Effect of Vacuum Environment on Micro Morphology and Porosity of Lunar Soil Concrete

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Abstract. Lunar soil concrete with sulfur system was prepared. The XRD, XRF and SEM datas of lunar soil concrete under atmospheric pressure and vacuum environment after 28 days of curing were compared. The changes of composition and microstructure during solidification of sulfur lunar soil under vacuum and atmospheric pressure were studied. The changes of porosity under atmospheric pressure and vacuum environment were analyzed and compared. Semi quantitative analysis and evaluation are carried out to compare different experimental phenomena. The results show that the vacuum environment has little effect on the element composition of lunar soil concrete. Sulfur exists in the form of single substance in lunar soil concrete, which has the effect of cementation and bonding. The porosity of lunar soil concrete becomes larger under vacuum environment. It can provide technical reserves for the follow-up utilization of in-situ lunar resources and the construction of lunar scientific research stations.

1. Introduction

The idea of lunar concrete was first put forward by Professor Beyer, L.A. of the University of Pittsburgh in 1985\cite{1}. Subsequently, experts from various countries put forward two ways to realize lunar concrete: one is the "aggregate+cement+water" mode, which is similar to traditional concrete; the other is the "aggregate+sulfur" mode, also known as sulfur concrete. The screened lunar soil and lunar rock are used as aggregates. Lunar surface construction should be combined with the in-situ utilization of lunar resources in order to reduce consumption. It is difficult to obtain water on the moon, and the composition of lunar soil is rich in sulfur, so the latter is very likely to be adopted for lunar surface construction\cite{2-3}.

The lunar high vacuum environment affects not only the formation of materials, but also their basic processes, such as multiphase flow, surface wetting and interfacial tension, both of which will affect their microstructure and porosity. Severe vacuum conditions can also cause material to vent and release volatiles, accelerating material degradation and fluid loss. The net effect of these effects leads to oxygen vacancies in the oxide, resulting in significant colour changes. Therefore, it is significant to study the changes of micro morphology and porosity of lunar soil concrete in vacuum environment\cite{4-5}.

2. Experimental Part

2.1. Reagents and Equipment

Simulated lunar soil, CUG-1, Beijing satellite manufacturing plant Co., Ltd
Sulfur, Chemically purity, Sinopharm Chemical Reagent Co., Ltd
Vacuum test device, temperature: 20±3°C, pressure: 100-5000pa, Beijing Jiaotong University
Muffle furnace, KSL-1700X-A1, Tsinghua University
X-ray diffractometer, D/max-2550, Tsinghua University
X-ray fluorescence spectrometer, ARL PERFORM X, Tsinghua University
Field emission environmental scanning electron microscope, Quanta 200 FEG, Tsinghua University
MatLab, R2021a, MathWorks Company

2.2. Experimental Process
Take sulfur and simulated lunar soil (40g sulfur and 80g simulated lunar soil) with a mass ratio, mix them evenly, and then divide them into two groups, with 60g in each group. Use muffle furnace to sinter the mixtures at 200°C for 5 min, wait for sulfur lunar soil to cool and solidify naturally, and cure them under atmospheric pressure (101kpa) and vacuum environment (100 Pa) for 28 days respectively.

2.3. Testing and Characterization
2.3.1. X-ray diffractometer technology (XRD). After curing, samples were crushed and grinded into powder with particle size less than 400 mesh. 1g was taken from each group, and the crystal structure was analyzed by X-ray diffractometer, in which the scanning method was step scanning and the 2θ angle range is 5°~70°.

2.3.2. X-ray fluorescence spectrum (XRF). After curing, the two groups of samples were crushed and grinded into powder with particle size less than 200 mesh. Each group was taken for tablet pressing treatment, and the qualitative analysis of elements was carried out by X-ray fluorescence spectrometer.

2.3.3. Scanning electron microscopy (SEM). Smash the two groups of samples after curing respectively, and take 3 small block samples of typical section for each group, with the size 5×5×5 mm. The section morphology and porosity of the bulk samples were observed under power (200X) and (500X) power.

2.3.4. Calculation of porosity by binary method. Based on the microscopic morphology photo of sulfur lunar soil obtained by SEM, it is transformed into a binary image by adjusting the brightness and contrast (the image only contains black and white RGB values), and then the RGB values of pixels in the photos are identified by MatLab to obtain the proportion of black and white in the whole image respectively, and the proportion of black pixels is qualitatively regarded as the porosity of the sample, Then, the effect of vacuum environment on porosity was observed.

3. Results and Discussion
Lunar soil concrete is cured under atmospheric pressure and vacuum environment (100Pa) respectively. Figure 1 shows the comparison of lunar soil concrete samples cured under atmospheric pressure and vacuum environment for 28 days.

![Figure 1](image-url)

(a) atmospheric environment  (b) vacuum environment

Figure 1. Concrete samples cured in atmospheric pressure and vacuum environment for 28 days
It can be seen from photos that the sulfur lunar soil has a certain shape and strength after bonding, indicating that the sulfur solidifies the lunar soil before and after melting and plays a bonding role. Compared with the lunar soil specimens cured under atmospheric pressure and vacuum for 28 days, it can be seen that the colour of lunar soil concrete under vacuum environment becomes lighter, and the loose and porous surface of concrete is more obvious, while the colour of lunar soil concrete under atmospheric pressure environment is darker and denser.

