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Development and Application of Lunar Sample Unsealing and Processing Equipment

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Abstract. The main goal of exploring outer space is to collect samples of extraterrestrial objects and return samples to Earth. Our country is going to launch the Chang'e-5 Lunar Probe Task to collect lunar samples and bring them back to Earth for sample analysis. Unsealing the vacuum lunar sample container and protecting the sample with high-purity nitrogen at the same time become problems urgently. This paper introduces one kind of equipment for unsealing and processing the vacuum lunar sample container in Chang'e-5 Lunar Probe Task. This equipment is divided into four parts: transfer chamber, operating chamber, unsealing chamber and purification unit, which can unseal samples with a vacuum of 10⁻³Pa and keep lunar samples in an environment protected by high-purity nitrogen with a water concentration less than 30ppm and an oxygen concentration less than 20ppm.

1. Introduction

Since the 1960s and 1970s, the United States, the former Soviet Union and other countries had conducted many lunar sampling missions and collected a large number of lunar samples which provided important research basis of major basic and cutting-edge scientific issues such as extraterrestrial resources, cosmic evolution, material structure, and origin of life. China has also proposed a three-step strategy for lunar exploration^[1], and will launch Chang'e-5 Lunar Probe before 2020 for lunar sample sampling mission.

Before the lunar dust samples are collected, they have been existed in vacuum environment on the lunar surface for billions of years. When the samples return to the earth, they contact the earth's atmosphere and chemical reactions may occur. For example, the iron in the samples reacts with the oxygen in the air to produce iron oxides, minerals in the sample and water in the air react to form clay, etc.^[2]. Chemical reactions affect the characteristics of the lunar sample and are of a great obstacle to the subsequent in-situ lunar sample analysis.

After sampling and sealing process is completed, how to unseal the vacuum lunar sample container and process the unsealed sample with protection have became the urgent problem.

In response to the above problems, an equipment for unsealing and sample processing of lunar samples has been developed. It can unseal lunar samples and collect rare gases released by lunar samples in vacuum environment. It can also protect lunar samples in high-purity nitrogen environment when perform collection, registration, description and other process.

Apollo Lunar Sample Lab 2.

Through the Apollo mission to the moon, the United States brought back more than 382 kg (842 pounds) lunar samples from the lunar surface.^[3-6] After the lunar samples were brought back by



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astronauts in vacuum environment, they were first sterilized and cleaned in lunar sample laboratory's equipment. ^[7]The first step was unsealing the lunar sample sealing device, and then confirmed samples, classified samples, photographed samples, weighed samples, microscopic samples, analyzed samples and transfered or stored samples. In the operations above, the samples were protected and a pollution-free environment was provided for the samples. The structure diagram of the US lunar sample storage and processing system is shown in Figure 1.The physical map of the US lunar sample storage and processing system is shown in Figure 2.

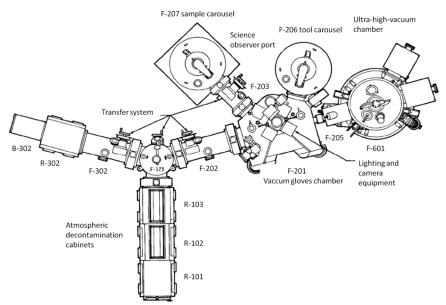


Figure 1. Structure diagram of the US lunar sample storage and processing system



Figure 2. Lunar sample storage and processing system in the US Lunar Sample Laboratory

The core device of the lunar sample storage and processing system is sample processing room, which includes lighting, camera, microscope, observation window, gas analysis device, vacuum gloves, etc. When the sample is under processing, the vacuum can reach up to 1.33×10^{-4} Pa, and the highest vacuum degree in the vacuum chamber for storing lunar samples can reach to 1.33×10^{-7} Pa $\sim 1.33 \times 10^{-9}$ Pa. The equipment system is extremely large, with many complex functions and structural design. It has to maintain a high vacuum state in the long run, which costs a lot. In addition, it has been proved that in high vacuum state, the vacuum gloves are easy to break, which will cause potential dangers in the experiment. After processing samples in Apollo 11 and Apollo 12 task, processing lunar samples in a high-purity nitrogen environment at 1 atmosphere pressure will not pollute the lunar samples, which is a safe and effective way. Figure 3 shows a schematic diagram of the sample processing chamber structure.

