Seal Design and Test Verification of Lunar Sample Container

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Abstract. One of the key tasks of China's lunar exploration phase three project is to collect lunar sample and return to earth under the condition of unmanned state, and to maintain the original state of the lunar sample, and to ensure the accuracy of the ground data analysis. Reliable sealing of lunar samples is key. This paper designs sealing scheme of lunar sample container, through the iteration test comparison and data analysis, seal structure is optimized, applicable sealing materials are determined. The test verification results show that the leakage rate of lunar sample container is better than that of 5×10^{-9} pa.m^3.s^{-1}, and the sealing performance meet index requirements, which can realize high purity seal of lunar sample and prevent contamination of lunar sample.

1. Introduction
For extraterrestrial sampling missions, the original state of the sample is critical, and it has to be sealed in a high-vacuum environment during the entire mission. Otherwise, after returning to the atmosphere, the entry of trace air will cause pollution and even chemical reaction to the samples, which will lead to the loss of scientific significance to the analysis of extraterrestrial sample and even the failure of the mission. According to the requirements of China's lunar automatic sampling and return mission[1], after the completion of lunar sample collection, lunar sample shall be automatically sealed firmly in sample container in an unmanned state, and can undergo complex mechanical environment conditions during earth-lunar orbit flight. The sample container must remain at a low leakage rate of 10^{-6}Pa.m^3.s^{-1}, ensuring that the lunar sample is original, to ensure the accuracy of the ground data analysis. Based on the requirements of the above tasks, on the basis of the investigation of foreign related technology, this paper through seal structure design and sealing material selection of lunar sample container meet the requirements of China's lunar sampling mission.

2. A review of sample sealing schemes abroad
Since the 1960s, the United States, the former Soviet Union, Japan and Europe have all carried out sampling and returning missions for lunar, asteroid, comet, cosmic dust and other extraterrestrial bodies. Typical missions include the Apollo lunar sampling and return missions and the former Soviet Union lunar exploration sampling and return missions [2-3], the US stardust comet sampling and return missions [4-5], and the Japanese falcon sampling and return missions [6-7]. Among them, the Apollo missions and the former Soviet Union "lunar exploration" sampling many times lunar soil and rock samples and return [8-14], collected about 400kg samples of the lunar. Currently, there are two kinds of sealing methods for the sample of extraterrestrial body after flight verification, rubber seal and metal seal[15]:the former Soviet Union lunar probe series adopts rubber ring seal, the American Apollo lunar probe used metal seal, the stardust comet sampling and the Japanese falcon asteroid probe all adopt rubber ring seal. However, through the ground analysis after the sampling return, the sample seal container returned with different degrees of leakage, resulting in the sample contamination,

affecting the accuracy of the ground analysis data. Based on the experience of sealing lunar sample from abroad, it is necessary to optimize and improve the seal structure and seal material to meet the practical needs of China's lunar sampling mission.

3. Sealing scheme of lunar sample container

Consideration of environmental factors on the surface of the lunar, according to actual structure of the lunar sample container, the sealing scheme adopts the metal knife edge squeezing seal as primary seal way[16-17], rubber ring seal as auxiliary seal way[18-19]for the redundancy seal.

Indium silver alloy and rubber ring are designed on the cover body, and the mouth of the container is machined into a ring knife edge. As in sealed container, need a locking mechanism can provide the vertical downward force to the cover body, which makes the container mouth knife edge blade into the indium silver alloy on the cover body, at the same time, the rubber ring on the cover body squeezed into the sample container of cylinder body and the inner wall of the container form a radial seal[20] to complete container sealing action. At this time, the cone of cover body and the cone of the sample container cooperate to form a mechanical limit, and complete the seal of the lunar sample. The schematic diagram of sealing scheme and sealing process is shown in Figure 1.
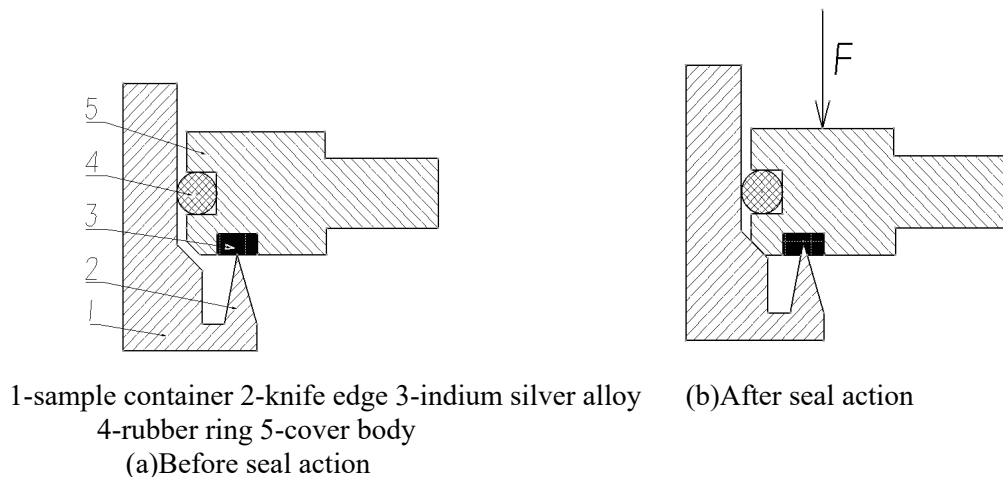

