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Experimental Study on Tritium Release from Li_2TiO_3 Pebbles as Tritium Breeder through International Collaboration between Korea and China

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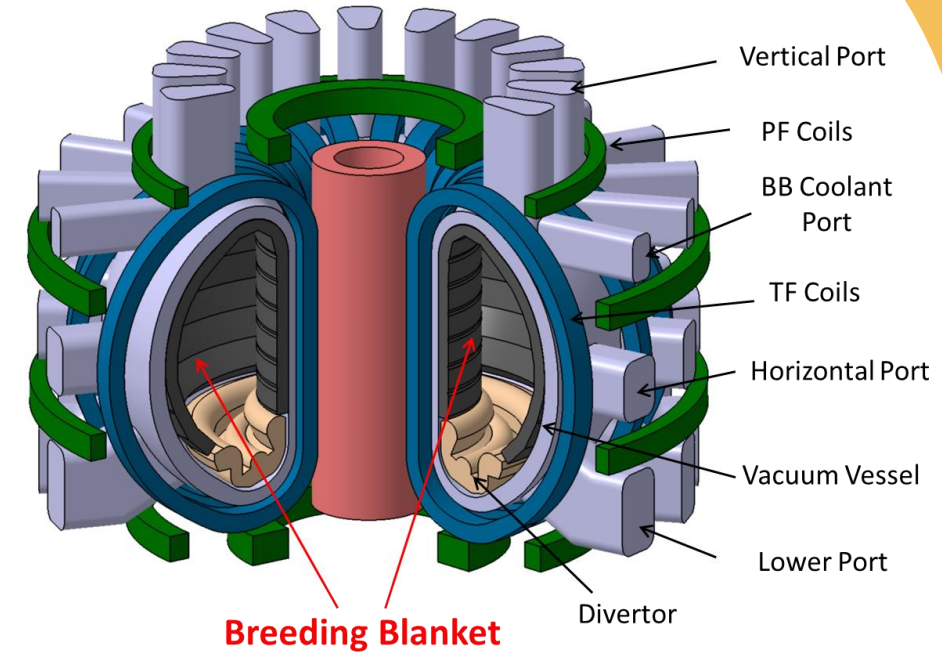
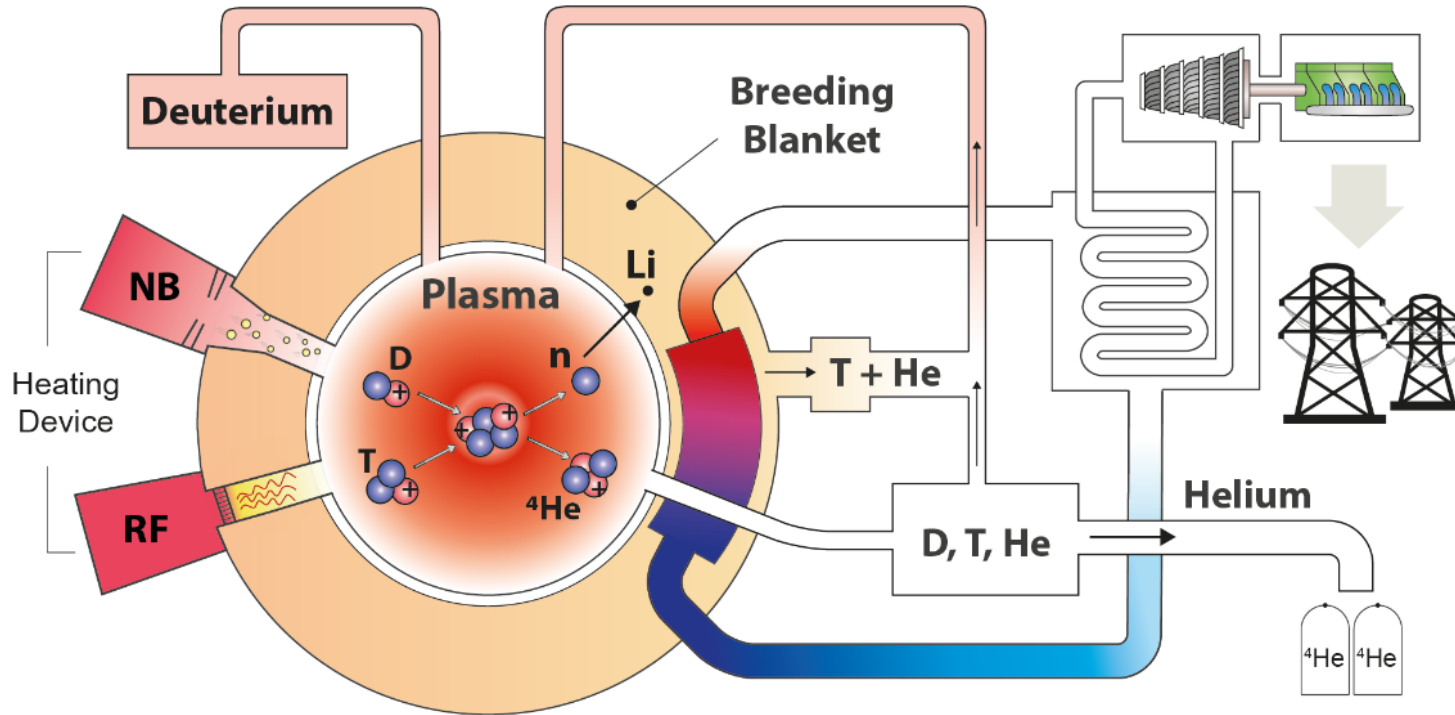
01

Background



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Concept of Fusion Power Plant and Breeding Blanket



- in Fusion Core :
 $D + T \rightarrow 4\text{He} + n + 17.6\text{ MeV}$

- in Breeding Blanket :
 ${}^6\text{Li} + n \rightarrow {}^4\text{He} + T + 4.8\text{ MeV}$
 ${}^7\text{Li} + n \rightarrow {}^4\text{He} + T + n' - 2.5\text{ MeV}$
 ${}^9\text{Be} + n \rightarrow {}^8\text{Be} + 2n - 2.5\text{ MeV}$

- Functions of Breeding Blanket

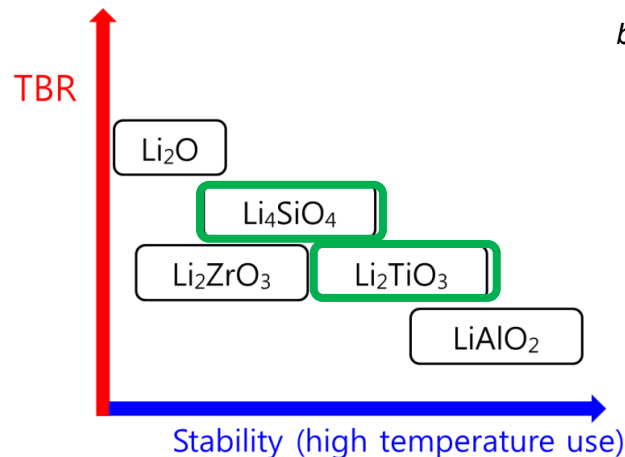
- **Tritium Breeding** to ensure fuel self-sufficiency
- **Heat Conversion and Extraction** for electricity generation
- Neutron and Gamma-ray **Shielding**

Korean Breeding Blanket Concepts and Materials

Parameters	HCCR ITER TBM	HCCP ITER TBM	HCCR DEMO	HCCP DEMO
FW Heat Flux	0.3 MW/m ²	0.3 MW/m ²	0.5 MW/m ²	0.5 MW/m ²
Neutron Wall Loading	0.78 MW/m ²	0.78 MW/m ²	1.5 MW/m ²	1.5 MW/m ²
Armor Material (Plasma Facing)	n/a	n/a	Tungsten	Tungsten
Structural Material	RAFM (ARAA)	RAFM (Eurofer/ARAA)	RAFM (ARAA)	RAFM (ARAA)
Tritium Breeder Material	Li ₂ TiO ₃	Li ₄ SiO ₄ /Li ₂ TiO ₃	Li ₂ TiO ₃	Li ₄ SiO ₄ /Li ₂ TiO ₃
Neutron Multiplier Material	Be	Be	Beryllide	Beryllide
Reflector Material	Graphite	n/a	Graphite	n/a
Max. dpa (Lifetime of blanket)	3	3	20 / 50	20 / 50
Primary Coolant	Helium	Helium	Helium	Helium
Coolant Inlet/Outlet Temperature	300/500 °C	300/500 °C	300/500 °C	300/500 °C
Coolant Pressure	8 MPa	8 MPa	8 MPa	8 MPa
Purge Gas	He with 0.1% H ₂	He with 0.1% H ₂	He with 0.1% H ₂	He with 0.1% H ₂
Enrichment (⁶ Li)	70%	70%	90%	90%

