

Reduction mass improvement by hydrogen addition in lunar resource laser alumina reduction

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Introduction

ISRU (In-Situ Resource Utilization)



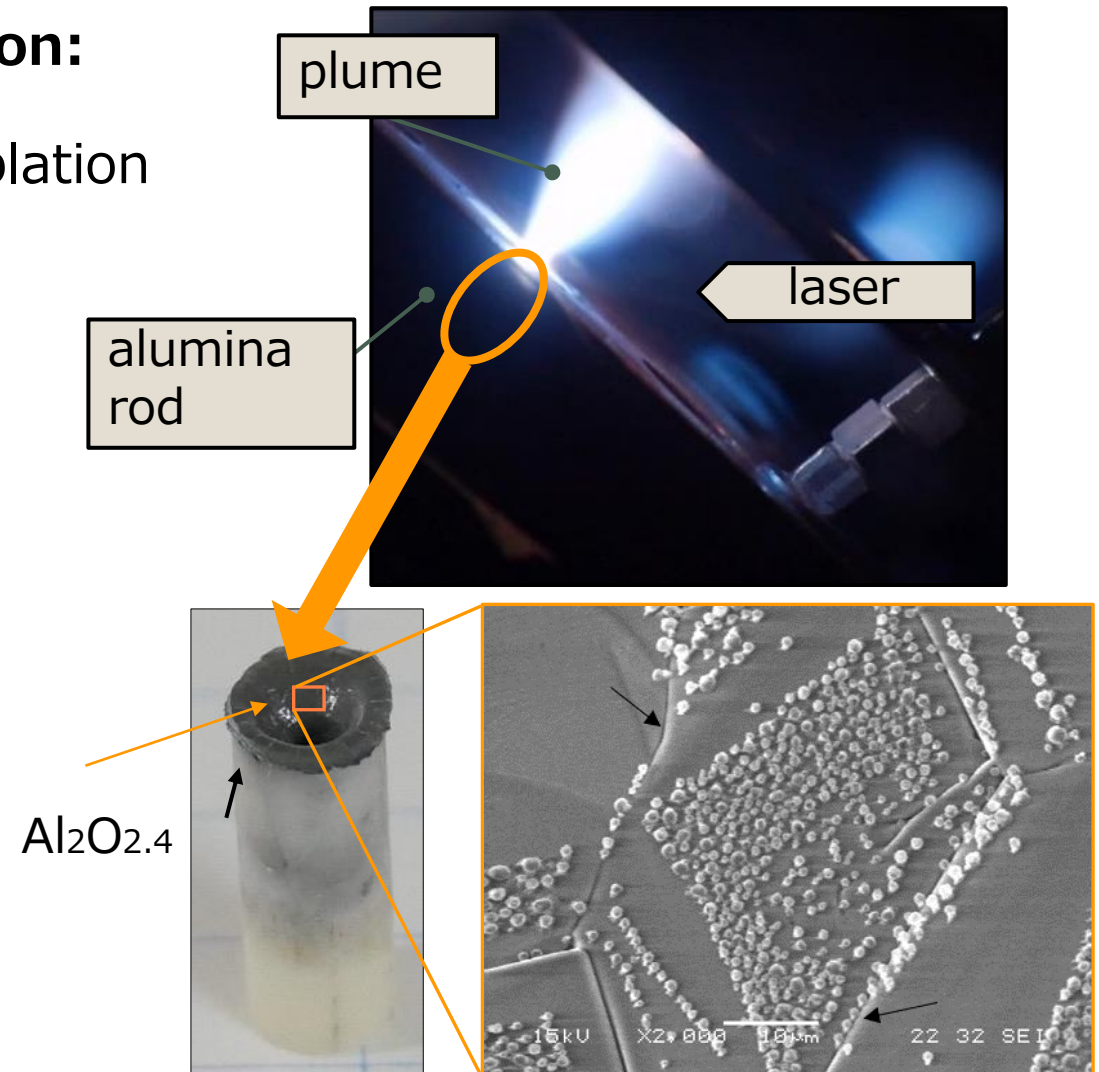
One of the Al collection method in Laser Ablation:

- Al particles are observed on rod surface after ablation
- Al particles are produced by precipitation from Oxygen-deficient alumina

Oxygen deficient Alumina:

- Lower Oxygen content than normal
- Made by laser ablation
- Al_2O_x (x: Oxygen deficiency, $x \leq 2.9$)

