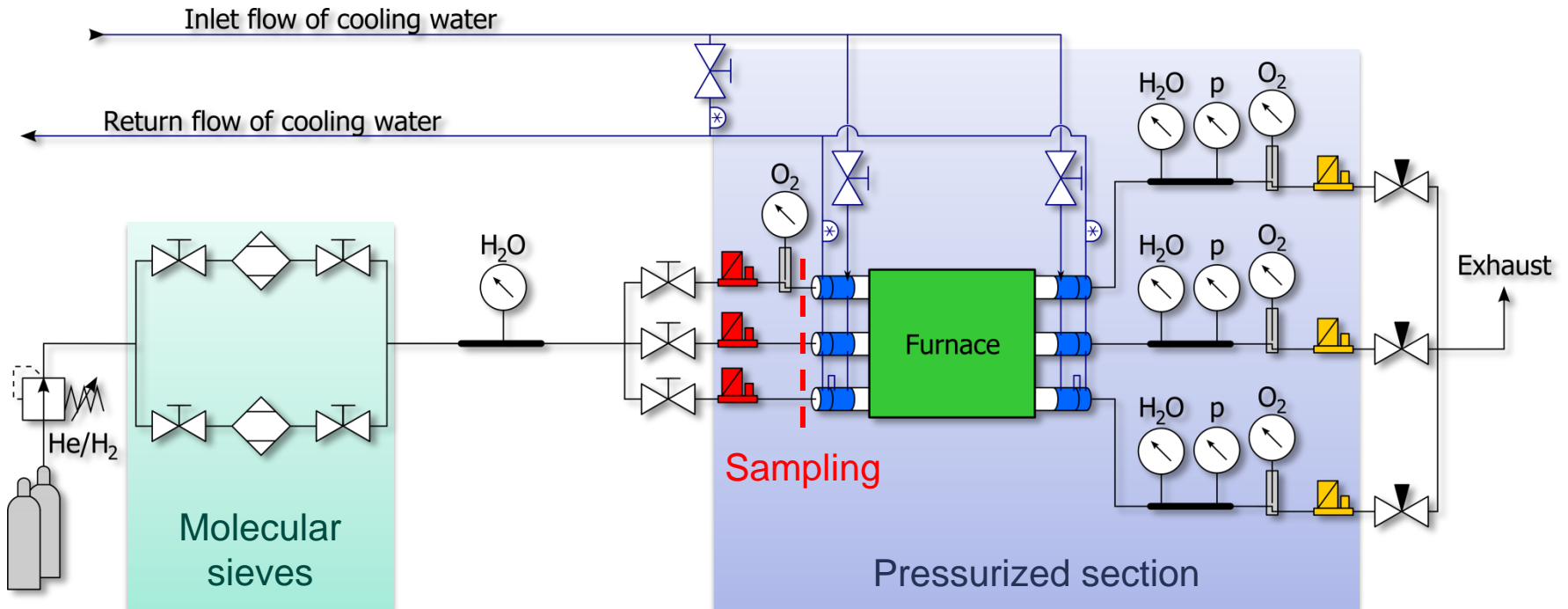
## Conclusions



Annealing setup & conditions

# EXPERIMENTAL

# Experimental setup



# Experimental conditions

## ▀ Samples

- Emulsion method pebbles
- Sol-gel method pebbles



## ▀ Annealing temperature:

900° C

## ▀ Annealing for:

4, 32, 64, 128 days

- Temperature during sampling:
- Heating/cooling rate:
- The period of sampling is not considered as annealing time

300° C

5 K/min

## ▀ Atmosphere:

He with 0.1 vol.% H<sub>2</sub>

- Pressure:
- Flow rate:

1200 mbar, absolute

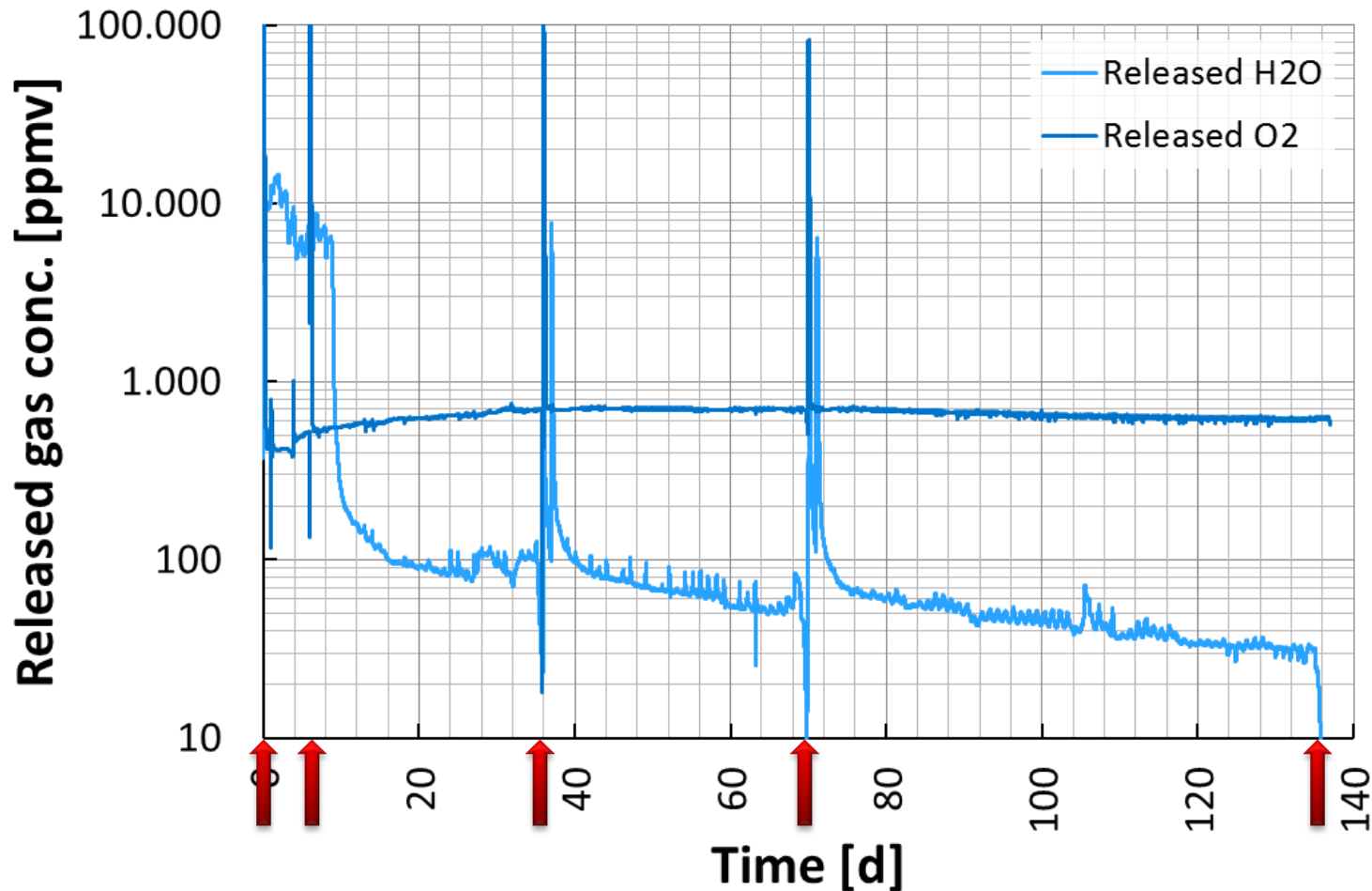
1.2 l/h (reference: 8000 l/h)