Candidates of Solid-type Tritium Breeders

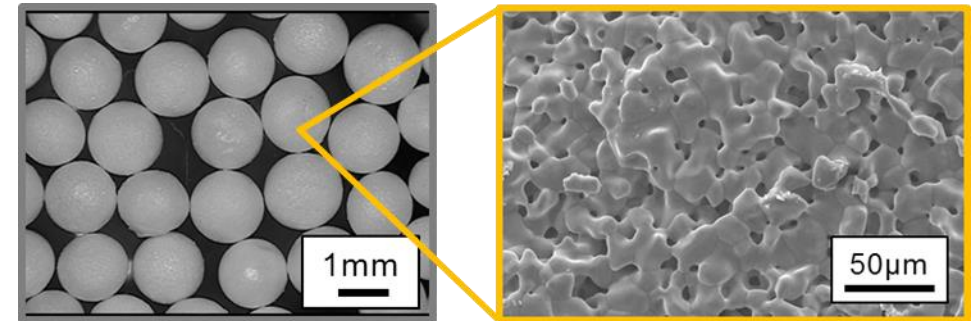
	Li_2O	Li_2AlO_2	Li_2ZrO_3	Li_4SiO_4	Li_2TiO_3
Li Vaporization (in additional H_2)	> 600°C	> 900°C	> 800°C	> 700°C	> 800°C
Long period use (2 years)	Instability (Li vaporization)	Stability	Instability (crack)	Instability (Li vaporization)	Instability (Reduction of Ti)
Tritium release (easy release)	> 400°C	> 400°C	> 400°C	> 350°C	> 300°C
Optimum operating temp.	400 - 600 °C	400 - 900°C	400 - 800°C	350 - 700°C	300 - 800°C
Tritium breeding ratio (TBR)	High	Lower	Middle	Middle	Middle
Thermal conductivity	High	Middle	Middle	Middle	Middle



by Dr. Hoshino,
CBBI-16, 2011

Shape of Tritium Breeder Ceramics : **Pebble**

- Stress Distribution
- Packing Behavior
- Thermal Conductance
- Purge Gas Flow
- ...



(Li_2TiO_3 Pebbles)

by Y.-H.Park et al,
JNM 455 (2014), 106

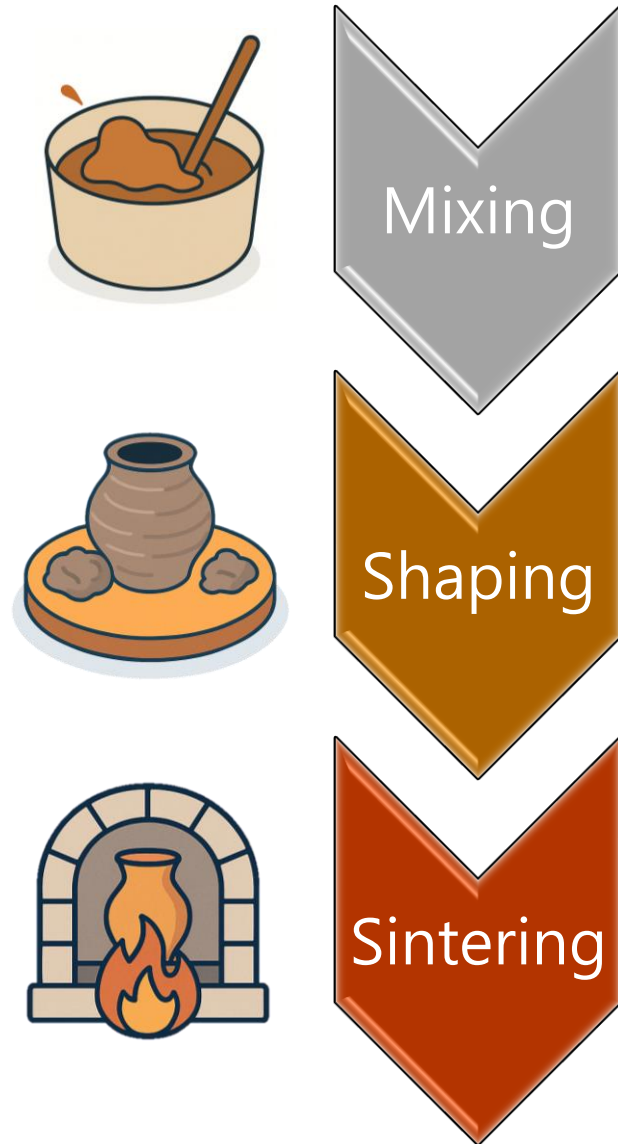
02

Manufacturing of Tritium Breeder Pebbles

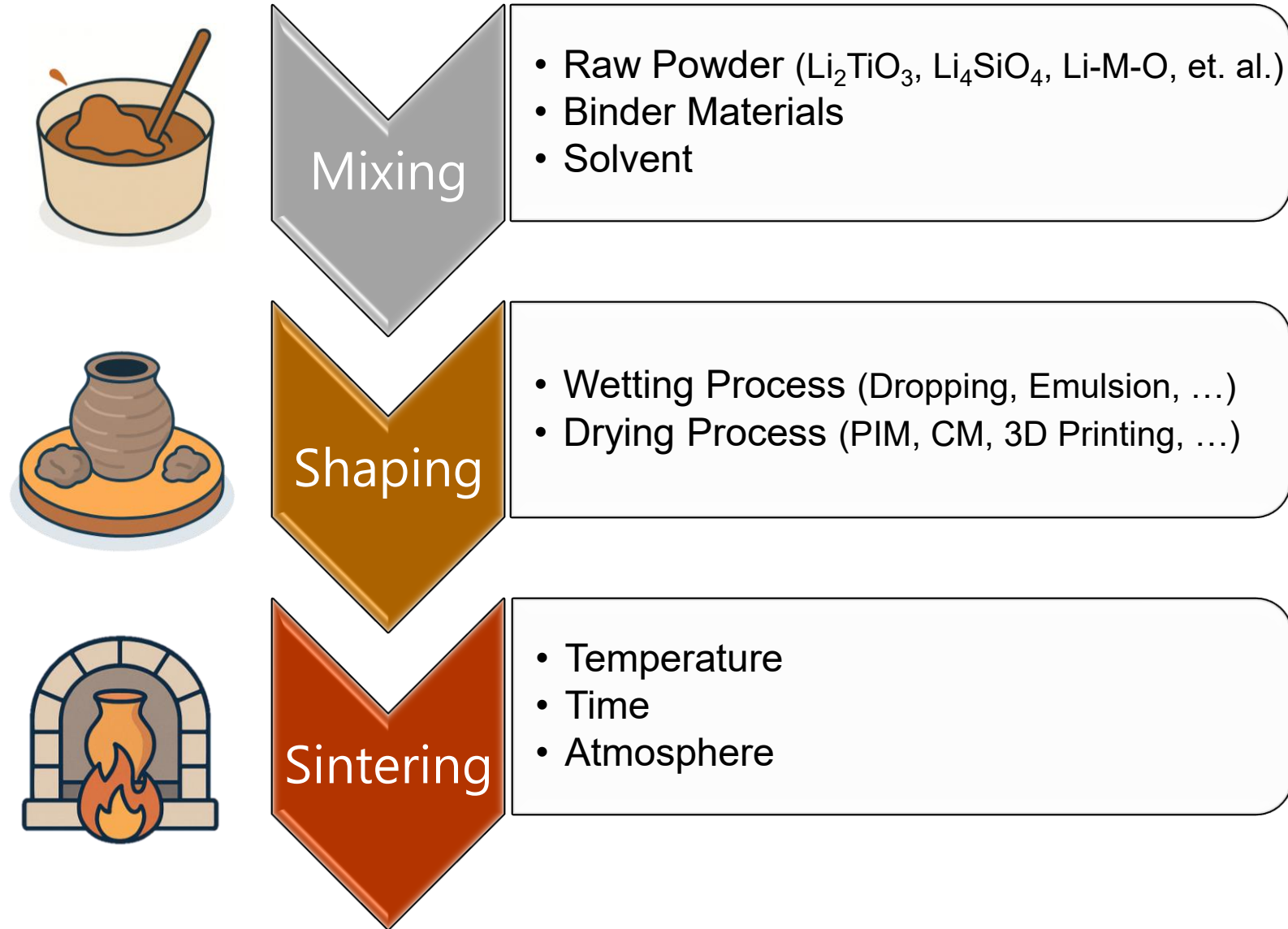


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Manufacturing Process for Ceramic Materials (using Sintering)