To improve collection efficiency, increase the oxygen deficiency is necessary

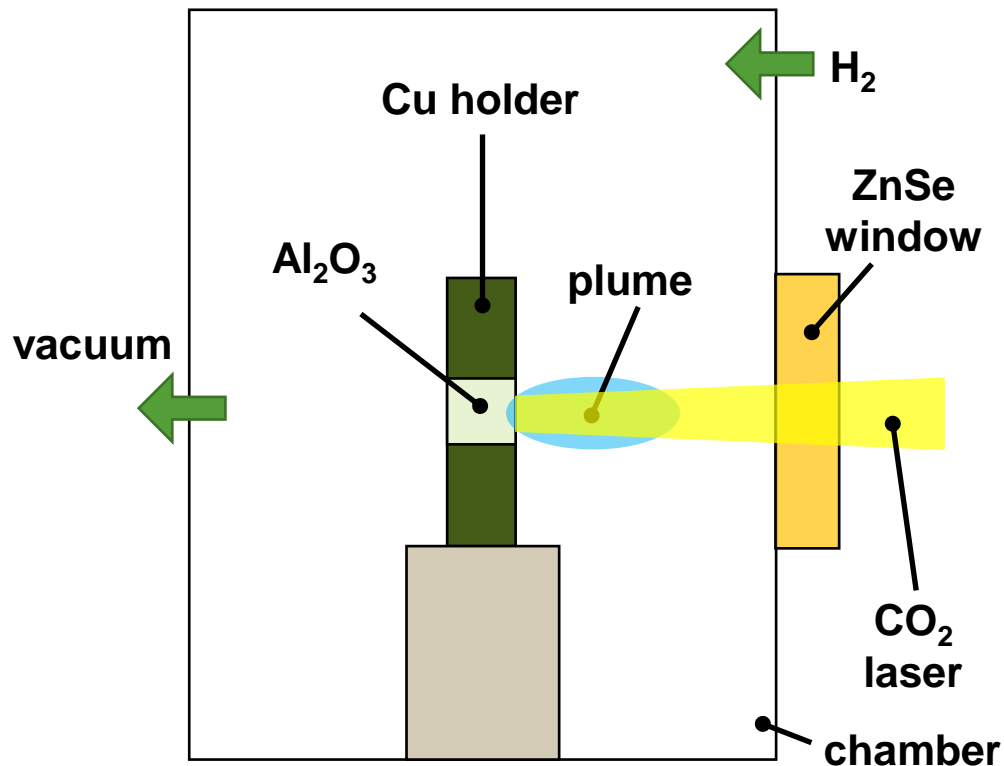


Previous experiment

Reduction experiment in hydrogen atmosphere

Ablation in **1 bar of H₂ atmosphere** conducted by N.Tanaka [1]
→ Expect H₂ to act as a **reducing agent**, to increase **Oxygen deficiency**

Experimental system

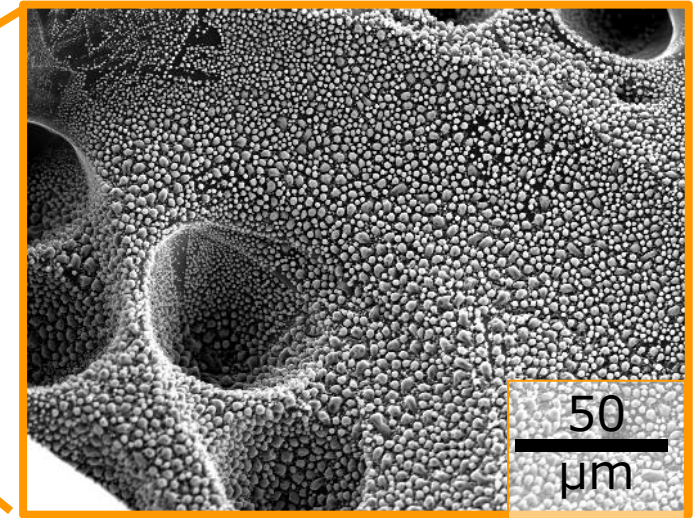
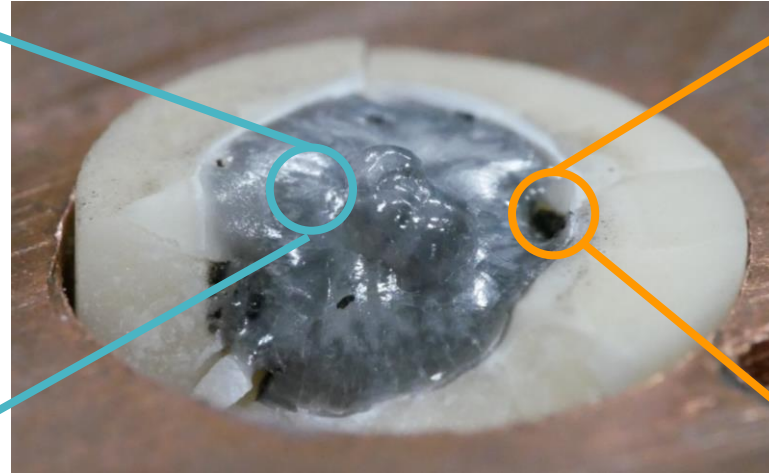
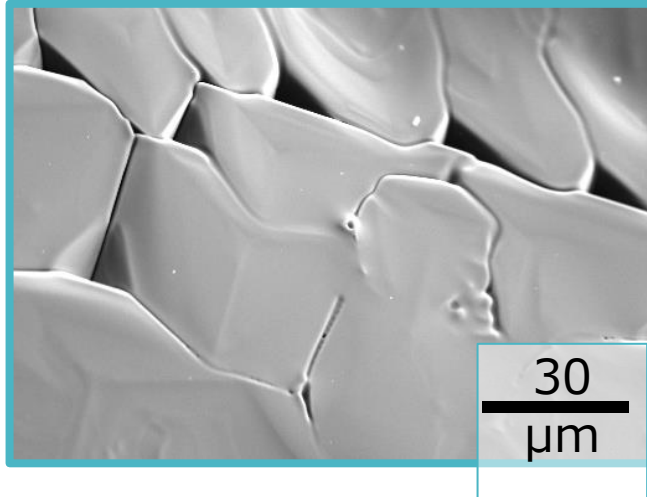


Experimental conditions

Laser: CO₂ laser, 2.0 kW
Laser spot radius : 2 mm
Irradiation time : 1.0 s
Atmosphere : **H₂ 1 atm**

[1] Naoki Tanaka. "アルミナレーザー還元過程における酸素欠陥型アルミナの生成とその制御". 東京大学修士論文. 2022.