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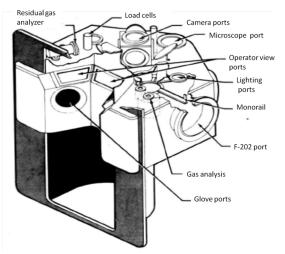


Figure 3. Schematic diagram of the structure of the lunar sample processing chamber

3. Composition of the lunar sample unsealing and processing equipment

Drawing experiences from the United States lunar sample laboratory and taking into account the experimental cost, we designed a subdivision, automatic lunar sample unsealing and processing equipment, which can unseal lunar sample container and collect lunar sample released gas in a high-vacuum environment and process and storage lunar samples in a high-purity nitrogen environment.

The lunar sample unsealing and processing equipment is mainly composed of 4 parts, transfer chamber, operate chamber, unseal chamber and gas purification system as shown in Figure 4.

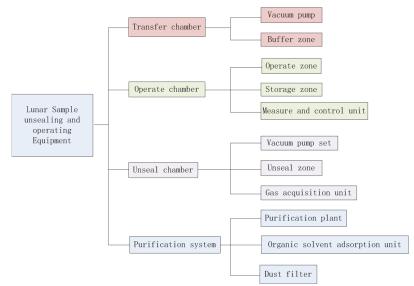


Figure 4. composition of the lunar sample unsealing and processing equipment

The transfer chamber is the entrance and exit of the vacuum lunar sample container, tools, instruments and sample processing equipment. It provides nitrogen spray to clean devices, tools, and instruments. After opening the transfer chamber's door by rotating the door handle, the tools or instruments to be pre-cleaned is placed in the transition chamber. And then turn on the vacuum pump to vacuum the transfer chamber. After pumping to a certain pressure, inject pure nitrogen into the transfer chamber. So that objects in the transfer chamber are pre-cleaned.

After lunar sample is unsealed, the operate chamber provides a high-purity nitrogen environment for separating and weighing the lunar sample to prevent the lunar sample from being oxidized. The bottom of the operate chamber is equipped with a storage area for tools and instruments. When the storage compartment is raised, temporarily unused instruments or tools can be placed on the storage compartment platform. The operate chamber is equipped with a water concentration sensor and an oxygen concentration sensor that can measure the concentration of water and oxygen in real time.

The unseal chamber mainly provides a vacuum environment for unsealing the vacuum lunar sample container. The unseal chamber is evacuated by a vacuum pump. After the internal and external pressure of the vacuum lunar sample container is almost the same, the unsealing mechanism is used to open the vacuum lunar sample container cover. The gas collection unit is used to collect the gas released by the lunar sample for gas composition analysis.

The gas purification system is used to establish and maintain high-purity nitrogen environment and micro-adjust pressure in the operation zone. Before entering the operate chamber, high-purity nitrogen must be purified by purification unit to remove excess water and oxygen to maintain high nitrogen purity. Subsequently, high-purity nitrogen in the operation chamber is circulated by the circulation unit to ensure that the operation chamber is always in a high-purity nitrogen environment during the operation. Sub unit is composed of a dust filter and an organic solvent adsorption device, which can effectively adsorb organic solvents or organic gases that may be generated during the process of sub-sampling and cataloging of lunar samples.

The composition of the lunar sample unsealing and processing equipment is shown in Figure 5. The transition chamber and the operate chamber are connected by the door between two chambers. The operate chamber and the unseal chamber are connected by the door between two chambers. The gas purification unit is connected to the operate chamber through a valve to provide high-purity nitrogen.