## ▀ Sample preparation:

1 hour at 300° C in vacuum

# Annealing atmosphere

## Released gases during the experiment





Morphological, mechanical and chemical characterization

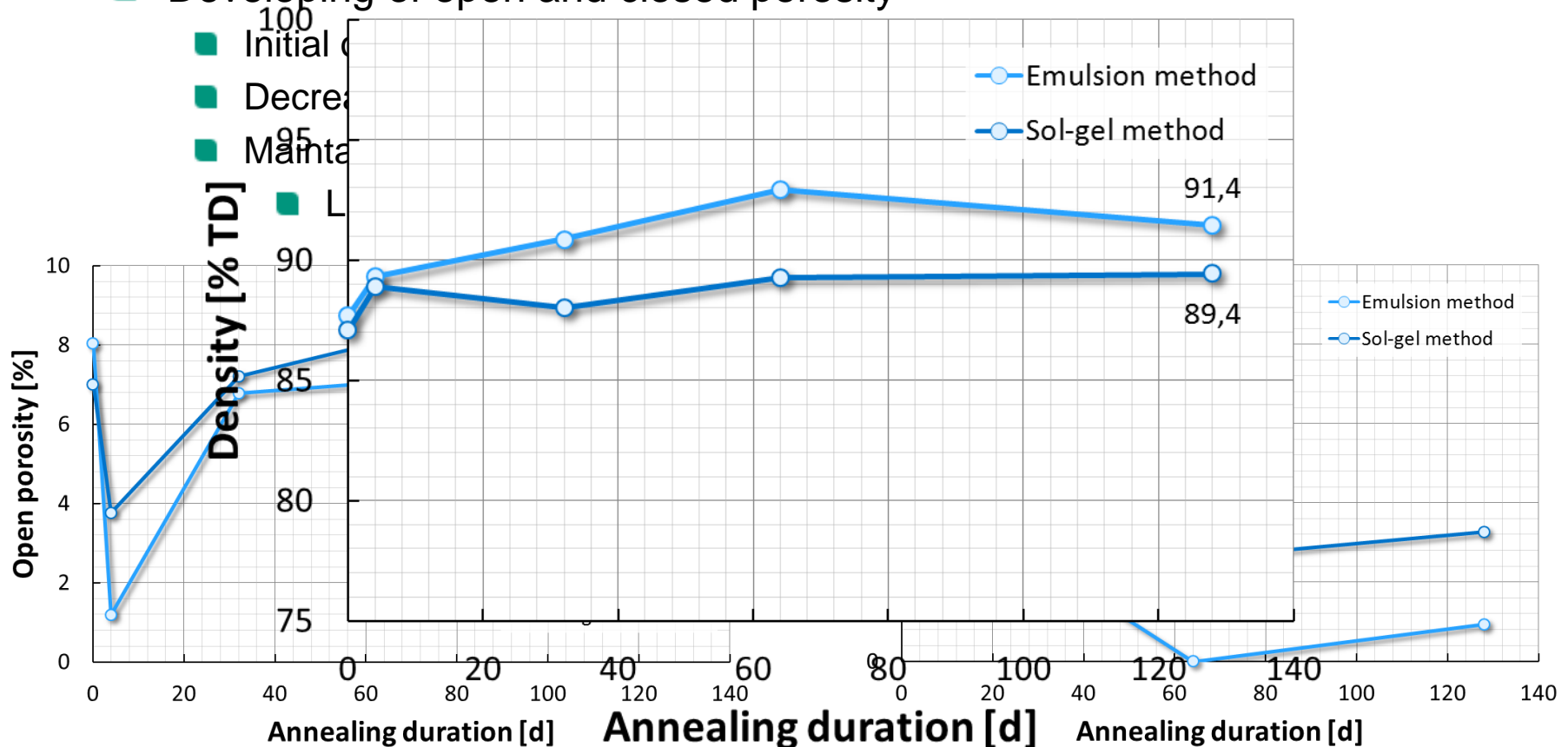