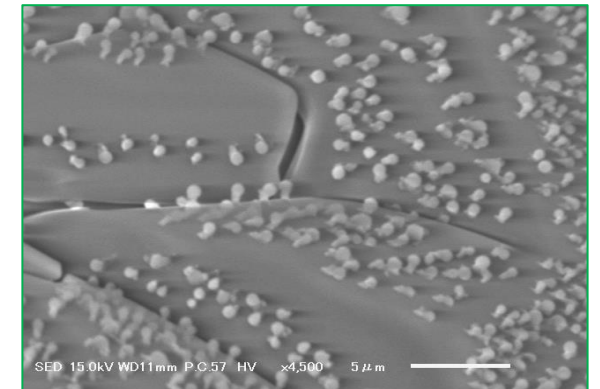
Reduction experiment in hydrogen atmosphere



Al particles in vacancy ($13\mu\text{g}/\text{mm}^2$)

Result:

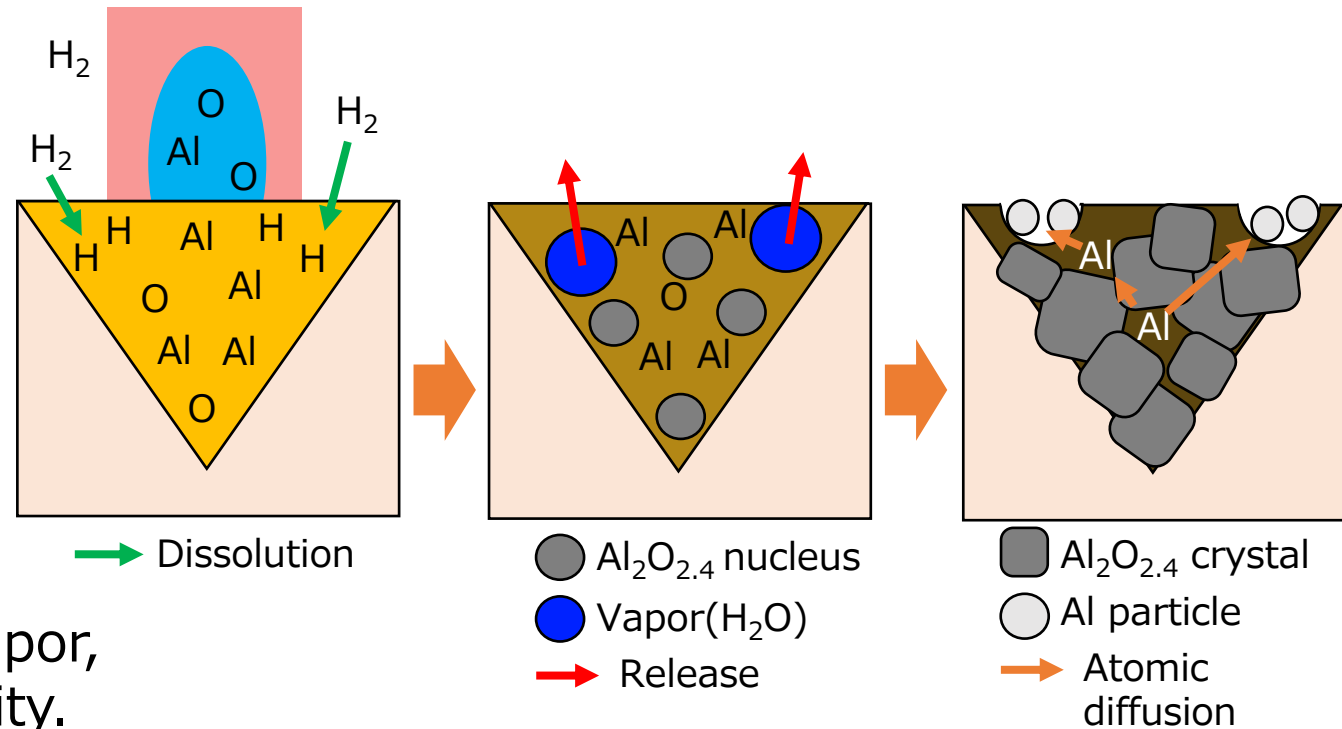
- Vacancies at the edge of the melting region, and Al precipitates in high-density inside
- No Al precipitation in other places
- Total amount of Al precipitation was $14\mu\text{g}$, which is same as experiment in Ar atmosphere.



Rod surface after ablation under Ar atmosphere ($\approx 0.3\mu\text{g}/\text{mm}^2$)

Explanation of results

- ① Hydrogen dissolves in molten edge of liquid alumina
- ② After the ablation, vacancies are formed due to the release of gas(H_2O/H_2) dissolved
- ③ The vacancies become high oxygen deficiency due to release of water vapor, Al particles precipitated in high density.