1-sample container 2-knife edge 3-indium silver alloy   (b)After seal action
4-rubber ring 5-cover body
(a)Before seal action
Figure 1. Schematic diagram of sealing scheme and sealing process

4. Seal design of lunar sample container

4.1. Rubber ring seal design

Rubber ring choose good temperature adaptability silicon material rubber ring, its temperature range can reach - 95 ℃ to 220 ℃, shore hardness75±5,fully meet the needs of the task. Due to the need of analysis of lunar sample on the ground, rubber rings are not allowed to use lubricating grease, and dry friction between the rubber rings and the sample container is formed. The locking mechanism of the sample container shall overcome the friction between the rubber ring and the sample container when sealing process. This friction is related to the structural parameters of the rubber ring seal, and the optimal sealing parameters shall be determined through tests[21]. The friction calculation formula of the rubber ring sealing surface is as formula (1) ~ (2):

$$F = \mu.P_r \qquad\qquad (1)$$

$$P_r = \pi.f.d.D.E \qquad\qquad (2)$$

In the formula:
F－friction [N];
μ－coefficient of friction, in dry friction 1～1.2,in formula assign 1.1;

Pr－rubber rings deformation pressure[N];

d－rubber rings cross section diameter [mm];

D－rubber rings inner diameter [mm];

E－rubber rings elastic modulus, associated with the hardness of rubber [MPa];

f－ rubber rings pressure coefficient.

The linear compression ratio of rubber rings can be calculated by formula (3) ~ (4).

$$E = d_2 - \frac{d_4 - d_3}{2} \tag{3}$$

$$E'\% = \frac{E}{d_2} \times 100 \tag{4}$$

In the formula:

d_2－diameter of rubber ring cross section [mm];

d_3－sealing groove bottom diameter [mm];

d_4－cylinder hole diameter [mm];

E －rubber ring linear compression quantity [mm];

E' －rubber ring linear compression ratio.

Through the above formula, the calculation results of linear compression ratio and friction are shown in Table 1.

Table1. The calculation results of linear compression ratio and friction

Linear compression ratio（E′）	Deformation pressure（N）		Friction（N）	
	Max	Min	Max	Min
20%	183.10	84.96	201.41	93.46
16.2%	141.15	65.25	155.27	71.78
13.2%	102.92	47.60	113.22	52.36
9.4%	66.16	30.59	72.78	33.65

According to the calculation results shown in Table1, it can be seen that the higher linear compression rate of rubber ring is, the greater the friction force of rubber ring sealing surface is. Within a certain range of linear compression rate, the higher linear compression rate is, the better the sealing performance is. According to literature [22], the general linear compression rate of rubber ring is between 15% and 25%. Considering the maximum sealing force provided by the locking mechanism and the friction between rubber ring and sample container, the design linear compression rate of rubber ring is about 16.2%.

4.2. Knife edge squeezing seal design

The knife edge squeezing seal technology is a method to form a seal by using a higher hardness knife edge blade into the sealing material with a lower hardness. Knife edge squeezing seal technology needs to determine the material of knife edge, sealing material and matrix material (in this paper is the cover body material), knife edge blade into depth, sealing compression force and other relevant parameters. Since there is no mature theory to calculate relevant parameters, a large number of tests have been carried out in current engineering applications, and relevant sealing parameters of knife edge seal have been determined through test data. Therefore, a large number of tests have to be carried out and these parameters are determined through test iteration.