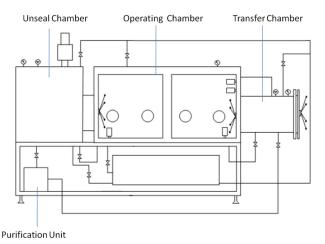
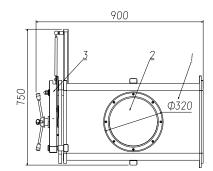


Figure 5. Lunar sample unsealing and processing equipment composition diagram

4. Transfer Chamber

The transfer chamber is a thin-walled square structure, which is composed of a body, a door, a sliding plate, an observation window, a vacuum pump and a pressure measurement unit whose material is 316L stainless steel. The least internal pressure is almost to be 10Pa, and the tray can withstand quality of 30kg. The specific structure of the transition chamber is shown in Figure 6.

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1- Transfer chamber body 2- Observation window 3- Chamber door Figure 6. Structure diagram of the transfer chamber

When using the transfer chamber, first rotate the door handle and open the transfer chamber's door, and pull out the sliding plate. After placing all the lunar unsealing devices, tools and instruments on the sliding plate, close the transfer door. Turn on the vacuum pump to pump out air in the transfer chamber, and stop pumping when the pressure in the transfer chamber is lower than 10Pa. High purity nitrogen in the operate chamber is filled into the transfer chamber, and the items in the transfer chamber are sprayed. After the above air extraction and blowing operations are repeated three times, the pre-cleaning operation is completed, and the door between the t transfer chamber and the operate chamber is opened to transfer the items to the operate chamber.

Since the items in the transfer chamber directly enter the operate chamber, it is necessary to take measures to avoid pollution to the high-purity nitrogen environment in the operate chamber. The most effective way is to perform several air extraction and inflation operations on the transfer chamber, so that the water concentration in the transition chamber to be lower than 30ppm, and the oxygen concentration to be lower than 20ppm, before mixing gas. At this time, the number of pumping times becomes the focus of the design.

When pumping a vacuum, the pressure in the transfer chamber is pumped from 0.1MPa to 10 Pa. After the first pumping, the pressure in the chamber is calculated according to formula 1:

$$P_a = \frac{F}{S} \tag{1}$$

Where:

P_a- Pressure in the transfer chamber, P_a=10Pa;

S- Internal surface area, S=1.2246m²;

F- Force on the inner wall, in N.

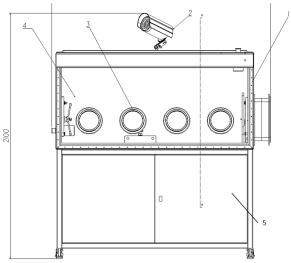
According to formula 1, F=12.246N. According to formula F=mg, the gas mass in the transfer chamber is calculated to be 1.2246kg.At this time, the gas composition in the transfer chamber is the same as the atmosphere, the oxygen concentration is 2095ppm, and the water concentration is 300ppm. The mass of gas in the chamber is 1.2246kg, and the mass of oxygen in the chamber is $m_{oxygen1}=0.2566$ kg, and the mass of water is $m_{water1}=3.674\times10^{-4}$ kg.After the first pumping is completed, fill the transfer chamber with high-purity nitrogen to 10^{5} Pa. In the high-purity nitrogen, the water concentration is 1ppm, and the oxygen concentration is 1ppm. The oxygen mass in the transfer chamber is $m_{oxygen2}=0.01246$ kg, the water mass is $m_{water2}=0.01246$ kg.The total mass of oxygen is $m_{oxygen3}=m_{oxygen1} + m_{water1} + m_{water2} = 0.0128274$ kg, and the total mass of oxygen is $m_{oxygen3}=m_{oxygen3}=0.26906$ kg. At one atmospheric pressure, the total gas mass M= 0.1246×105 kg. The calculated oxygen concentration is 21.6 ppm and the water concentration is 1.03 ppm.After purifying the transfer chamber for the second time, the total mass of oxygen in the transfer chamber is $m_{oxygen4}=0.01249$ kg, and the total mass M = 0.1246×10^{5} kg, the water concentration is 1 ppm, and the oxygen occentration is 20.01246 × 10^{5} kg, the water concentration is 1 ppm, and the oxygen $M = 0.1246 \times 10^{5}$ kg, the water concentration is 1 ppm, and the oxygen $M = 0.1246 \times 10^{5}$ kg, the water concentration is 1 ppm, and the oxygen $M = 0.1246 \times 10^{5}$ kg, the water concentration is 1 ppm, and the oxygen $M = 0.1246 \times 10^{5}$ kg, the water concentration is 1 ppm, and the oxygen $M = 0.1246 \times 10^{5}$ kg, the water concentration is 1 ppm, and the oxygen concentration is 20.01246 × 10^{5} kg, the water concentration is 1 ppm, and the oxygen concentration is 20.01246 × 10^{5} kg, the water concentration is 1 ppm, and the oxygen concentration is 20.01240 ppm.