A clue to increase Al precipitation mass

Being blocked by the plume, H_2 was dissolved only in molten edge,
Vacancies with high density Al precipitation occur only at the edge

[2] 山中伸介, "水素と金属, 酸化物セラミックス," 生産と技術 第49巻 第3号, 1997.

[3] Ueno, et.al, "一方向凝固法によるロータス型ポーラスセラミックの作製", まてりあ, 2008.

Previous research:

- In the reduction experiment with hydrogen, although some **vacancies with high density aluminum precipitation** were observed, **the overall precipitation was not increased**

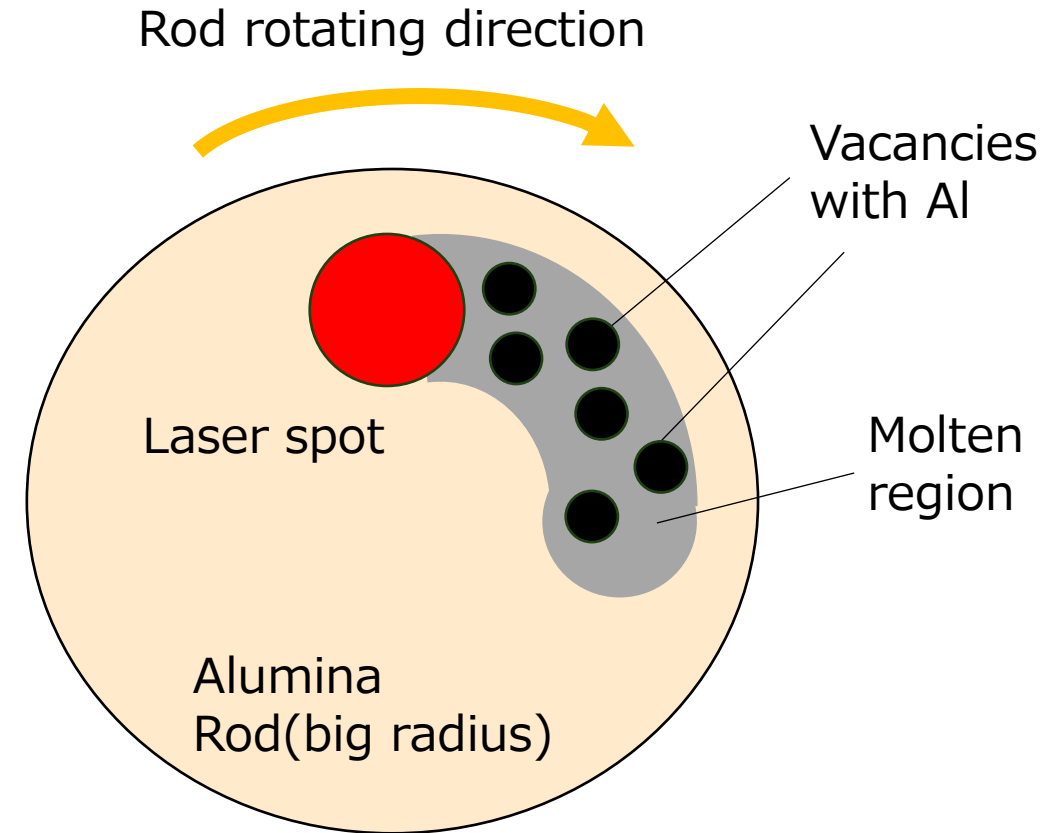
Objective:

Devise and conduct experiment to let H₂ dissolve into entire molten region, increase the number of vacancies with high density Al precipitation, and **increase Al precipitation mass**

Experiment

The way to increase the number of vacancies:

- Use large diameter alumina rods rotating at a constant speed
- Laser irradiates off-center of rod rotation

