

Sulfur “concrete” for lunar applications – Sublimation concerns

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Abstract

Melting sulfur and mixing it with an aggregate to form “concrete” is commercially well established and constitutes a material that is particularly well-suited for use in corrosive environments. Discovery of the mineral troilite (FeS) on the moon poses the question of extracting the sulfur for use as a lunar construction material. This would be an attractive alternative to conventional concrete as it does not require water. However, the viability of sulfur concrete in a lunar environment, which is characterized by lack of an atmosphere and extreme temperatures, is not well understood. Here it is assumed that the lunar ore can be mined, refined, and the raw sulfur melded with appropriate lunar regolith to form, for example, bricks. This study evaluates pure sulfur and two sets of small sulfur concrete samples that have been prepared using JSC-1 lunar simulant and SiO₂ powder as aggregate additions. Each set was subjected to extended periods in a vacuum environment to evaluate sublimation issues. Results from these experiments are presented and discussed within the context of the lunar environment.

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1. Introduction

Conventional concrete consists of sand, a coarser aggregate, and a hydraulic binder based on calcium silicate. Added water chemically reacts with the calcium silicate which then effectively sets-up and hardens into the mass known as concrete. Sulfur “concrete” is somewhat a misnomer as very little, if any, chemical reaction occurs between the constituent materials. Basically the sulfur, a thermoplastic material, is melted and mixed with an aggregate after which the mixture poured, molded, and allowed to harden. Regardless, it is an established construction material (ACI, 1993; Crick and Whitmore, 1998; Czarnecki and Gillott, 1990; Head, 1981; Khalooand and Ghafouri, 1992; Lin et al., 1995; Loov et al., 1974; Malhotra, 1975; Okumura, 1998; Nevin, 1998; Vroom, 1998a,b) that has gained wide acceptance, particularly for use in environ-

ments subjected to acids and salts. It exhibits good mechanical properties, low water permeability, and rapid setup times.

The “concrete” composition generally ranges from 12 to 22 wt% sulfur and 78 to 88 wt% of aggregate which can consist of any number of materials including rock sands, minerals, and glasses. The mixture can also contain 5% of a group of compounds termed plasticizers that mitigate cracking as the sulfur goes through, at ~96 °C, a reversible monoclinic–rhombic crystalline phase change. One downside is sulfur’s narrow working range. It melts at ~120 °C and the liquid “stiffens” above 148 °C. Making and applying sulfur concrete is generally constrained between 130 and 140 °C and, obviously, it cannot be used in an environment that exceeds 120 °C.

Sulfur has been found on the moon in the form of the mineral troilite, FeS (Vaniman et al., 1992), and raises the question of reducing the ore to obtain sulfur for construction purposes. This is an attractive alternative to conventional concrete as water, a precious resource, is not required. Reducing troilite to elemental sulfur and using

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sulfur concrete on the moon has been previously discussed (Casanova, 1997; Gracia and Casanova, 1998; Leonard and Johnson, 1988; Roqueta and Casanova, 2000; Toutanji et al.; Vaniman et al., 1992). For our purposes it is assumed that elemental sulfur is available on the lunar surface and sulfur concrete products such as bricks can be made.

Acknowledging that environmental conditions on Earth are relevant to the use of sulfur concrete it follows that lunar applications would entail additional concerns. Lunar temperatures at the equator range from +123 to -180°C with an average of -20°C and, at the poles, from -60 to -220°C . These are extreme temperatures and, perhaps more important, extreme temperature cycles. The moon's environment is also characterized by a lack of atmosphere, generally assumed to be $\sim 1 \times 10^{-12}$ torr (1.33×10^{-10} Pa). This low pressure brings into question sublimation effects in which case a solid material now prefers to be a gas. For example, on Earth solid carbon dioxide (dry ice) when left in a room effectively transforms to a gaseous state.