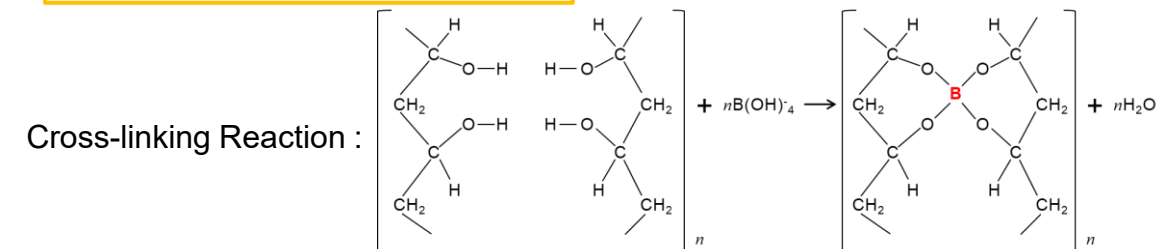
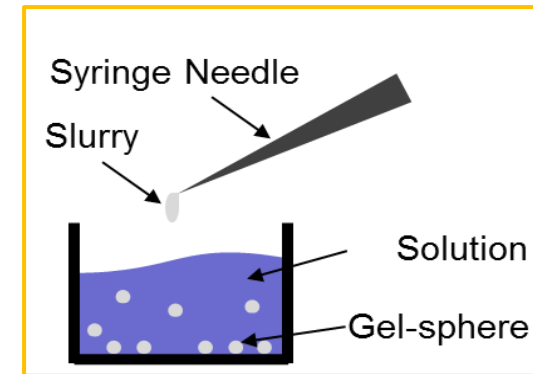
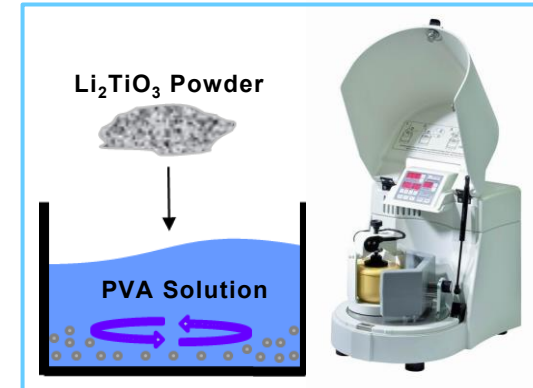
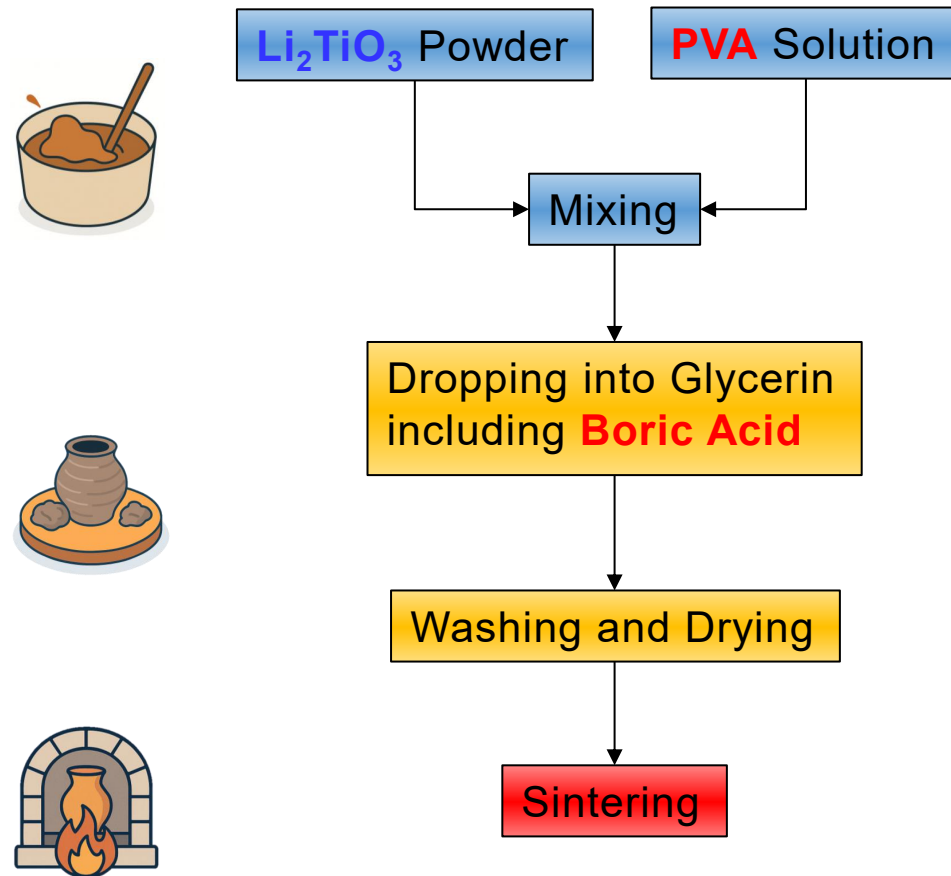
Manufacturing Process for Ceramic Pebble (using Sintering)



Development of Manufacturing Process for Li_2TiO_3 Pebbles

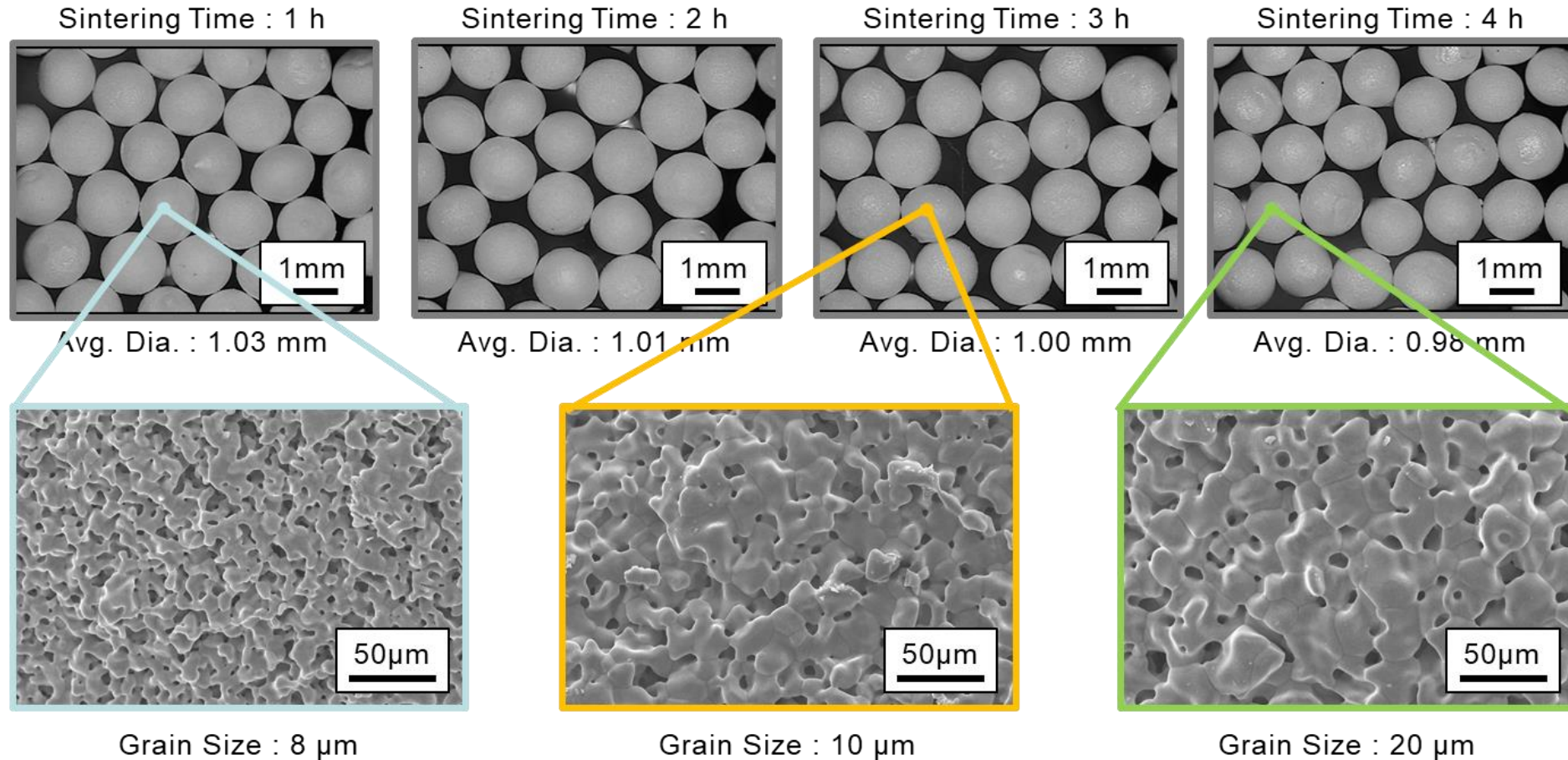
- **Slurry Droplet Wetting Method** based on the Cross-linking Reaction between PVA and Boric Acid

Manufacturing Procedure



Parametric Study on Sintering Conditions for Li_2TiO_3 Pebbles

● Effect of Sintering Time (1200 °C, Air atmosphere)