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Therefore, after the transfer chamber is purified for three times, the water and oxygen concentration in the transfer chamber can be guaranteed to be less than 20 ppm.

5. Operating Chamber

The operating chamber is mainly composed of chamber body, chamber door, crawling mechanism, storage chamber, observation window, vacuum gloves, camera unit, lighting unit, pressure, temperature, water and oxygen concentration detector, and camera unit. The specific structure is shown in Figure 7.



1- Chamber body 2- Camera unit 3- Vacuum gloves 4- Observation window 5- Storage chamber Figure 7. Schematic diagram of operate chamber

In order to avoid contamination of the lunar samples by the materials in contact with the samples, 316L stainless steel is selected for the operating chamber body. Due to sealing requirements of the operating chamber, and the internal water concentration and the oxygen concentration requirements, a mushroom like sealing ring is designed between the chamber body and the glass observation window. This specific sealing structure is shown in Figure 8. After tested, the sealing leakage rate of this structure is less than 1.13×10^{-2} Pa•m³/s.

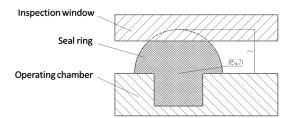


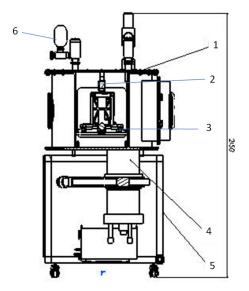
Figure 8 The sealing structure between the operating chamber and the observation window

In order to protect the samples in the chamber, it is necessary to take measures to ensure that the gas leaks from the inside to the outside, the high-purity nitrogen in the chamber mainly flows to the atmosphere outside the chamber, and minimize the leakage of water and oxygen in the atmosphere outside the chamber into the operating chamber. When not working, the gas in the chamber leaks outside slowly. When the pressure drops to 100Pa (relative pressure), the gas purification unit supplies air to the operating chamber to keep the operating chamber at a slight positive pressure of about 300Pa (relative pressure). When the experimenter uses the operating chamber, the air pressure in the chamber rises rapidly. The pump starts to exhaust gas in the operating chamber, so that the pressure in the

chamber drops back to 300Pa (relative pressure). Since the operation in the chamber may cause pressure change, the pressure range that the operating chamber can withstand is $-2000Pa \sim +2000Pa$ (relative pressure), ensuring that experimenter will not be hurt by operating chamber due to pressure changes during operation.

6. Unseal Chamber

The unseal chamber is mainly composed of chamber body, lid unsealing mechanism, vacuum transmission mechanism, positioning mechanism, gas collection assembly, vacuum pumping unit, camera, lighting, pressure measurement, electronic control unit and bracket, etc. The specific composition is shown in Figure 9.



 1- Chamber body 2- Lid unsealing mechanism 3- Vacuum transfer mechanism and positioning mechanism 4- Vacuum pumping unit 5- Bracket 6- Gas collection assembly Figure 9. Schematic diagram of the unseal chamber

In order to avoid contamination, the same as the operate chamber, the material used in the unseal chamber is 316L stainless steel. Since the sampling work is performed on the surface of the moon in the pressure of $1 \times 10^{-9} Pa \cdot m3/s$.^[8-9] After the vacuum lunar sample container is sealed, the internal pressure is about $1 \times 10^{-9} Pa \cdot m3/s$. Taking into account the leakage rate of the sealing container on the way back, the outgassing of the material and the lunar sample, the gas penetration through the material, and the time to return to the ground, it can be estimated that the internal pressure of the vacuum lunar sample container is about to be $10^{-2} \sim 20 Pa^{[10]}$. Therefore, when the unseal chamber is designed, the lowest pressure is $10^{-3} Pa$, and the leak rate of the unseal chamber is less than $1.13 \times 10^{-2} Pa \cdot m3/s$.