# RESULTS



# Porosity

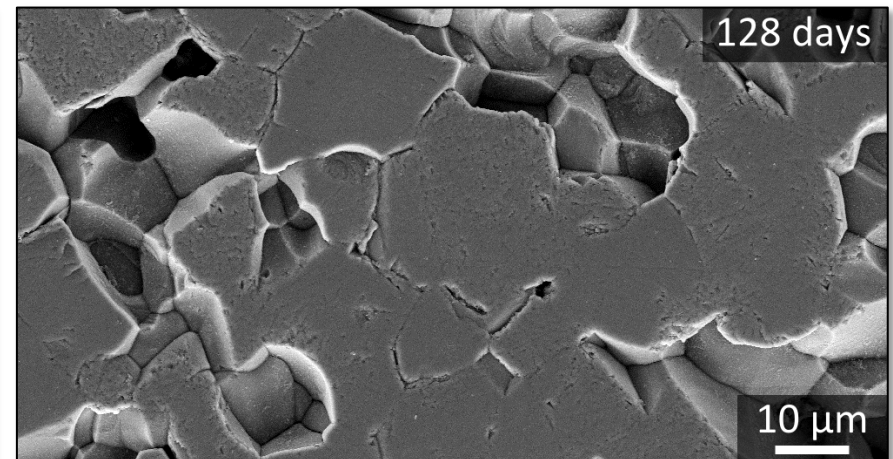
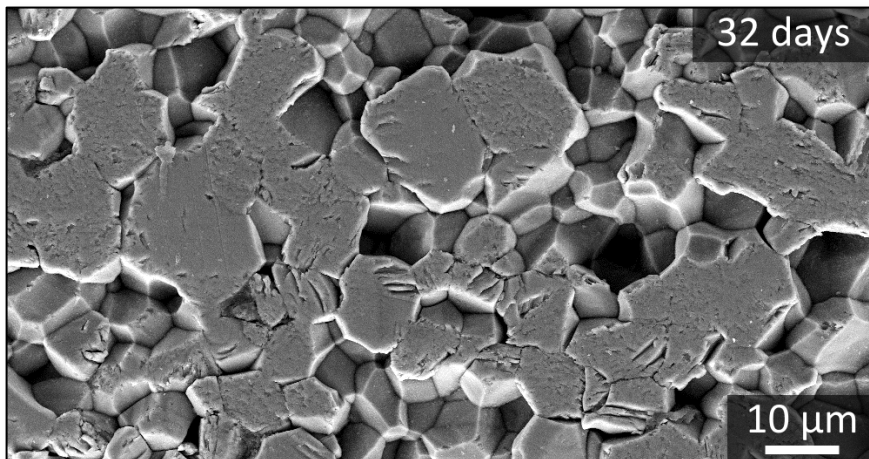
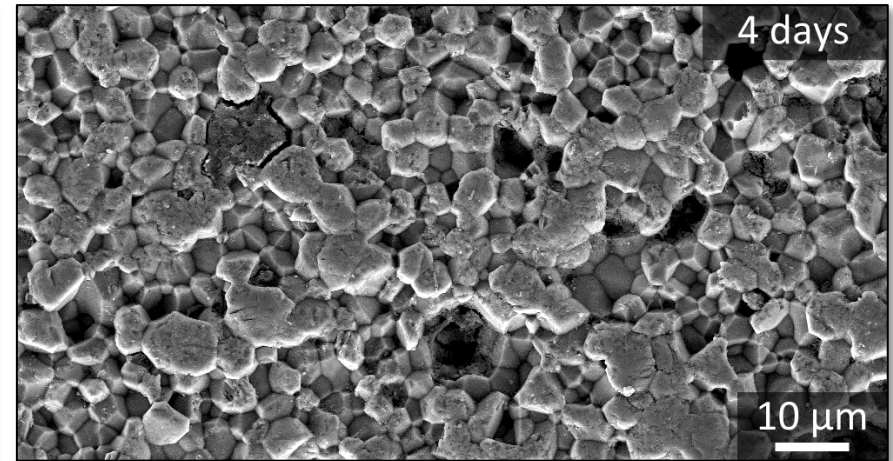
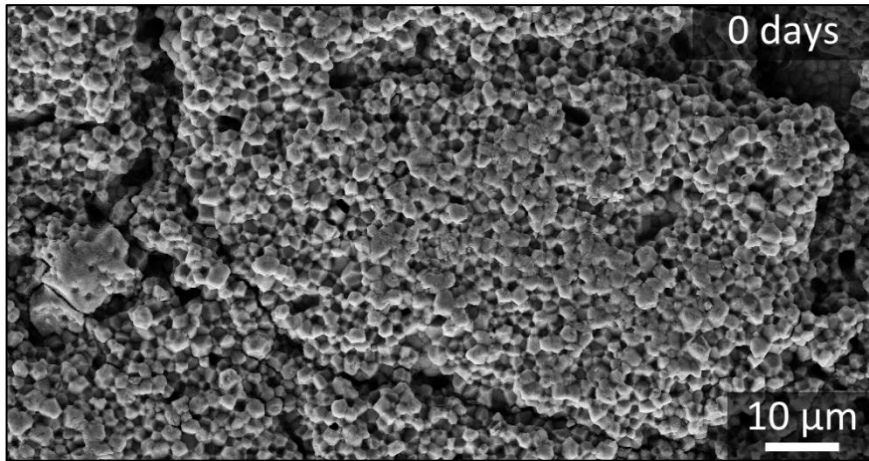
▀ Mild increase in density over annealing time