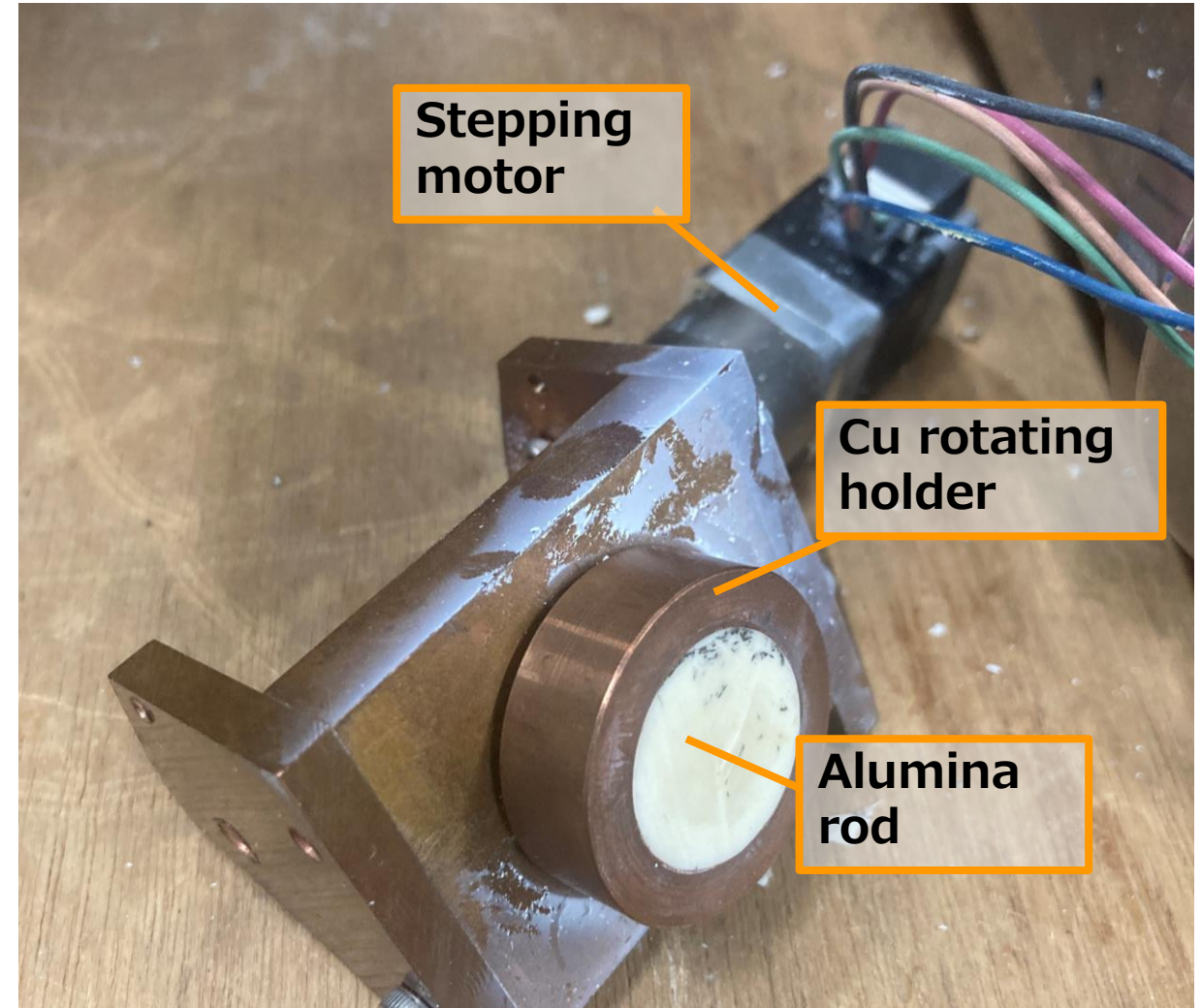


Conceptual diagram of rod rotation system

→ The edge of Laser spot sweeps the entire molten region, allowing hydrogen to dissolve over all the region

Overview of rod rotation system

- Using $\phi 20$ Alumina rod held by copper holder
- The rod are rotated by stepping motor through bevel gear