According to the investigation, three kinds of materials, such as 7075 aluminum alloy, TC4 titanium alloy and 1Cr18Ni9Ti stainless steel, are commonly used in the knife edge material and matrix material verified by space flight at present. The sealing materials are usually pure gold, pure silver and indium silver alloy, among which indium silver alloy has a low melting point and a lower hardness, and also used in the sealing of American lunar sampling project, but the proportion of

indium silver alloy is unknown. Through a lot of silver indium alloy seal material test found that the lower the silver content in silver indium alloys melting point is lower, the lower hardness. Considering the environment temperature of lunar surface and the lightweight design of the sample container, TC4 titanium alloy was selected as the knife edge material and indium silver alloy containing 5% silver was selected as the sealing material. After the sealing material and knife edge material have been determined, then knife edge squeezing sealing experiment was carried out. After each experiment, measuring the sealing leakage rate of the sample container is detected by helium mass spectrometer[23], and the test data is shown in the following Table 2.

Table 2. Test data of knife edge squeezing sealing experiment

Serial number	Matrix material	Sealing material	Knife edge material	Knife edge blade into depth（mm）	Sample container Sealing leakage rate（Pa.m^3.s^{-1}）
1	7075 aluminum alloy	indium silver alloy 95In5Ag	TC4 titanium alloy	1.0	3.7×10^{-5}
				0.8	5.6×10^{-6}
				0.5	8.8×10^{-6}
2	TC4 titanium alloy			1.0	4.5×10^{-5}
				0.5	6.7×10^{-6}
				0.5	7.4×10^{-6}
3	1Cr18Ni9Ti stainless steel			1.0	2.7×10^{-5}
				0.5	5.7×10^{-6}
				0.5	7.7×10^{-6}

As can be seen from above Table 2, the sealing leakage rate of knife edge squeezing seal is no better than that of 10^{-6}pa.m^3.s^{-1}. Through analysis, it is found that: the sealing material is not infiltrated with the matrix material and the knife edge material, so the sealing leakage rate of sample container cannot meet the requirements of 5×10^{-9}Pa.m^3.s^{-1}.

Through many infiltration tests, it is found that the surface of the matrix material can be cleaned and then treated with gold plating or silver plating, which can meet the requirement of indium silver alloy infiltration. Before gold plating or silver plating, the surface of the matrix material needs to be plated metal M first. By controlling the brazing temperature and brazing time, the brazing between the sealing material and the matrix material can be well realized. At the same time, it is found that the infiltration between silver plating layer and indium silver alloy is better than that of gold plating layer and indium silver alloy through experiments, optimized the experiments results as shown in Table 3.

Table 3. Optimized the experiments results

Serial number	Matrix material	Transition layer	plating layer	Knife edge blade into depth（mm）	Sample container Sealing leakage rate（Pa.m^3.s^{-1}）
1	7075 aluminum alloy	Transition layer M	Silver plating	1.0	7.2×10^{-10}
2	TC4 titanium alloy				4.7×10^{-9}
3	1Cr18Ni9Ti stainless steel				5.5×10^{-9}
4	7075 aluminum alloy	Transition layer M1	Gold plating	1.0	3.7×10^{-9}
5	TC4 titanium alloy				3.5×10^{-8}
6	1Cr18Ni9Ti				4.1×10^{-8}

	stainless steel				
7	7075 aluminum alloy	Transition layer M2	Silver plating	1.0	3.5×10^{-11}
8	TC4 titanium alloy				8.0×10^{-10}
9	1Cr18Ni9Ti stainless steel				6.8×10^{-10}

Note: the transition layer metals M, M1 and M2 are the elements of 7075 aluminum alloy materials.

By analyzing the sealing test data in Table 2 and Table 3, the following results can be obtained:

1) Sealing leakage rate is the best when the knife edge blade into depth is 1.0mm;

2) No matter which matrix material, the sealing leakage rate of silver plating is significantly better than that of gold plating;

3) 7075 aluminum alloy matrix material, the transition layer of M2 and silver plating combinations, during blade into depth of 1.0 mm sealing leakage rate is higher than the index requirements of 2 orders of magnitude, the optimal combination way.