- Post-sintering densification
- Developing of open and closed porosity



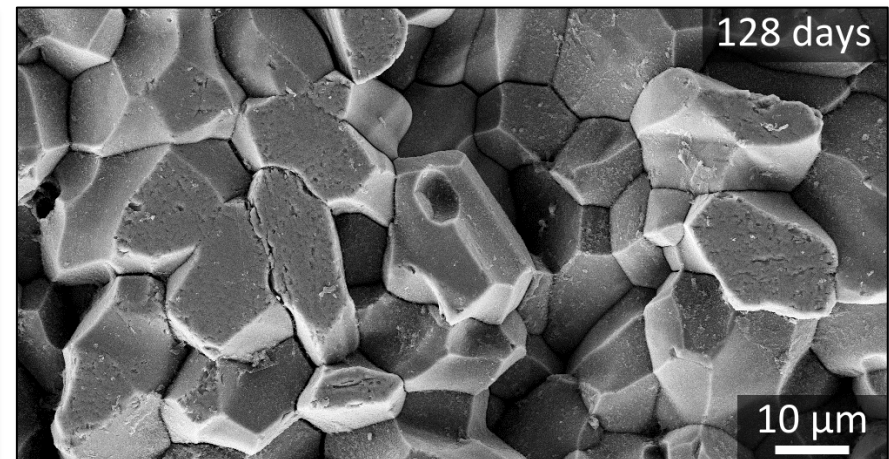
