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# 連続回転炉および流動層を用いた月土壌の水素還元プロセス

# Hydrogen Reduction of Lunar Soil by Continuous Screw Reactor and Fluidized Bed Reactor

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## 1. Introduction

In-situ resource utilization (ISRU) technology will be important for the engineering purposes for future lunar and planetary sustainable exploration and development. ISRU technology requires the use of locally derived materials since transportation from the earth requires much time, cost, and labor. ISRU will provide production of water, oxygen, helium-3, metals, and other materials.

In these materials, water and oxygen are the most important in early human exploration phase. Over 20 processes of oxygen production on the moon have been proposed [1)]. Among these processes, oxygen production employing hydrogen reduction is the most feasible process [2)]. Oxygen can be produced from the reduction of lunar soil by hydrogen as shown in **Fig. 1**. Reaction (1) is the reduction of ilmenite contained in lunar soil with hydrogen producing water. Oxygen is subsequently produced by electrolysis (2). Hydrogen produced in reaction (2) can be recycled in reaction (1).

$$FeTiO_3(s) + H_2(g) \Rightarrow Fe(s) + TiO_2(s) + H_2O(g) \qquad (1)$$

$$H_2O \Rightarrow H_2 + 1/2O_2 \qquad (2)$$

Reaction (1) is endothermic with 11 kJ/mol under 1,000 ℃. Since the free energy formation in this reaction is relatively low, ilmenite can be easily reduced.

The purpose of this research is to investigate the water-production mechanism by hydrogen reduction using fluidized bed reactor and continuous screw reactor. Understanding of the hydrogen reduction mechanism of lunar soil is important for the mission of utilizing the lunar soil.

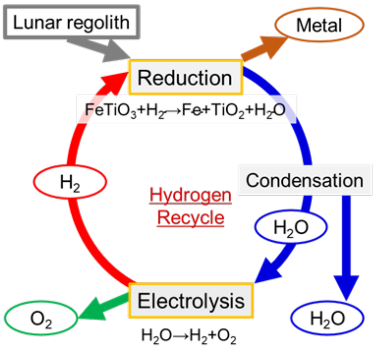

**Fig. 1**　Schematic diagram of hydrogen reduction system for lunar soil

## 2. Continuous Screw Reactor

### 2.1 Experimental setups

A schematic diagram of the continuous screw reactor fabricated in this study is shown in **Fig. 2**. The sample can be continuously fed from the hopper to the reactor by the rotation of the screw. Hydrogen was introduced as a counter flow in the reactor. The reactor is heated to a predetermined temperature in an electric furnace. Water vapor produced in the reduction is measured as absolute humidity using a moisture meter.

The sample used in the experiments is lunar soil simulant, FJS-1, with similar chemical and mechanical properties of lunar soil. Lunar soil simulant is made by Shimizu Corp., Tokyo, Japan. Lunar soil simulant has the median particle size of 70 μm, bulk density of 1.55x$10^3$ kg/$cm^3$, specific gravity of 2.94.

The total flow rate of hydrogen-argon mixture was 10 L/min with the hydrogen concentration at 3%. The inlet pressure was 300 kPa. The effect of the reduction temperature and the reduction time were varied from 1173-1373 K and 10-26 min, respectively. The reduced samples were evaluated by X-ray diffraction (XRD) analysis, cross-sectional observation by scanning electron microscopy (SEM) and compositional elemental analysis using energy-dispersive X-ray analysis (EDS).

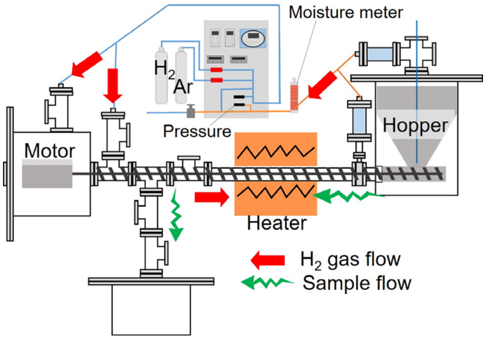

**Fig. 2** Schematic diagram of continuous screw reactor

### 2.2 Experimental results

Reduction experiments were conducted at 1173 K, 1273 K, and 1373 K with reduction times of 10 min. The rate of water production is shown in **Fig. 3**. The reduction of ilmenite in the lunar soil simulant by hydrogen was experimentally confirmed. The steady-state water production rate at 1173 K, 1273 K, and 1373 K was 0.012, 0.017 and 0.010 g/min, respectively. The rate of water production at 1273 K was higher than that at 1173 K due to the increase in the reaction rate at higher temperatures. In contrast, the steady-state water production rate was lower at 1373 K. This is due to the decrease in specific surface area resulting from the melting of alkaline components in the lunar soil simulant as the temperature increases.

The SEM images and EDS elemental mapping after the reduction are shown in **Fig. 4**. Pores, which cannot be found before the reduction, appeared in the cross-section, due to the loss of oxygen bound to Fe and Ti by hydrogen reduction. The cross-sectional observation showed that hydrogen penetrated into the inner part of the particles.