- Grain size was increased with increasing sintering time.
- The whole pore of the sintered pebble was open type.
- The open porosity was about 10 %. (Sintering Time : 3 h)

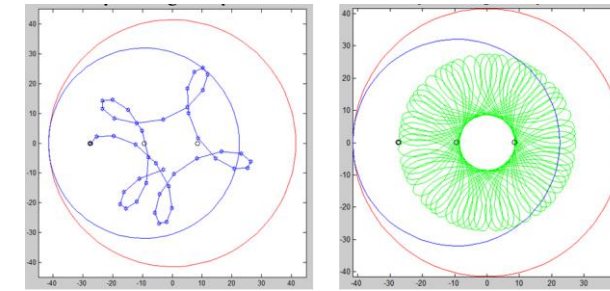
Mass-production System for Slurry Droplet Wetting Method

Automatic Slurry Dispensing System



Dispensing Unit

; for instillation of Li_2TiO_3 slurry

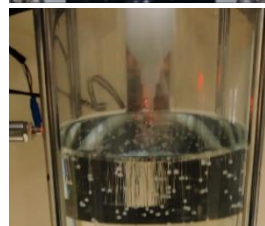
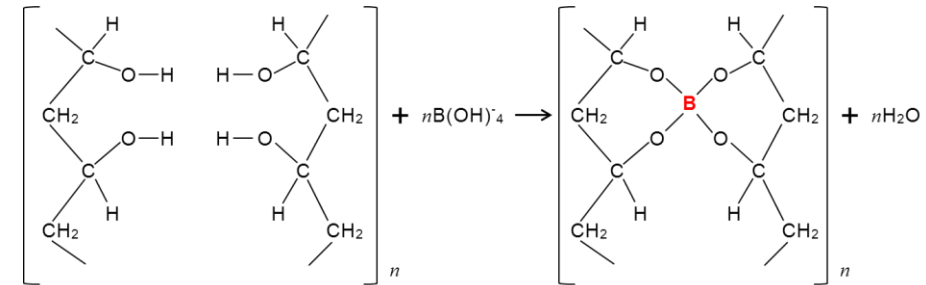


Trace of Drop Point through the Moving Needle



Glycerin Bath

; for hardening of droplets

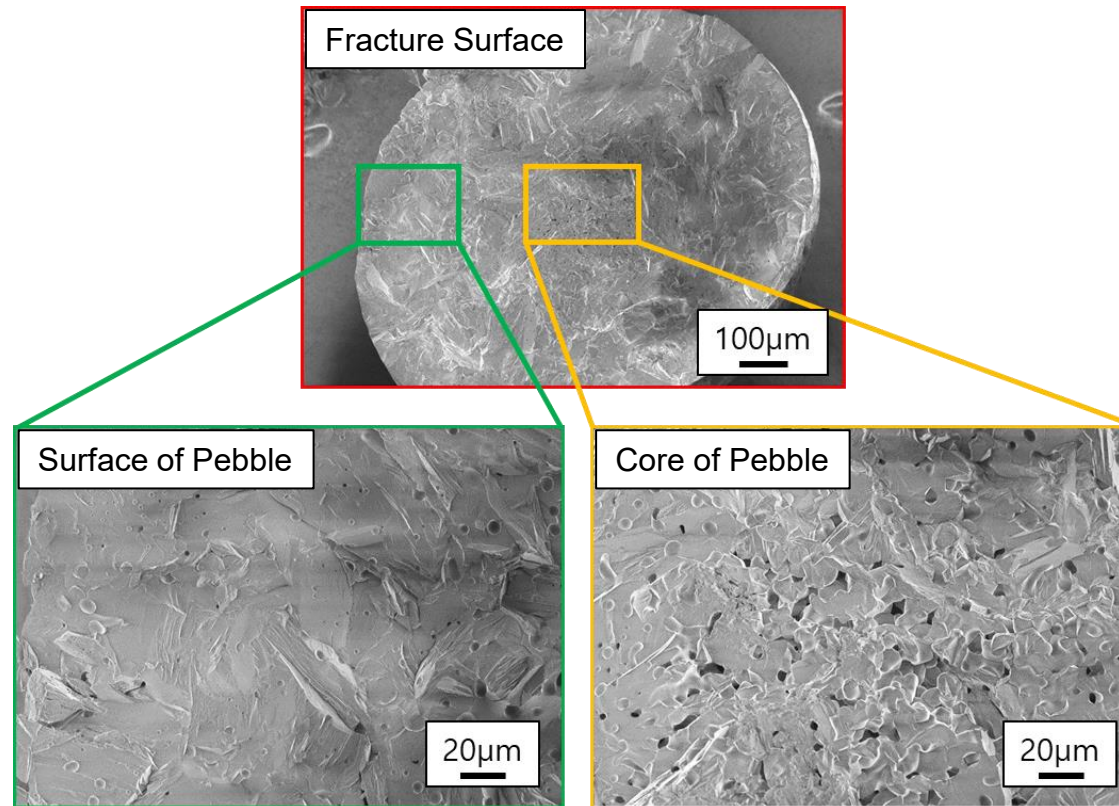


Automatic maintaining Unit

; for constant distance between syringe needle and glycerin surface

Shortcoming of Droplet Wetting Method for Mass-production

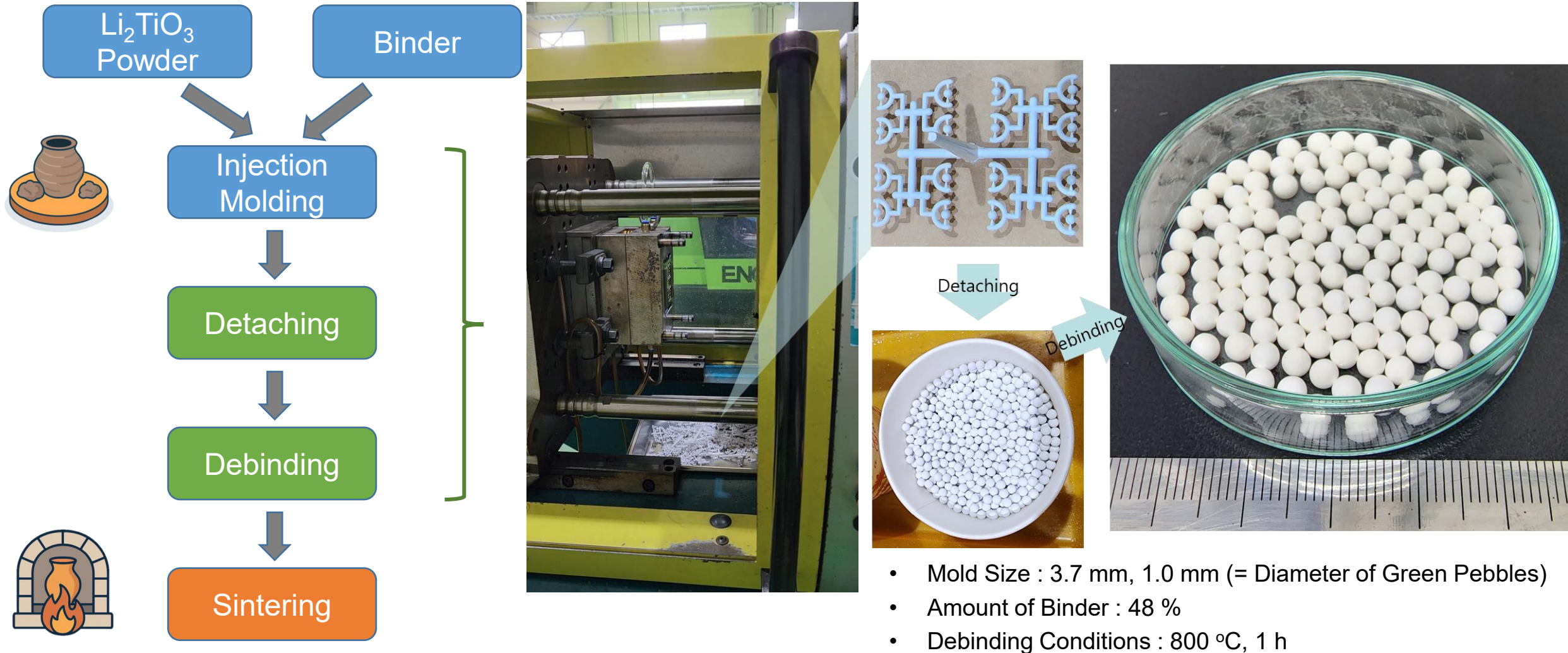
- **Non-uniform Microstructure** in Sintered Pebbles caused by Changing Concentration of Boric Acid
 - Decrease in the concentration of boric acid due to its consumption in the cross-linking reaction
 - Difference in hardening speed between the surface and the core of the droplets