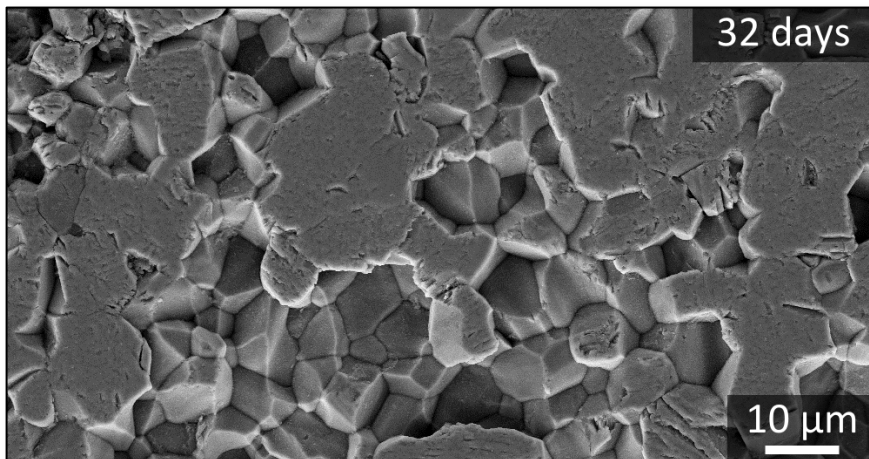
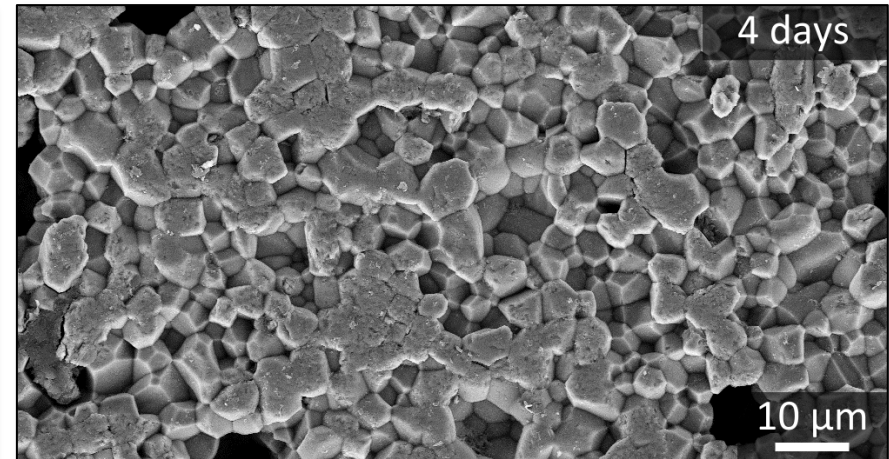
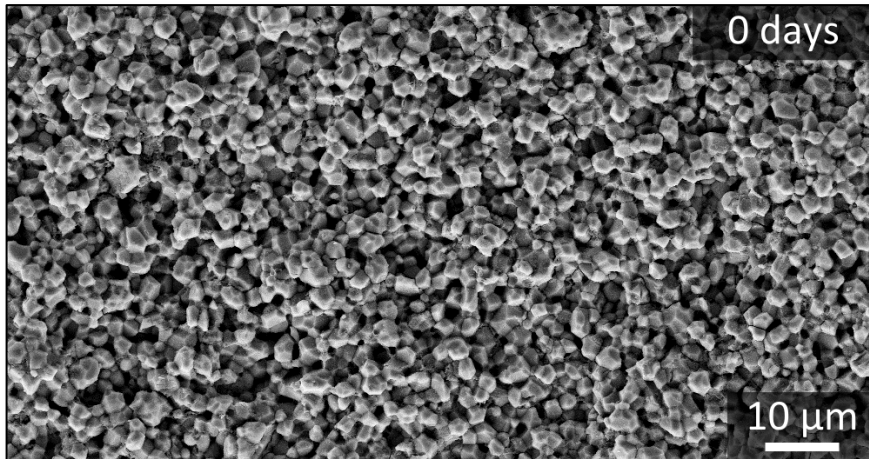
# Microstructure