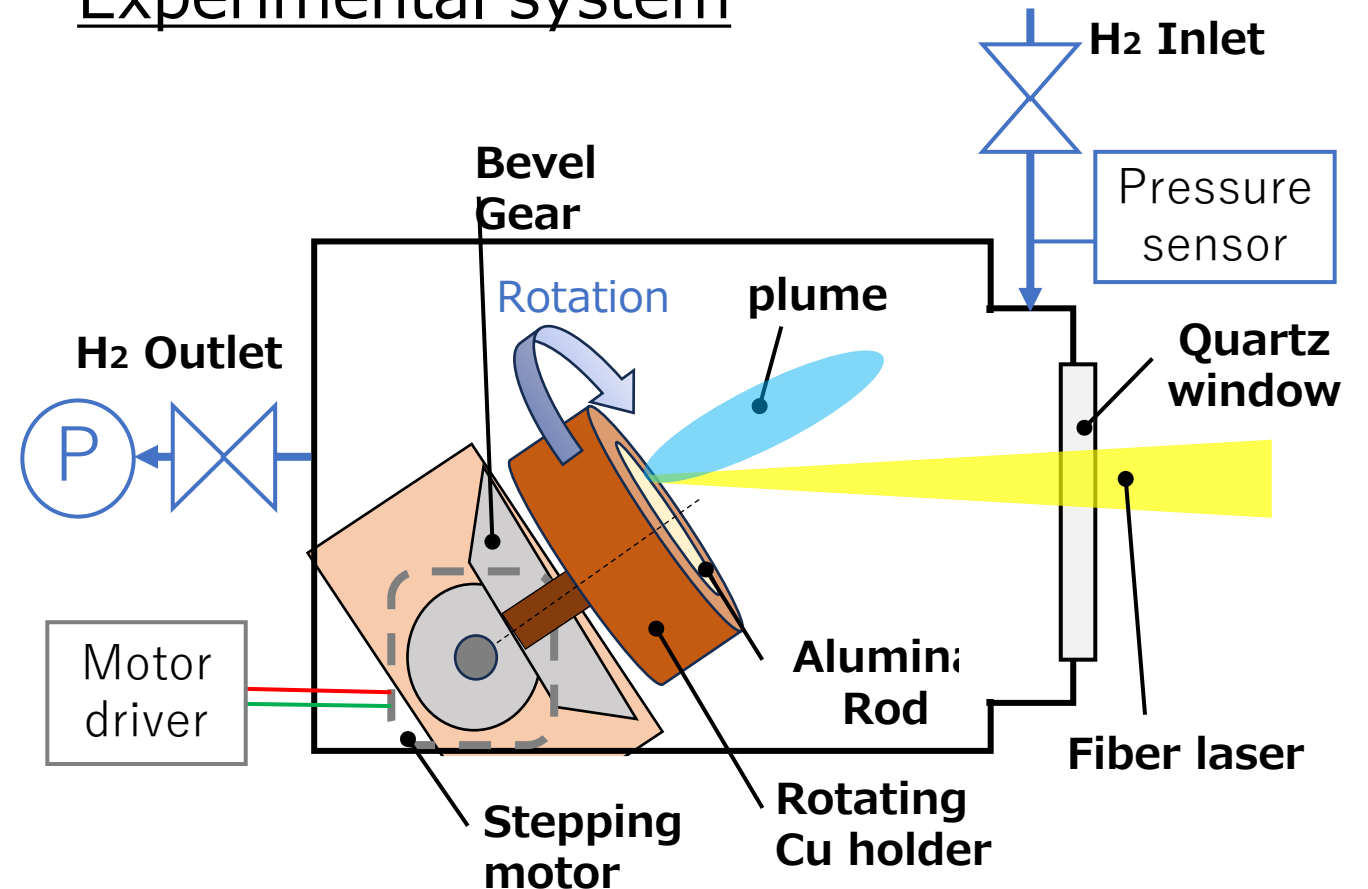


A picture of rod rotation system

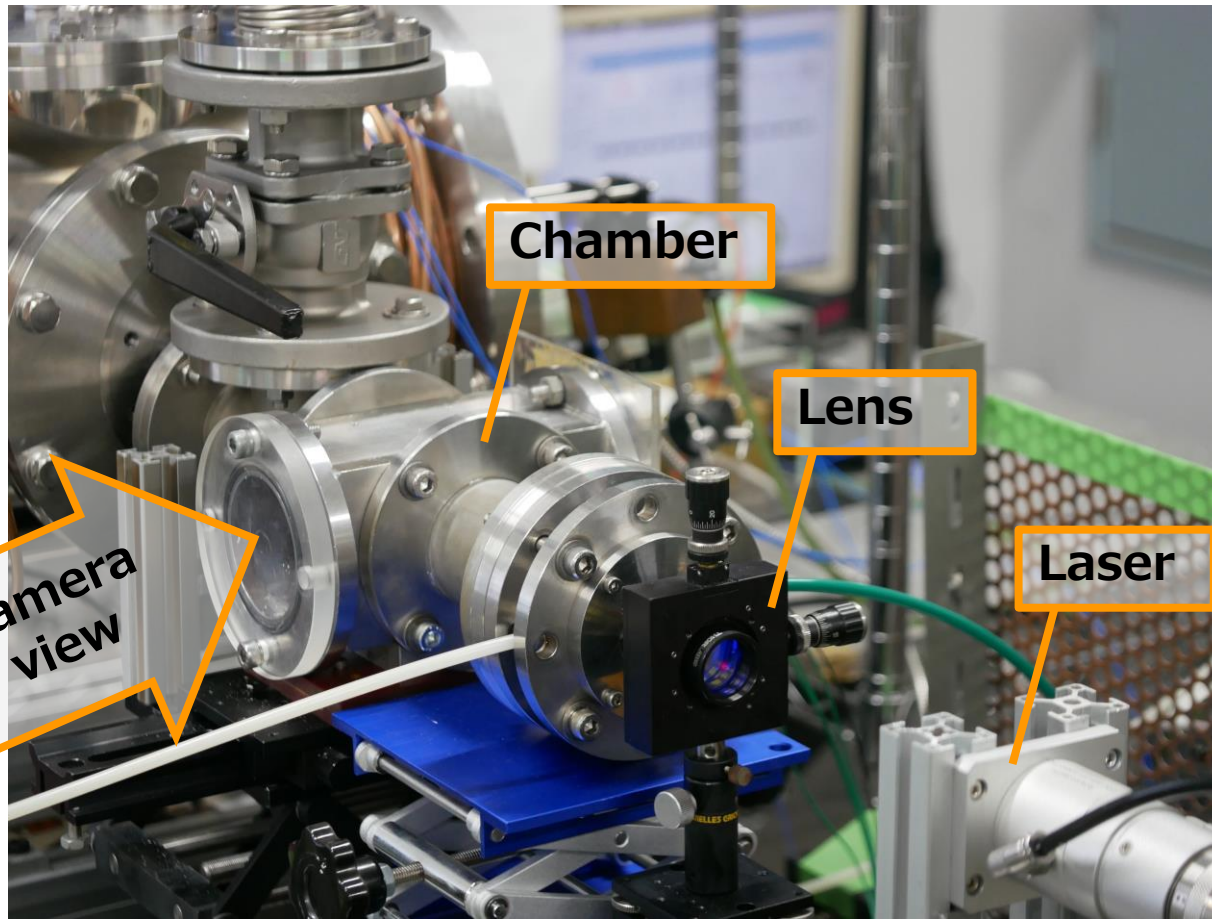
Experimental conditions

- Atmosphere: **H₂ 0.9 atm**
- Laser: Fiber Laser, 1.5 kW, 5s
- Laser spot radius: 1.22mm
- Intensity: 0.32 GW/m²
- spot position from rod center: 6mm
- Rod rotation speed: **4rpm/ 8rpm**