When the vacuum lunar sample container in China is sealed and locked, the rotation of the locking disk exerts a vertical downward force on the cover, so that the knife edge of the sample container enters the indium-silver alloy on the cover to realizing vacuum sealing.^[11-12] Therefore, to design the unsealing device, two operations are required: reverse rotate the locking disc of the sealing lid and lift the sealing cover body. We designed two motors to realize those two operations above. The function of motor one is to rotate the locking disk clockwise or counterclockwise in the radial direction and the function of another motor is to lift the cover and the lock disk up in the radial direction. A gearbox is designed to change moving direction of the screw rod and achieve different speed. According to the sealing experiment data, the unsealing torque is about 30 N·m, and the unsealing rotating torque is

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90N•m, which can realize continuous rotation around the axis or set the rotation angle, and can move continuously in the axial direction up to 150mm to ensure the lid of container to be separated.

7. Purification Unit

The gas purification unit is used to establish and maintain a high-purity nitrogen environment in the lunar sample unsealing and processing equipment. The purification unit is mainly composed of a purification unit, an organic solvent adsorption and two dust filter. Its circulation principle diagram is shown in Figure 10.

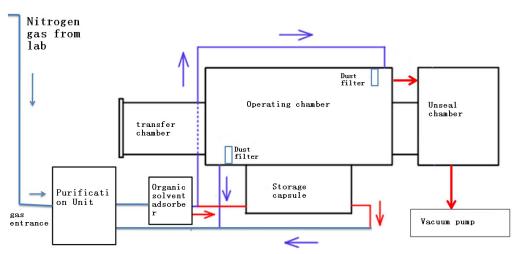


Figure 10. Circulation principle diagram of gas circulation purification system

After passing through the purification unit and the organic solvent adsorber, the high-purity nitrogen flowing from the laboratory is divided into two paths. One air flow goes to the top of the operating chamber. After circulating in the operating chamber, gas flows back to the purification unit from the bottom of the operating chamber. The other air flow goes into the storage chamber from one side and flows out from the other side. When unsealing operation is required, the unseal chamber is vacuumed by the pump unit. When the unsealing process is completed, the high-purity nitrogen in the operating chamber flows into the unsealing chamber through the valve to keep the pressure between the two chambers to be balance. The gas purification unit controls air flow by solenoid valves, and controls the nitrogen filling rate and the pressure in the chamber through the feedback control of the pressure sensor.

The gas purification unit contains a purification column to purify the high-purity nitrogen entering the operating chamber. The water concentration is 1ppm and the oxygen concentration is 1ppm after purification. The dewatering material in the purification column is molecular sieve, and the oxygen removal material is copper catalyst. When the gas purification unit is used for a period of time, the purification column is saturated causing the content of H_2O and O_2 to increase. At this time, the purification column needs to be regenerated. In order to purify the operate chamber continuously, two sets of purification columns are designed. When one set is regenerated, another set can be used for circulation. The organic solvent absorber contains block or granular activated carbon, which can effectively remove organic gas in the nitrogen supplied by the laboratory to avoid contamination. Dust filters are installed at the air inlet and outlet in the operate chamber. 0.2µm particles can filter 99.995%, which can further protect the gas environment in the operating chamber.

8. Conclusion

According to the requirements for cost control and experimental reliability of the third-phase ground application system of our country's lunar exploration project, the lunar sample unsealing and nitrogen

protection proposal China used is different from foreign proposals. Through the investigation of the US Lunar Sample Laboratory and the sealing technology of the vacuum lunar sample container, the first equipment suitable for the unsealing and sample processing of lunar samples in China was developed. The overall configuration of the equipment such as the connection and installation relationship of each part, the working process of indicators is described in details. Through the analysis above, the equipment for unsealing and sample processing of lunar samples can meet the requirements.

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