According to the above test, the knife edge sealing parameters are determined: the matrix material is 7075 aluminum alloy, the knife edge material is TC4 titanium alloy, the sealing material is 95In5Ag, and the blade into depth of the knife edge is 1.0mm. Matrix material and sealing material brazed joint face was analyzed by using Japanese electron optics corporation JSM-5600LV low vacuum scanning electron microscope (SEM), whose junction plane is as shown in Figure 2.
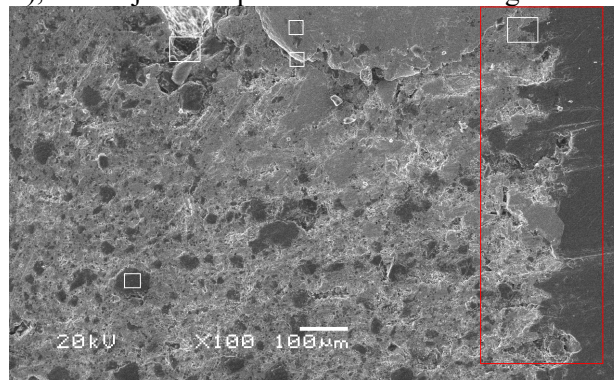

Figure 2. Photo of matrix material and sealing material brazed joint face（red line）

Through the SEM morphological analysis shows that the matrix material 7075 aluminum alloy and the sealing material 95In5Ag are good in the infiltration, the exterior and the interior of the material are tight and uniform, and the density is effective enough to prevent the gas from permeation and diffusion.

5. Test verification seal of lunar sample container

After the determination of the seal structure and material parameters of lunar sample, the 20 times sample container seal test were carried out under the environment condition, the test environment cover the sample container all possible experiences of high temperature, low temperature and vacuum environment. The locking force for sealing generated by locking mechanism movement, that complete the knife edge squeezing seal and rubber ring seal. After the sample container is restored to normal temperature and normal pressure, the sealing leakage rate of sample container is tested. The test data are shown in Table 4.

Table 4. Test data under the environment condition of sample container

Serial number	Environmental conditions during sealing		Sealing leakage rate value （Pa·m³·s⁻¹）	Serial number	Environmental conditions during sealing		Sealing leakage rate value （Pa·m³·s⁻¹）
	Temperature (℃)	Vacuum degree (Pa)			Temperature (℃)	Vacuum degree (Pa)	
1	Normal	Normal	3.5×10^{-9}	11	Normal	$\leq 6.65 \times 10^{-3}$	1.1×10^{-9}

2	temperature	pressure	3.1×10^{-9}	12	temperature	$\leq6.65\times10^{-3}$	8.7×10^{-10}
3			2.7×10^{-9}	13	75℃		7.5×10^{-10}
4			4.2×10^{-9}	14	85℃		8.9×10^{-10}
5	75℃		7.3×10^{-10}	15	95℃	$\leq6.65\times10^{-3}$	1.3×10^{-9}
6	85℃		9.8×10^{-10}	16	110℃		9.5×10^{-10}
7	95℃	Normal pressure	5.7×10^{-10}	17			1.1×10^{-9}
8	110℃		9.8×10^{-10}	18	120℃		1.2×10^{-9}
9	120℃		7.1×10^{-10}	19	-45℃	$\leq6.65\times10^{-3}$	1.3×10^{-9}
10	-45℃		1.1×10^{-9}	20			8.5×10^{-10}

After the lunar sample container seal test, extraction of 4 pieces of indium silver alloy test pieces use PLASTIFORM FULL CASE replicable measuring tool for copy mold of circular squeezing sealing surface morphology, and then use the JT12A Z projector to observe and measure the indentation mold, the morphology image is shown in the Figure 3.

Figure 3. Morphology image of after be squeezed indium silver alloy

The results of the seal test of lunar sample and the measurement of the indentation projector show that the indentation at the blade of indium silver alloy is symmetrical, indentation the shallowest depth is 1.01 mm and the deepest is 1.07 mm. In this state, the maximum leakage rate of seal is 3.5×10^{-9}Pa.m^3.s^{-1}, which is better than the 5×10^{-9}Pa.m^3.s^{-1}required by the index.

6. Conclusions

In this paper, through seal structural design and seal material selection of lunar sample container, and the parameters are improved and optimized by test, which enhances the infiltration of the matrix material and seal material, and improves the sealing performance of the sample container. Through the normal temperature, the high temperature, normal pressure and the vacuum environment sealing test, the sealing performance of the lunar sample container has been tested. It can be concluded that the sealing leakage rate of sample container can maintain 10^{-9}Pa.m^3.s^{-1}order of magnitude, which can be used to seal the lunar sample, to keep the lunar sample in original state, and to prevent the contamination of the lunar sample.

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