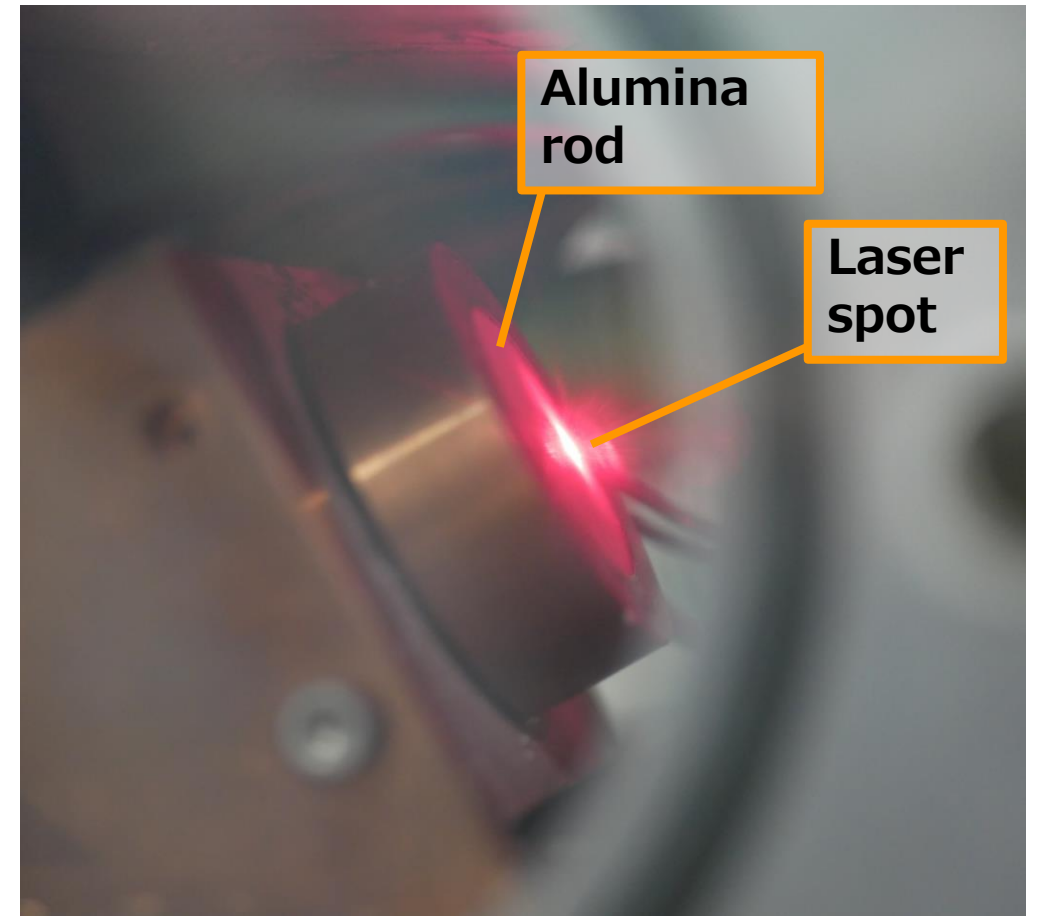
Experimental system



Overview of the experimental system

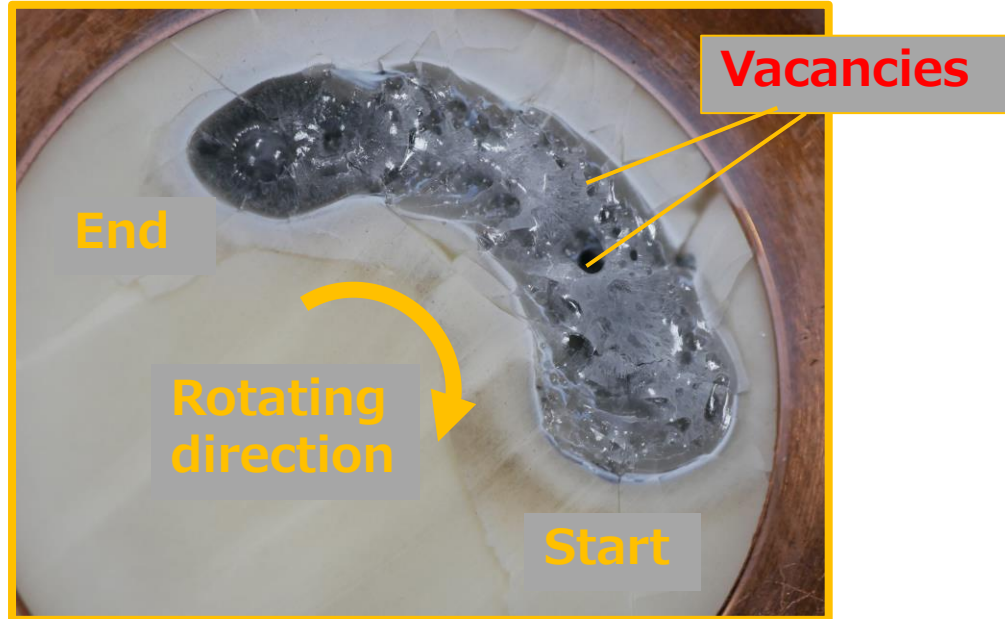


Overview of optics and experimental chamber



The alumina rod set in the chamber

Observation of rod surface after ablation



H₂ 0.9 atm, 4 rpm



Ar 0.9 atm, 4 rpm

- Under H₂ atmosphere, **vacancies were generated entire molten region**
- The molten region swelled in H₂ condition, while it deflated in Ar condition
- Vacancies were not homogeneous, and few vacancies in areas with severe swelling