## Emulsion method pebbles



# Microstructure

## ▀ Sol-gel method pebbles



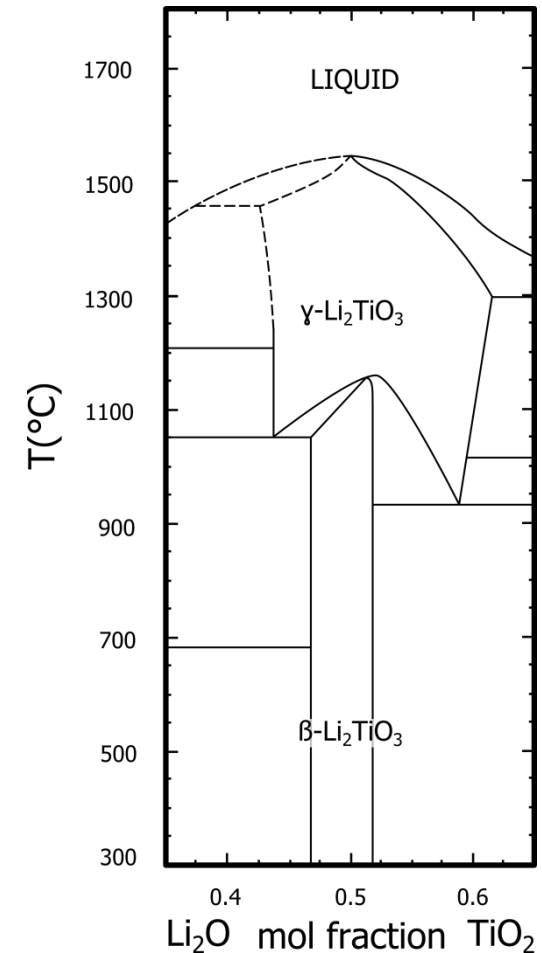
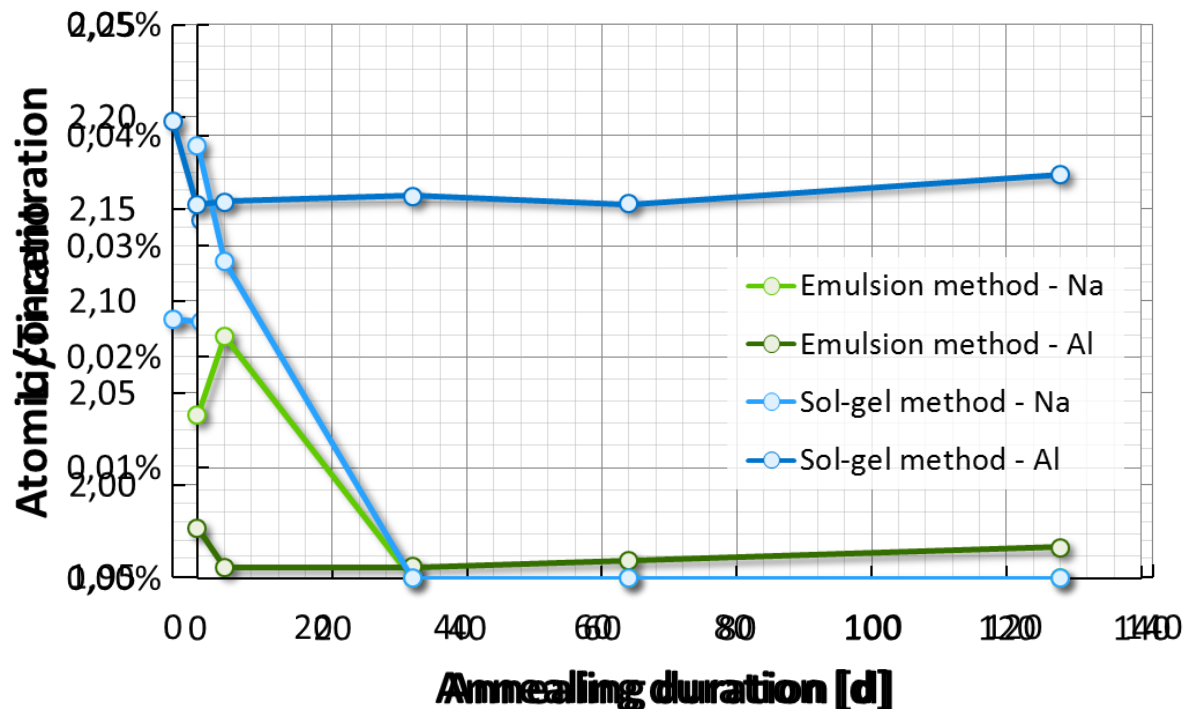
# Chemical & phase analysis

## ▀ Vaporization of lithium compounds

- Li/Ti-ratio equally reduced
- Equilibrium phase after 128 days:  $\beta$ - $\text{Li}_2\text{TiO}_3$

## ▀ Low level of impurities in general

- Rapid decline of sodium levels



Reproduced from Mergos et al. (2009) Materials Characterization 60 (8)