[Microstructure of Sintered Li_2TiO_3 Pebble after Crush Test]

Development of Manufacturing Process for Li_2TiO_3 Pebbles

- Li_2TiO_3 pebbles have been successfully manufactured using **Powder Injection Molding (PIM)** process



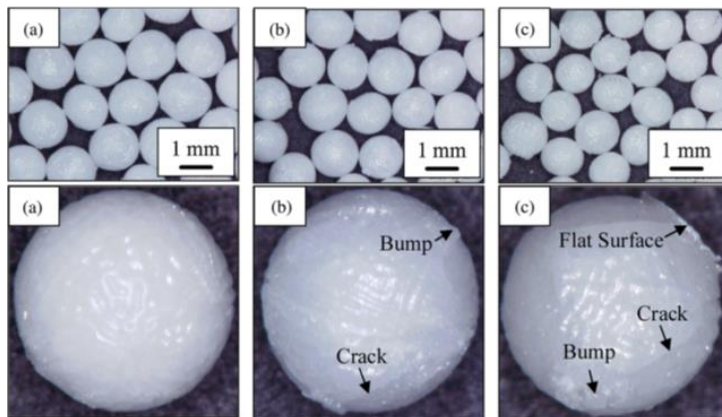
Improvement of Sintering Process for Mass-production

Formation of Defects caused by Batch Process

- ▶ Sintering between the contacted neighboring pebbles
→ Crack, Bump, Flatted Spot, and so on.

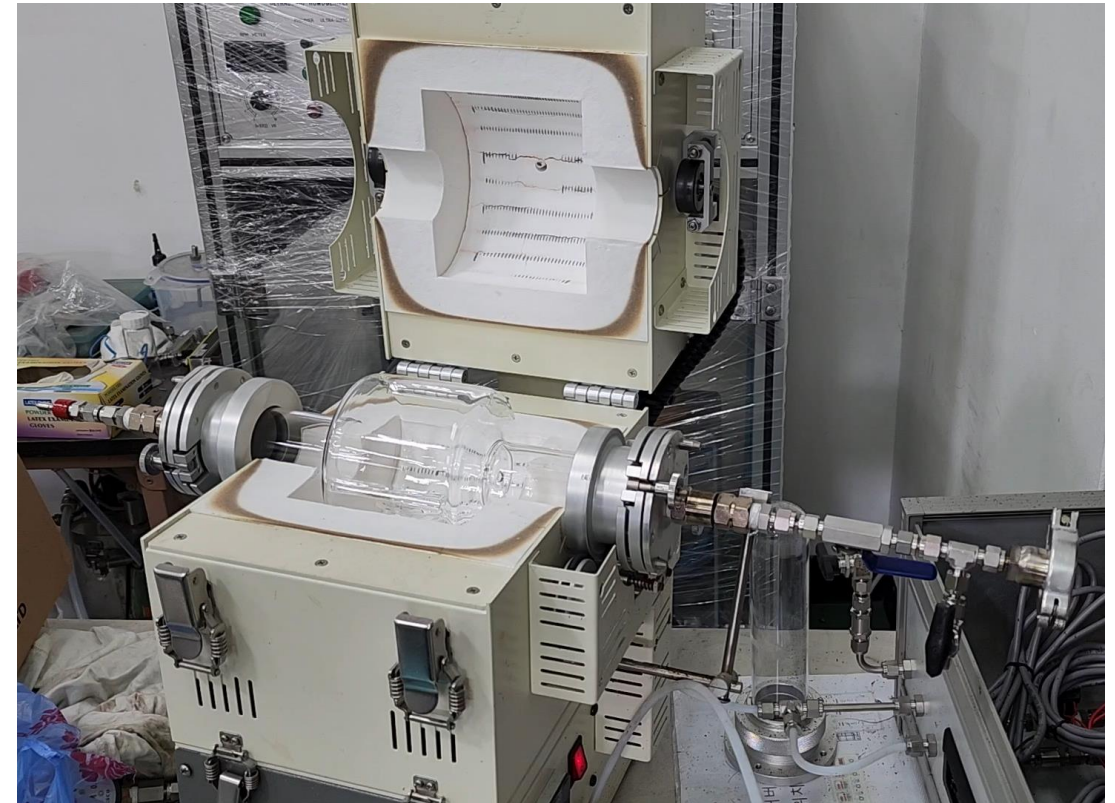


Improvement

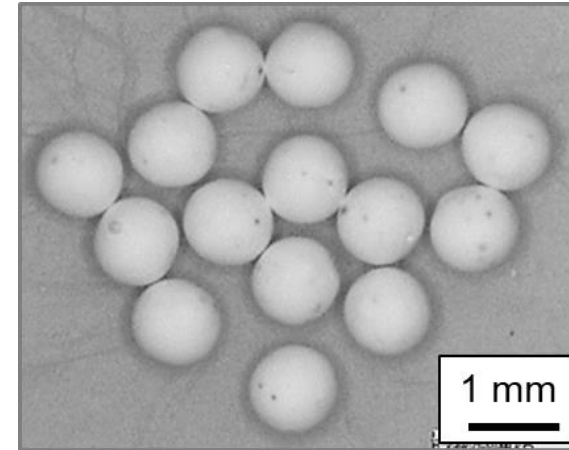
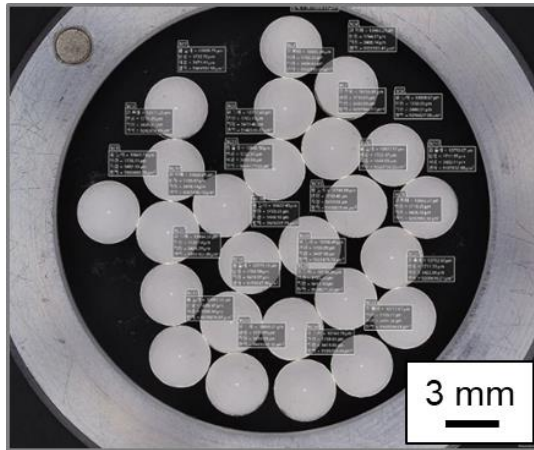


by Y.-H.Park et al, FED 109-111 (2016), 443

- ▶ **Rotating Sintering System** was adopted to prevent the sintering between neighboring pebbles in the crucible.

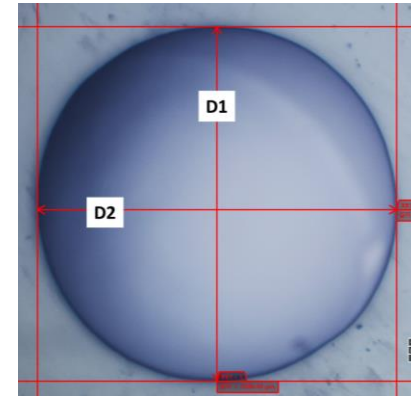


Morphology of Li_2TiO_3 Pebbles manufactured using PIM Process



Value	Item	Value
3.70 mm	Mold Size	1.00 mm
3.43 mm	Average Diameter	0.96 mm
< 1.01	Average Sphericity	< 1.01
16 %	Volume Shrinkage Ratio	15 %

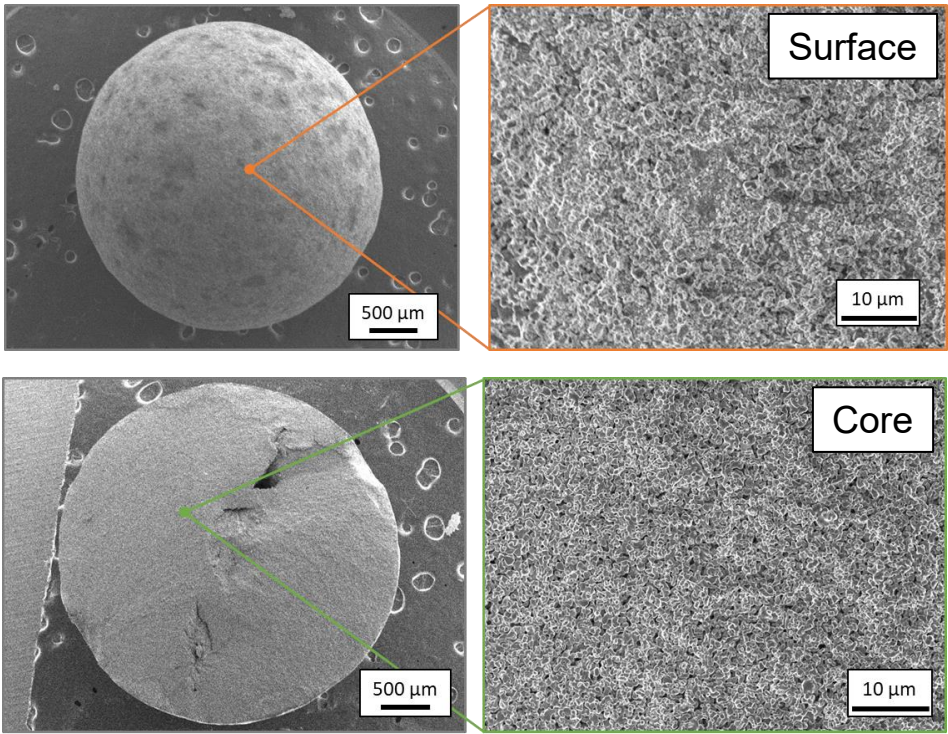
Sphericity = $D1 / D2$, ($D1 > D2$)