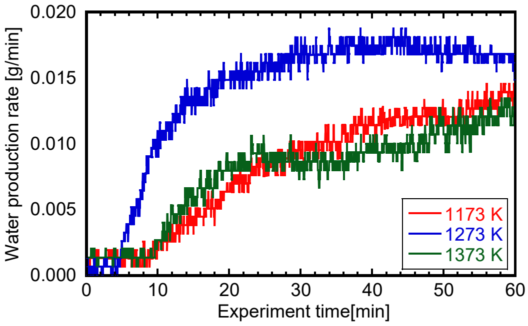

**Fig. 3** Effect of reduction temperature on water production rate in 3vol% hydrogen

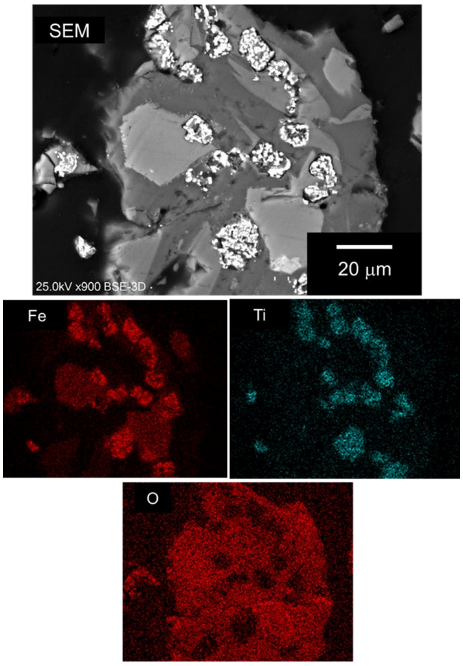

**Fig. 4** SEM photograph and EDS mapping of reduced lunar simulant

## 3. Fluidized Bed Reactor

### 3.1 Experimental setups

A schematic of the fluidized bed for hydrogen reduction system fabricated in this study is shown in **Fig. 5**. The sample powder is placed in an Inconel reactor with an inner diameter of 40 mm. In this experiment, hydrogen is constantly flowing into the system, and the water vapor generated is excluded from the reactor.

Lunar soil simulant used in the experiments was 10 g, and the experimental conditions were 300 kPa at the entrance of the reactor and the reaction temperature from 873 to 1273 K. The flow rate of the hydrogen-helium mixture was fixed at 2.0 L/min. Uniform flow was obtained by the fluidization under the same conditions. The hydrogen concentration was controlled in the range of 20-100% by varying the gas mixing ratio.

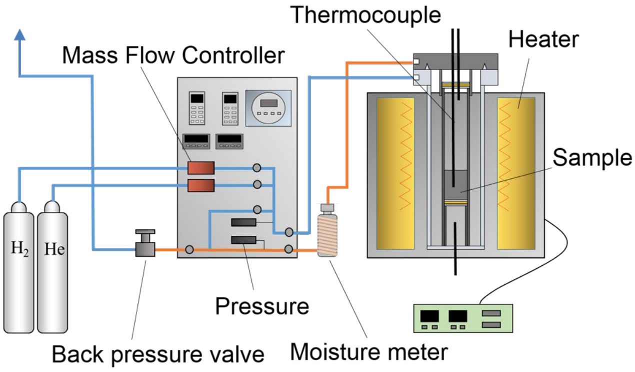

**Fig. 5** Schematic diagram of fluidized bed reactor

### 3.2 Experimental results

**Figure 6** shows the rate of water formation by hydrogen reduction at different reaction temperatures. The maximum water production rate is reached immediately after the start of the reaction and decreases thereafter, and the maximum water production rate increases at 1073 K and 1173 K due to the increase in reaction rate at higher temperatures. In contrast, the maximum water production rate decreases at 1273 K. This is thought to be due to the melting of alkaline components contained in the simulant of the lunar soil as a result of the increase in temperature and the decrease in specific surface area. Similar to the water production rate, the cumulative water content was highest at 1173 K.

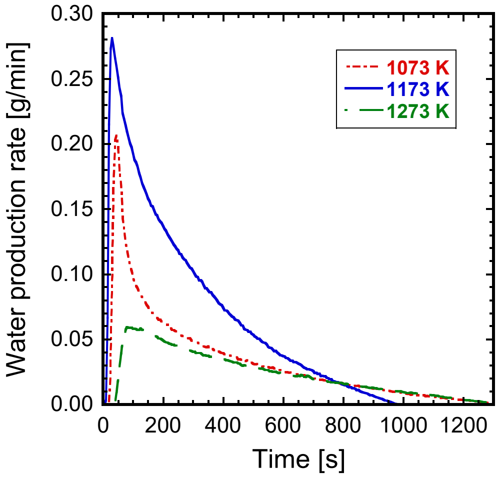

**Fig. 6** Effect of reaction temperature on water production rate

## 4. Conclusions

Hydrogen reduction of lunar soil simulants by a fixed bed reactor has drawbacks of a decrease in the reaction rate due to temperature distribution and uneven filling of the sample. One of the processes to solve these problems is the fluidized bed. Hydrogen reduction experiments have been conducted on lunar soil simulants of 100 g by a fluidized bed reactor. Although the fluidized bed gives uniform temperature distribution and high efficiency in the reaction, gravity and the powder characteristics have a significant effect on the fluidization. In addition, continuous operation is difficult for the fluidized bed reaction system.

In this study, a continuous reactor has been built for hydrogen reduction of lunar soil simulants. The continuous screw reactor can handle a wide variety of samples for continuous operation. The reaction efficiency is high due to the large contact area of the sample as it passes through the reactor while rotating.

The developed system will be used for hydrogen reduction of metal oxides with the aim of industrial application. Production of oxygen-deficient metal oxides are expected to be used as highly functional materials in various fields.

## References

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2) R.A. Briggs and A. Sacco. Jr: J. Mater. Res, **6** (1991) 574.