# Appearance

0 days

4 days

32 days

64 days

128 days

96 days  
EU LMT  
2003

Emulsion method

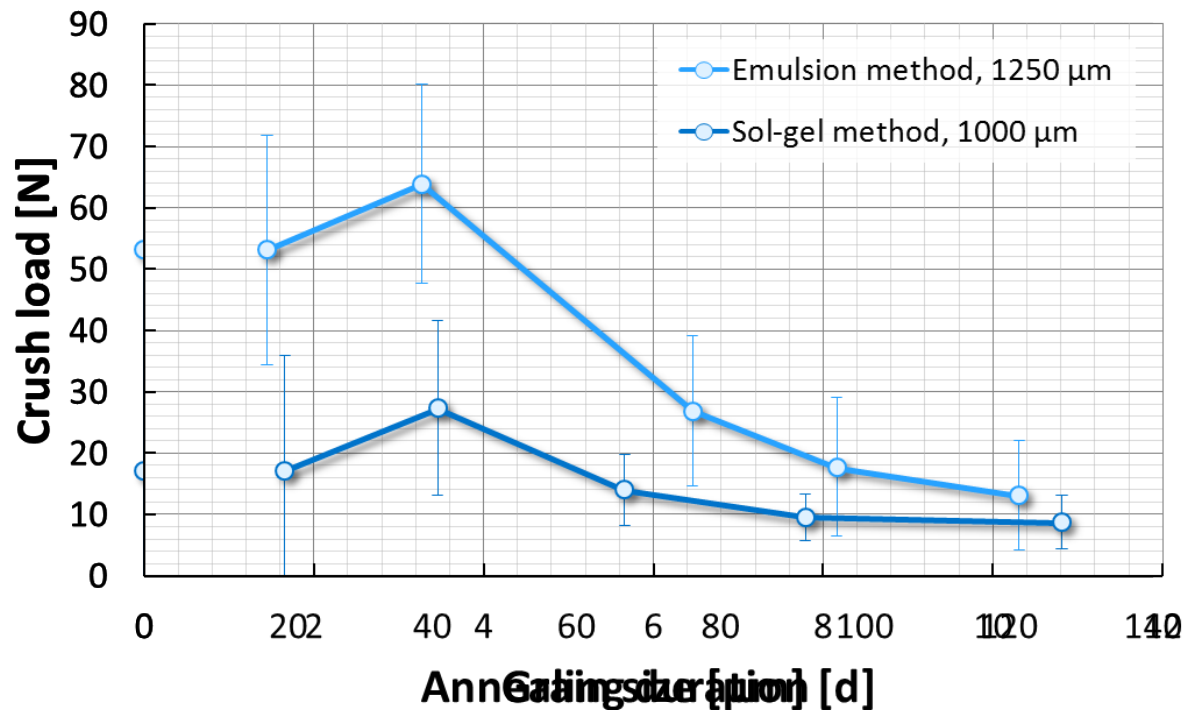


Sol-gel method



# Mechanical properties

- Very high initial rigidity
- Significant decrease of crush load with continued annealing
  - Commonly observed grain size dependence for non-cubic ceramics
    - Usually explained by thermal expansion anisotropy
    - Comparably small optimum grain size hints at high anisotropy



# Conclusions



- 🌿 Long-term annealing for a maximum of 128 days
  - Continuous monitoring of process values
  - Reasonable mimicking of the blanket conditions
  - Continuous release of gaseous species
  
- 🌿 Advanced lithium metatitanate breeder pebbles withstand these conditions well over 128 days of annealing
  - Grain size of the pebbles increases significantly
  - Single phase  $\beta$ -Li<sub>2</sub>TiO<sub>3</sub> pebbles are maintained
    - Almost linear loss of lithium might be an issue in the very long-term
  - Reasonable crush load values after 128 days of annealing
    - Strong dependence of the crush load on the grain size
    - Optimum grain size for maximum rigidity: about 4  $\mu$ m
    - Limiting grain growth as a potential future target
  - Fabrication method does not impact the long-term behavior of the pebbles

# Thank you for your attention

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