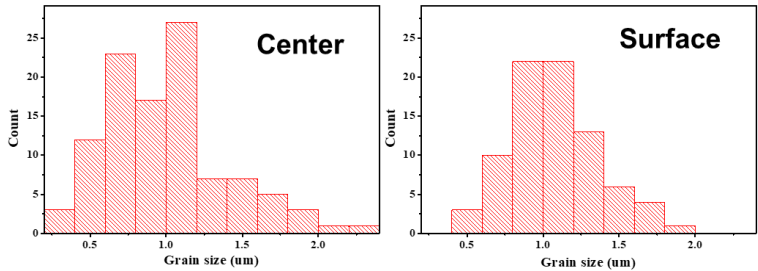
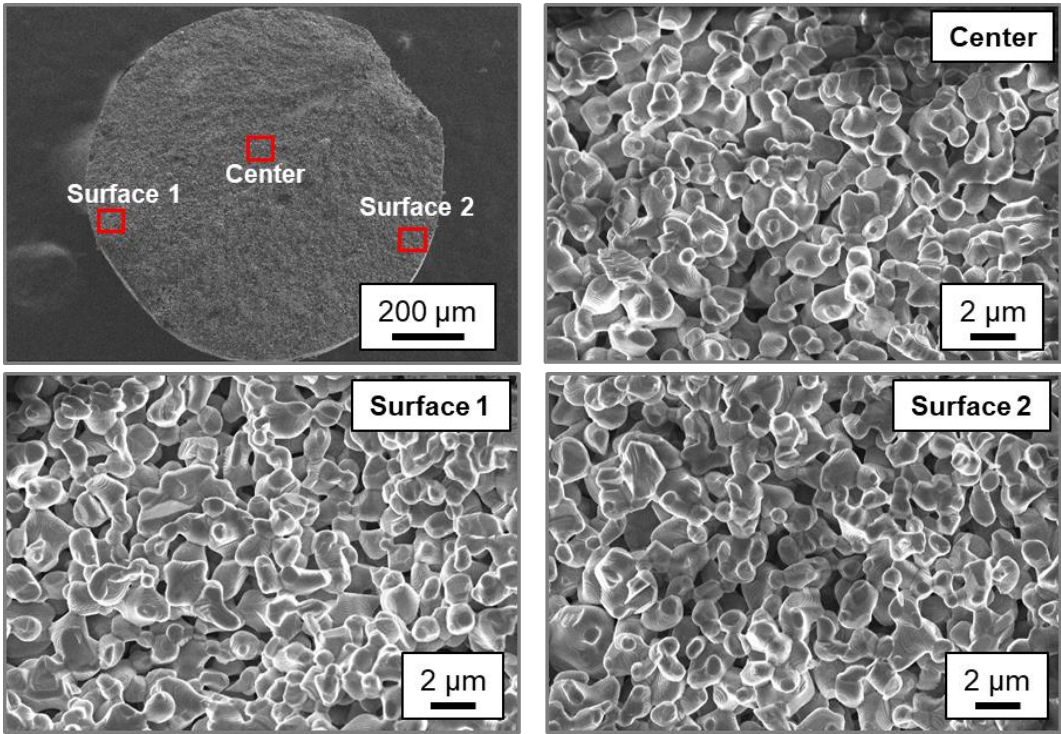
- Combined pebbles were not observed due to the pebble moving in the rotating tube during sintering process.
 - Defects (crack, bump, flatted surface, and so on) were not formed at the surface of sintered pebbles.
- The shrinkage ratio can be used as a parameter to design the mold in order to control the pebble diameter.

Microstructure of Li_2TiO_3 Pebbles manufactured by PIM Process

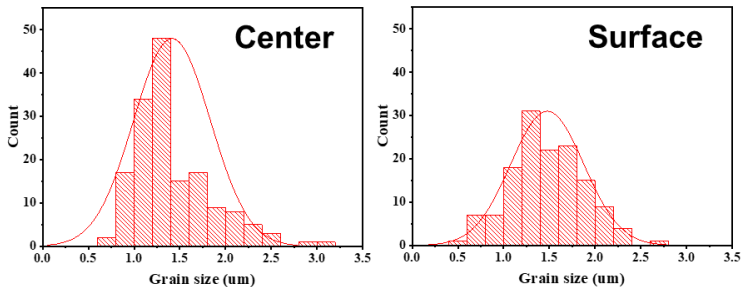
Diameter : 3.43 mm



Diameter : 0.96 mm



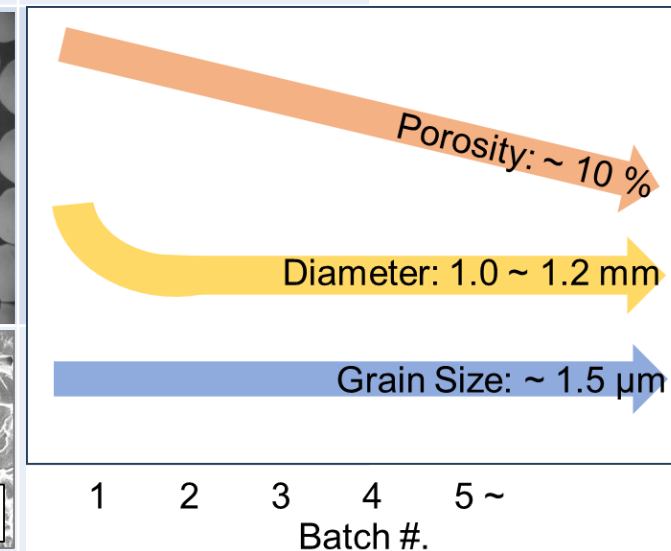
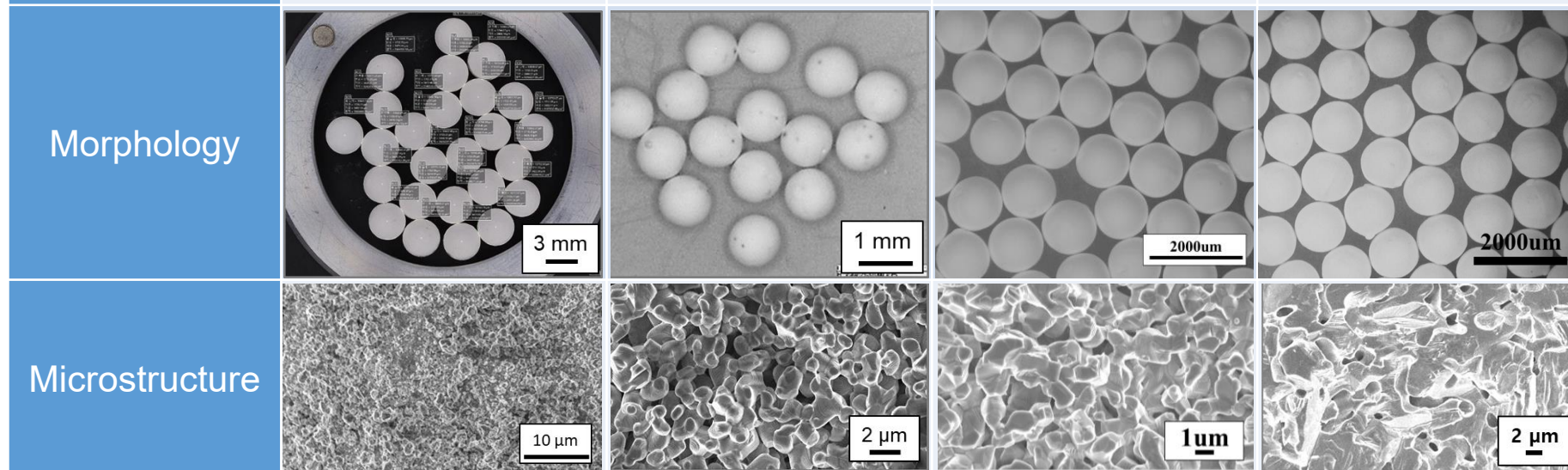
Value	Item	Value
1.00 μm	Grain Size	1.35 μm
39.7 %	Porosity	34.2 %



 A uniform microstructure with open pores in the surface and the core of sintered pebbles was confirmed.