Inside of the rod after ablation in H₂ atmosphere

- Split the rod and check the cross section

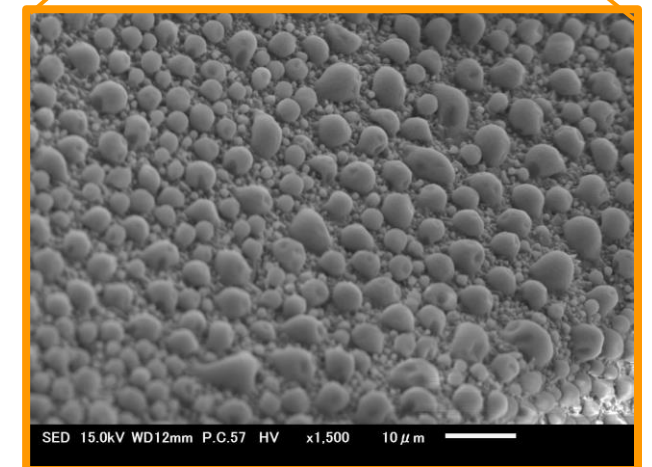
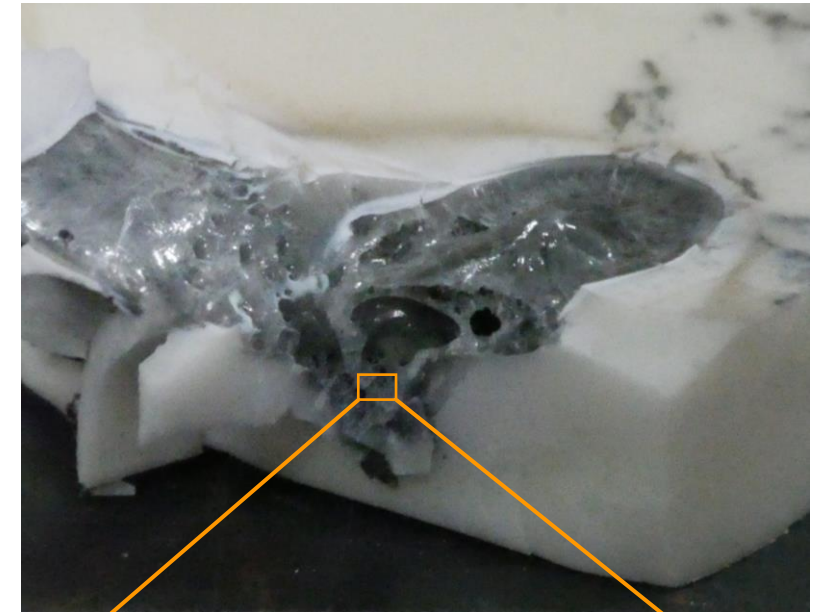
→ Cavities are generated behind the swelling molten region



Reason why the number of vacancies on the surface was not homogeneous

- Electron microscopic observation of the inside

→ Al particles confirmed on inner surface



Measurement of produced Al mass

Item	Single-shot ablation (Previous research)	Continuous ablation 4 rpm	Continuous ablation 8 rpm
Condition	H2 1 atm, Fixed rod, Ablation 1s	H2 0.9 atm, Rotating rod, Ablation 5s	H2 0.9 atm, Rotating rod, Ablation 5s
Al production	14 μg	85 \pm 8 μg	93\pm9 μg
Al production per sec.	14 $\mu\text{g/s}$	17 \pm 2 $\mu\text{g/s}$	19\pm2 $\mu\text{g/s}$

- Compared to the previous study, total production **increased 6.6 times**
- **1.4 times** increase in production per unit time
- **No significant difference** in produced Al mass at different rotation speeds was observed

- **Total Al precipitation increased 6.6 times**
 - The amount of production will be proportional to ablation time, and this method is useful for mass production
- **Al is also formed inside of the rod in H₂ environment**
 - Al particles inside are not good for collection
 - Making the thickness of the melting region thinner
 - Melt again and precipitate on the surface
- **Precipitation mass didn't change with rod rotation speed**
 - Rotation speed can change heating time, cooling rate, etc.
Contrary to the expectation that there is an optimal speed
 - Changes may be seen in a wider range of rotation speed
 - Multiple effects may cancel out each other
 - further experiment is required to determine

- By conducting ablation on a rod moving perpendicular to the laser path in a H₂ atmosphere, Al precipitation mass increased **6.6 times**, and mass per seconds increased **1.4 times**
- In H₂ condition, the inside of the rod also contains Al particles
- In this experiment, there was no significant change in precipitation mass by rotation speed, but further experiments are needed to conclude the relationship between the rotation speed and precipitation mass