**Figure 2.** XRD spectrums of lunar soil concrete under atmospheric pressure and vacuum environment

From the XRD spectrums above, the sulfur does not change in the process of sulfur melting, and exists in the form of sulfur, which plays a role of cementation and bonding. Lunar soil solidifies under the gelation of sulfur, and its main mineral phases are albite (AlNaO₈Si₃), diopside (Al₀.6Ca₁.0Mg₀.7O₆Si₁.7) and augite (Al₀.2Ca₀.9Fe₀.1MgO₆Si₁.8C). Compared with normal pressure and vacuum environment, it can be seen that the phase compositions are similar, only slightly different in pyroxene phase.

**Table 1.** XRF analysis results of lunar soil concrete

| Specimen   | S  | Si | O   | Fe | Ca | Al | Mg | Na | K  | Ti | Else |
|------------|----|----|-----|----|----|----|----|----|----|----|------|
| N-S-1      | 31.78 | 16.92 | 13.19 | 11.42 | 8.28 | 6.97 | 3.60 | 2.71 | 2.46 | 1.54 | /     |
| V-S-1      | 29.29 | 17.85 | 13.12 | 11.49 | 8.62 | 7.47 | 3.56 | 2.89 | 2.56 | 1.70 | /     |

Notes: N-S-1 is lunar soil concrete under atmospheric pressure. V-S-1 is lunar soil concrete in vacuum environment.

Because there is less oxygen in lunar soil, elements are directly used for component comparison. Compared with the XRF characterization analysis results of lunar soil concrete, whether in vacuum environment or atmospheric environment, the content of S element after lunar soil solidification is still the most, accounting for about 30%, which is basically consistent with the content of sulfur in concrete ratio. The characteristic peak of sulfur in XRD test results also shows that sulfur does not participate in chemical reaction.

Figure 3 is the SEM photos of the typical micro morphology of lunar soil concrete. It can be seen from the Figure that the porosity of lunar soil concrete under atmospheric pressure is smaller than that in vacuum environment, the bulk density of lunar soil particles is larger, and the bonding effect of sulfur is more complete. The lunar soil under vacuum environment has poor micro morphology and large pores, which is related to the effect of vacuum environment.
The micro morphology of sulfur lunar soil in vacuum environment within the selected field of view is analyzed by scanning electron microscope. Section 1 is the whole area of Figure 4, Section 2 is the
bright area of red rectangle in Figure 4, and section 3 is the dark area of red rectangle in Figure 4. From the element analysis results of the selected area, it can be seen that the element content of section 2 is basically the same as that of section 1, while Section 3 does not contain sulfur compared with Section 1. Therefore, it can be qualitatively considered that the flocculent material in the bright part of the picture is elemental sulfur, which is unevenly distributed in the mixture of sulfur lunar soil, which will lead to the failure of sulfur to give full play to the melting and bonding effect, which is closely related to the heating mode and whether the mixing is uniform.

According to the electron microscope photos of lunar soil concrete, the binary image of lunar soil concrete at low magnification can be obtained by adjusting the brightness and contrast of the picture, as shown in the Figure.

![Figure 5. Binary images of SEM photos of typical low magnification section of lunar soil concrete](image)

Then, the pixel recognition is carried out through MatLab programming, and the proportion of black and white in the image is recognized respectively. It is approximately considered that the proportion of black is the porosity of the typical section of the material, and the results are shown in the table. It should be noted that in order to ensure the consistency and accuracy of the test pictures, it is necessary to ensure the consistency of the time line taken by each electron microscope photo. Therefore, if the SEM photos of the same material under atmospheric pressure and vacuum want to show an approximate porosity representative image, the changes of contrast and brightness can approximately represent the changes of porosity. From this point of view, This porosity method belongs to the semi quantitative method.

| Specimen | Image magnification | contrast ratio, % | Brightness, % | Black scale, % | White scale, % | Porosity, % |
|----------|---------------------|------------------|-------------|-------------|-------------|------------|
| N-S-1    | 200                 | 100              | 35          | 13.22       | 86.78       | 13.22      |
| V-S-1    | 200                 | 100              | 35          | 18.61       | 81.39       | 18.61      |

For sulfur lunar soil, the porosity under vacuum environment is 18.61%, which is higher than 13.22% under atmospheric pressure, which is consistent with the test results of SEM photos and related to vacuum environment. On the basis of clarifying the solidification mechanism in vacuum environment, it is necessary to clarify the feasibility of sulfur lunar soil concrete as lunar base construction material.

4. Conclusion
The lunar soil concrete with sulfur system was prepared. The difference of composition change and microstructure in the curing process of sulfur lunar soil under vacuum and atmospheric pressure were studied, and the porosity of lunar soil concrete under atmospheric pressure and vacuum was semi quantitatively analyzed and evaluated. The results show that the vacuum environment has little effect
on the element composition of lunar soil concrete. Sulfur exists in the form of single substance in lunar soil concrete, which has the effect of cementation and bonding. Under the vacuum environment, the colour of lunar soil concrete becomes lighter and the porosity becomes larger. Using the gelation of sulfur to bond lunar soil directly is a new way of in-situ utilization of lunar soil, which provides technical reserves for the follow-up in-situ utilization of lunar resources and the construction of lunar research station.

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6. References
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