Manufacturing Status of Li_2TiO_3 Pebbles based on Drying Process

Batch #	# 1	# 2	# 3	# 4	# 5 ~ (target)
Diameter	3.43 mm	0.96 mm	1.19 mm	1.19 mm	1.00 ~ 1.20 mm
Porosity	39.7 %	34.2 %	32.6 %	25.9 %	~ 10 %
Pore Type	Open	Open	Open	Open + Closed	Open
Grain Size	1.00 μm	1.35 μm	1.35 μm	-	1.00 ~ 1.50 μm
Shaping Proc.	PIM	PIM	PIM	PIM	PIM or 3DP
Manuf. Status	Done	Done	Done	Done	On-going



03

Neutron Irradiation Test and Tritium Release Test of Breeder Pebbles



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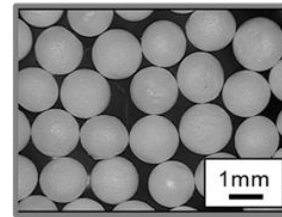
Breeding and Release Research under KO-CN International Collaboration Program

Neutron Irradiation and Tritium Release of Li_2TiO_3 Pebbles using D-T Fusion Neutron Source Facility

- Phase 1 : 2021.10 ~ 2023.10
- Phase 2 : 2024.12 ~ 2026.12

Memorandum of Understanding between Korea Institute of Fusion Energy (KFE), Republic of Korea and Institute of Nuclear Energy Safety Technology, Hefei Institutes of Physical Science, Chinese Academy of Sciences (INEST, HFIPS, CAS), People's Republic of China	
This Memorandum of Understanding (hereinafter referred to as "MoU") is entered into between the Korea Institute of Fusion Energy (KFE), Republic of Korea and the Institute of Nuclear Energy Safety Technology, Hefei Institutes of Physical Science, Chinese Academy of Sciences (INEST, HFIPS, CAS), People's Republic of China, (hereinafter referred to as "Parties"). This MoU is an extension of the MoU between KFE and INEST, currently known as the Korea Institute of Fusion Energy Safety Technology that was concluded on [Date].	
ARTICLE III. COLLABORATIVE ACTIVITIES Form of cooperative activities under this MoU may include such areas of mutual interest as: 3.1. Exchange of personnel 3.2. Utilization of equipment, devices, materials, and instrumentations 3.3. Exchange of technical information, data and experience 3.4. Participation in seminars, workshops and other activities 3.5. Other collaboration as the Parties may agree	
ARTICLE IV. SOURCE OF FUNDING Cooperative activities under this MoU will be carried out on a non-exclusive basis. The Parties shall make mutual arrangement between the Parties.	
ARTICLE V. INTELLECTUAL PROPERTY 4.1. Either Party has a non-exclusive, irrevocable license to the other Party for its own internal research and development information shall be the subject of a separate case-by-case basis. The information may not be disclosed to third parties without the written consent of the Party that supplies the information. ARTICLE VI. ENTRY INTO FORCE AND TERM This MoU shall enter into force after having been signed by both Parties for five (5) years, unless terminated earlier by either Party. This MoU may be modified or amended by mutual agreement of both Parties. The termination of this MoU shall not affect the validity of the activities initiated prior to such termination.	
IN WITNESS WHEREOF, each of the Parties has signed and affixed its seal to two copies of this MoU, one of which shall remain with each Party.	

Signature: [Signature] Date: 7/16/2024 OH Yeongkook President Korea Institute of Fusion Energy Republic of Korea	Signature: [Signature] Date: 7/11/2024 Yu Jie Director General Institute of Nuclear Energy Safety Technology, Hefei Institutes of Physical Science, Chinese Academy of Sciences People's Republic of China
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(KFE) Li_2TiO_3 Breeder Pebbles

- Synthesis of Li_2TiO_3 Raw Powder
- Fabrication of Li_2TiO_3 Breeder Pebbles
- Evaluation of un-irradiated Li_2TiO_3 Pebbles



Irradiation
Exp. Data



(HFIPS) D-T Neutron Source

- Neutron Irradiation on Li_2TiO_3 Pebbles
- Measurement of Bred Tritium
- Evaluation of irradiated Li_2TiO_3 Pebbles

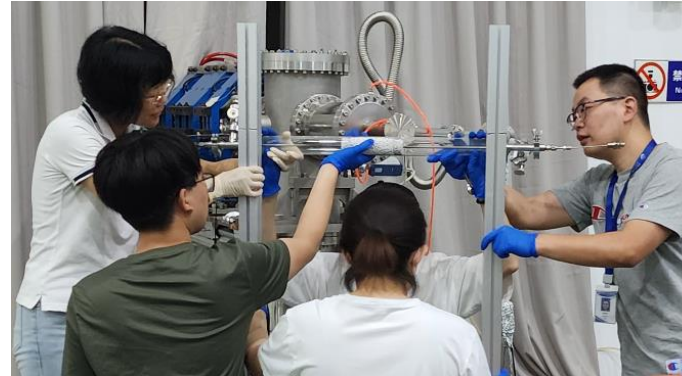


Li_2TiO_3
Breeder Pebbles

MoU between KFE and INEST

1st Neutron Irradiation Test on Li_2TiO_3 Pebbles

- Date : 2023.06
- Neutron Source : HINEG-CAS
 - Neutron Energy : 14.1 MeV
 - Neutron Yield : $\sim 10^{12}$ n/s
- Irradiation Test Conditions
 - Duration Time : 6 h
 - Total Number of Neutron : 1.104×10^{15} n
 - Neutron Fluence at Pebble : 9.96×10^{11} n/cm²
- Tritium Breeder Sample
 - Material : Li_2TiO_3 Pebbles
 - Diameter : 3.43 mm
 - Sphericity : < 1.01
 - Porosity : 39.7 %
 - Amount : 274 g



Nb Foil

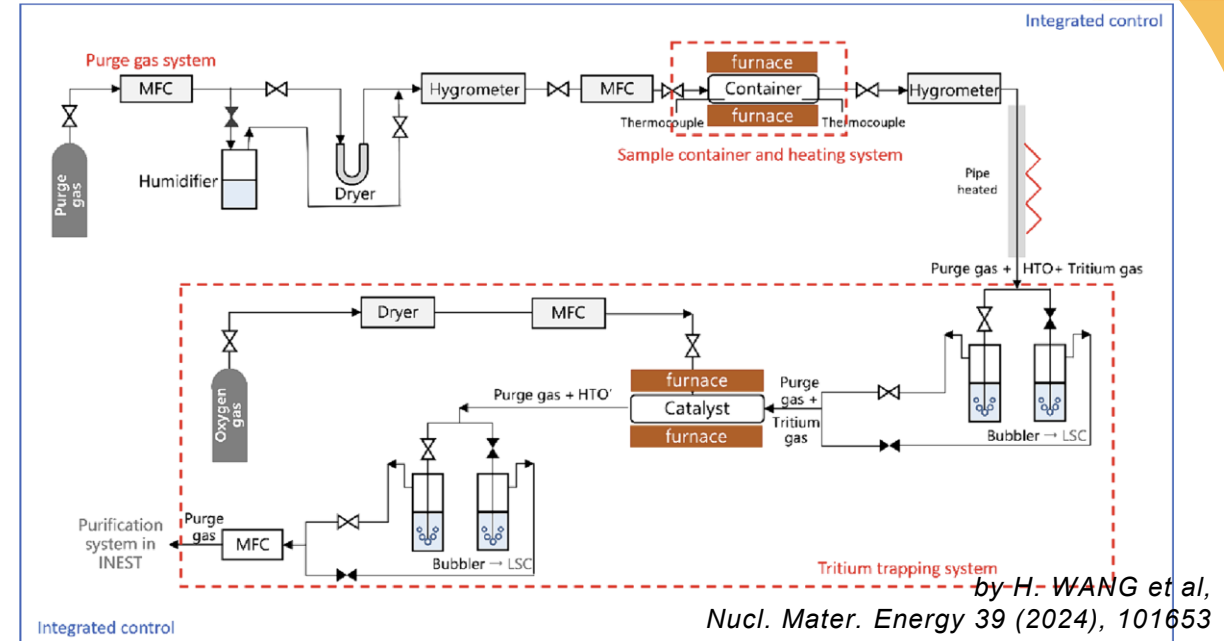
Cadmium Sheet



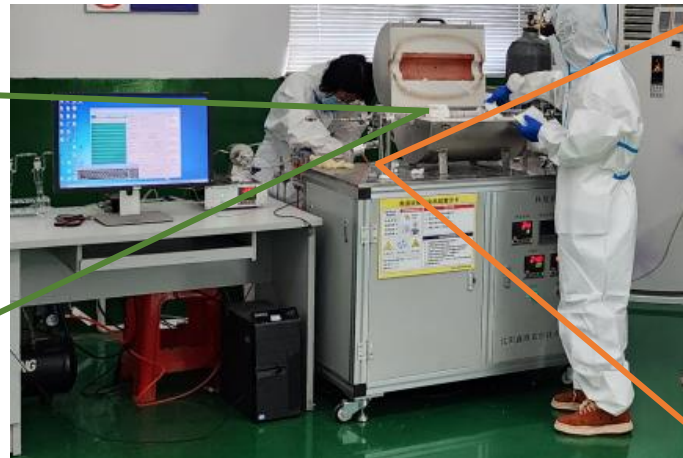
Tritium Release Test of Irradiated Li_2TiO_3 Pebbles

Test Conditions

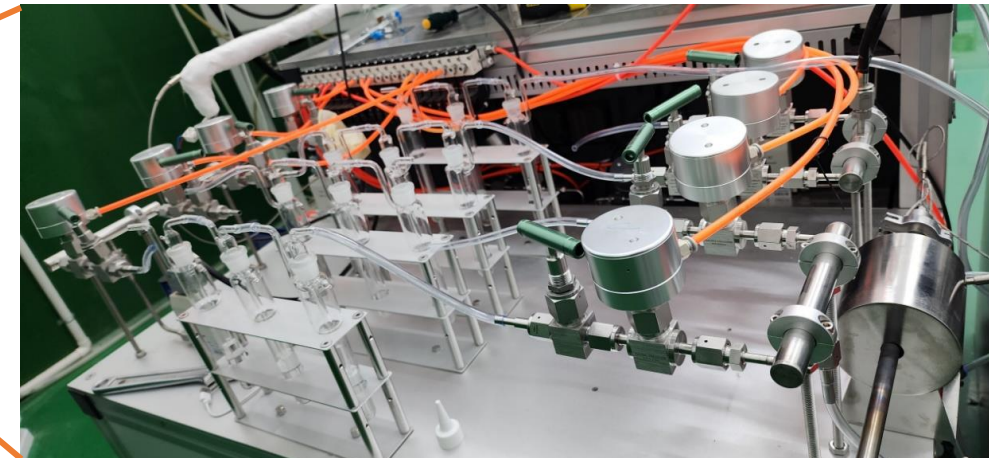
- Purge Gas : $\text{He} + 1\% \text{H}_2$
- Flow Rate of Purge Gas : 100 ml/min
- Humidity Level : $< 15 \text{ ppm}$
- Tritium Release Temperature : R.T. $\sim 800^\circ\text{C}$
- Catalyzer to change HT $\rightarrow$ HTO : Pt/SDB
- Tritium Measurement : LSC Method
(Lower Detection Limit : 0.7 Bq/L)



<Irradiated Pebble Assembly>



<Tritium Release System>



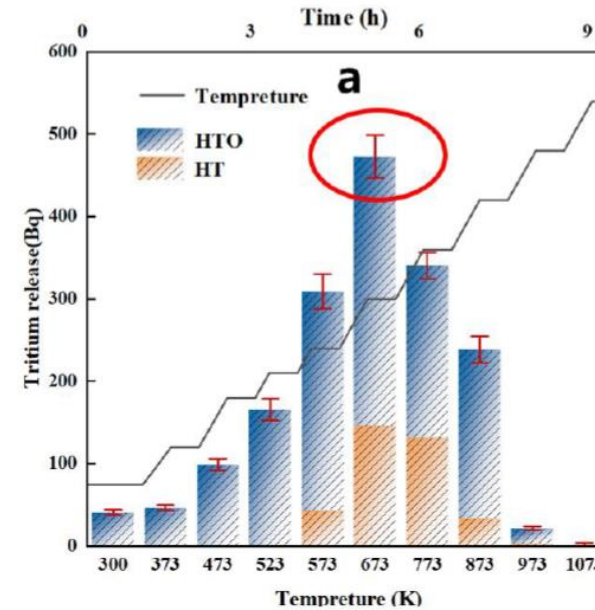
<Bubblers to trap HTO>

1st Tritium Release Test Results from Irradiated Li₂TiO₃ Pebbles

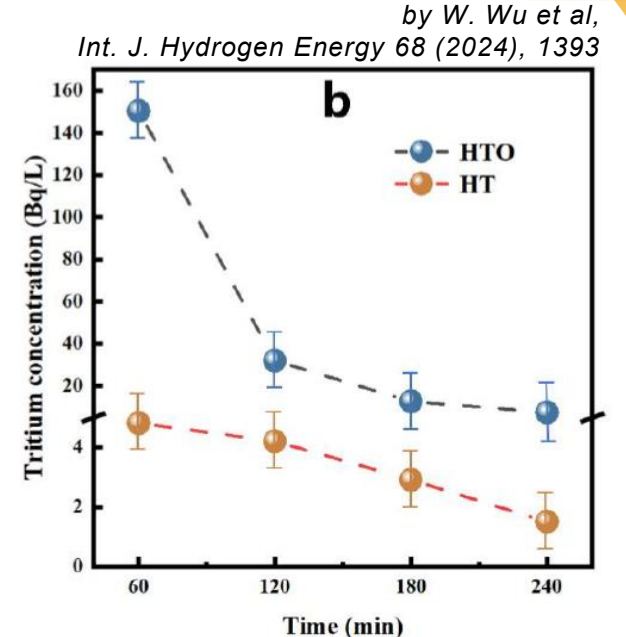
<Tritium Radioactivity in Bubblers>

T(K)	Radioactivity (Bq)					
	1#	2#	3#	4#	5#	6#
RT	39.1	0.1		1.2	0.1	
373	46.0	0.1		0.2	0.1	
473	98.8	0.2		0.1	0.1	
523	163.9	0.6		1.1	0.1	
573	264.1	0.9	BG	43.8	0.1	BG
673	325.2	1.5		145.6	0.5	
773	208.0	0.8		131.4	0.6	
873	204.0	0.9		33.5	0.1	
873'	22.2	0.1		3.5	0.1	
Total	HTO: 1376.5 Bq			HT: 362.2 Bq		

*BG: Background



Distribution curves of tritium release at different temperature



Tritium measurement results at 1073 K for 4 h

- Before heating (during irradiation): A certain amount of HTO (54.4 Bq) and HT (23.6 Bq) was released from the surface of Li₂TiO₃ pebbles.
- At elevated temperature: Tritium release peaked at 400 °C, with the release ratio of HTO to HT being approximately 79.3 % to 20.7 %.
- At 800 °C: The tritium concentration decreased to background levels, indicating no residual tritium retention in the pebbles after 4 h.

04

Summary



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- Li_2TiO_3 pebbles with high sphericity for tritium breeder material were successfully fabricated using Slurry Droplet Wetting process and Powder Injection Molding (PIM) process.
 - In the PIMed Li_2TiO_3 pebbles, a uniform microstructure was observed both at the surface and the core of the sintered pebbles, while no defects resulting from sintering between neighboring pebbles were detected.
- Tritium production and release experiments were conducted on Li_2TiO_3 pebbles with 14.1 MeV fusion neutron source HINEG-CAS and tritium release system.
 - The total radioactivity of tritium released from the irradiated Li_2TiO_3 pebbles with a weight of 274 g was approximately 1,866.4 Bq.
 - The tritium release peaked at 400 °C, with the release ratio of HTO to HT being approximately 79.3 % to 20.7 %.
- *Future Works;*
 - Validate the performance of oxidizers in tritium release system
 - Investigate the effects of purge gas composition
 - Compare the influence of breeder pebble properties

Thank you for your kind attention !!!

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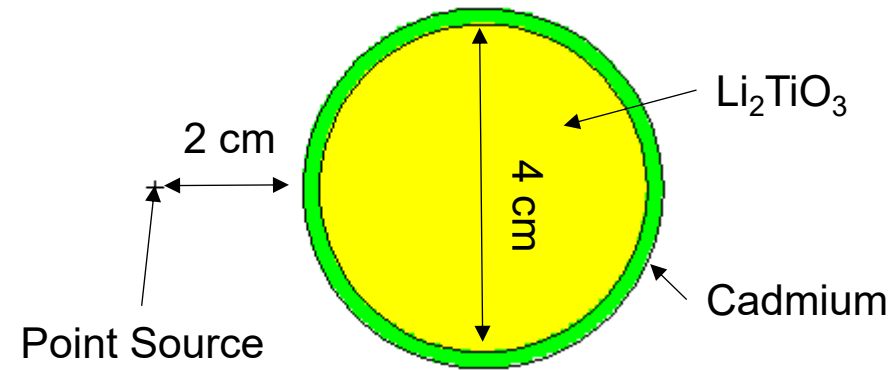


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MCNP Result of Tritium Production

MCNP Model

- Source : 14.1 MeV Point Source
- Material : Li_2TiO_3 (Natural Lithium)
- Cd Thickness : 2 mm
- Weight of Li_2TiO_3 : 274 g



Results

- T Production (with Cd) : 3,787 Bq (2.24×10^{12}) regarding neutron fluence
- T Production (w/o Cd) : 4,146 Bq (2.33×10^{12}) regarding neutron fluence
- Neutron Spectrum