Some insights into sulfur sublimation can be gained by examination of its pressure–temperature phase diagram. Note that this phase diagram, of which there are several versions, is not exactly understood. However, in general terms, on Earth at 760 torr (1 atmosphere) as the temperature rises from room temperature ($\sim 20^{\circ}\text{C}$) sulfur undergoes a solid phase transition ($\sim 90^{\circ}\text{C}$) and then melts at $\sim 120^{\circ}\text{C}$. In contrast, at pressures on the order of 7.6×10^{-3} torr (1.0132 Pa) solid sulfur transforms directly (sublimates) to a gaseous phase at $\sim 90^{\circ}\text{C}$; the transition temperature continues to decrease with decreasing pressure (Honig and Kramer, 1969). Recall the pressure on the moon, $\sim 1 \times 10^{-12}$ torr (1.33×10^{-10} Pa), and it is realistic to assume that the sulfur in sulfur concrete would be prone to sublimate resulting in a deteriorated, unsound, structure. With that in mind, the intent of this work is to study the effect of a high and prolonged vacuum on pure sulfur and two presumed lunar-like sulfur “concrete” compositions.

2. Experimental procedure

Details of making the concrete samples and the experimental procedure have been described elsewhere (Toutanji et al.; Grugel and Toutanji, 2006). Briefly, pure sulfur was cast into small circular hard plastic molds. Two “concrete” mixtures were prepared having the following approximate compositions: (1) 35 wt% sulfur, 65 wt% JSC-1; JSC-1 is an established lunar simulant soil (McKay et al., 1994) and (2) 45 wt% sulfur–silica binder (25% sulfur and 20% silica, SiO_2) and 55 wt% JSC-1. Small sections, on the order of $10 \times 5 \times 3$ mm, were cut from cubes representative of the two concrete compositions and weighed. Pieces of each composition were placed, large surface area down, in a small aluminum foil weighing pans and then placed in a vacuum chamber which is capable of achieving a vacuum level on the order of 5×10^{-7} torr (6.67×10^{-5} Pa), a level below which sublimation of sulfur at 18 – 20°C is expected.

The pure sulfur samples were tested independently of the concrete samples. Every 5–10 days the chamber was opened and the samples removed for weighing; one composition set was kept out and the remaining were put back in the vacuum chamber. Generally speaking, a vacuum level on the order of 3 – 7×10^{-6} torr (4 – 9.3×10^{-4} Pa) was observed during processing. This scenario was kept up, more or less, for 60 days. The samples removed during testing were weighed and their surfaces photographed. The exposed surface area of each sample was measured and the recorded weight loss from sublimated sulfur was expressed as milligrams per square millimeter.

3. Experimental results

Fig. 1a–c shows representative surfaces of the as-cast pure sulfur and samples that have been exposed to vacuum. First of all, the surface of an “as-cast” sample, Fig. 1a, is not smooth or uniform, the obvious irregularities being shrinkage effects such as cavities and dendrite in-filling with a glazed appearance. After 11 days in vacuum, Fig. 1b, sulfur sublimation clearly reveals the intricate nature of the primary dendrites. Fig. 1c shows a typical surface after 54 days in vacuum. Here the shrinkage cavities have grown and the fibrous nature of the crystals is accentuated. Figs. 2 and 3 are representative micrographs of sample sulfur “concrete” surfaces. Fig. 2a shows the surface of the as-cast, sulfur – 65 wt% JSC-1, sample, Fig. 2b after 8 days exposure to vacuum, and Fig. 2c after 58 days. Fig. 3a–c are similar except that the samples contain SiO_2 and less sulfur. The as-cast surfaces are relatively smooth, clear degradation of the surface is seen after 8 days, more so after 58. The extent of degradation is obviously more apparent in Fig. 4, the concrete samples containing the greatest amount of sulfur.

Fig. 4 plots the measured sulfur weight loss, normalized to milligram per square millimeter of exposed surface area, as a function of time. As seen, the number of points for a given composition decreases with time due to the removal of one sample at each examination period to assess surface degradation. Although scatter within the individual samples is seen, clear trends for the given sample compositions are apparent. The graph shows, as Figs. 1–3 suggest, the greater the amount of initial sulfur in the sample the more that will sublimate away over a given period. The rate of sublimation also decreases with time and one might also infer, particularly for the aggregate containing samples, that they will reach some constant value.

4. Discussion

Fig. 5 shows clear trends in sulfur sublimation as a function of sample material. Still, there is obvious scatter in the weight loss/surface area for a given set of samples. This is attributed to several factors. For instance, the actual surface area measurement of the sample is probably off by

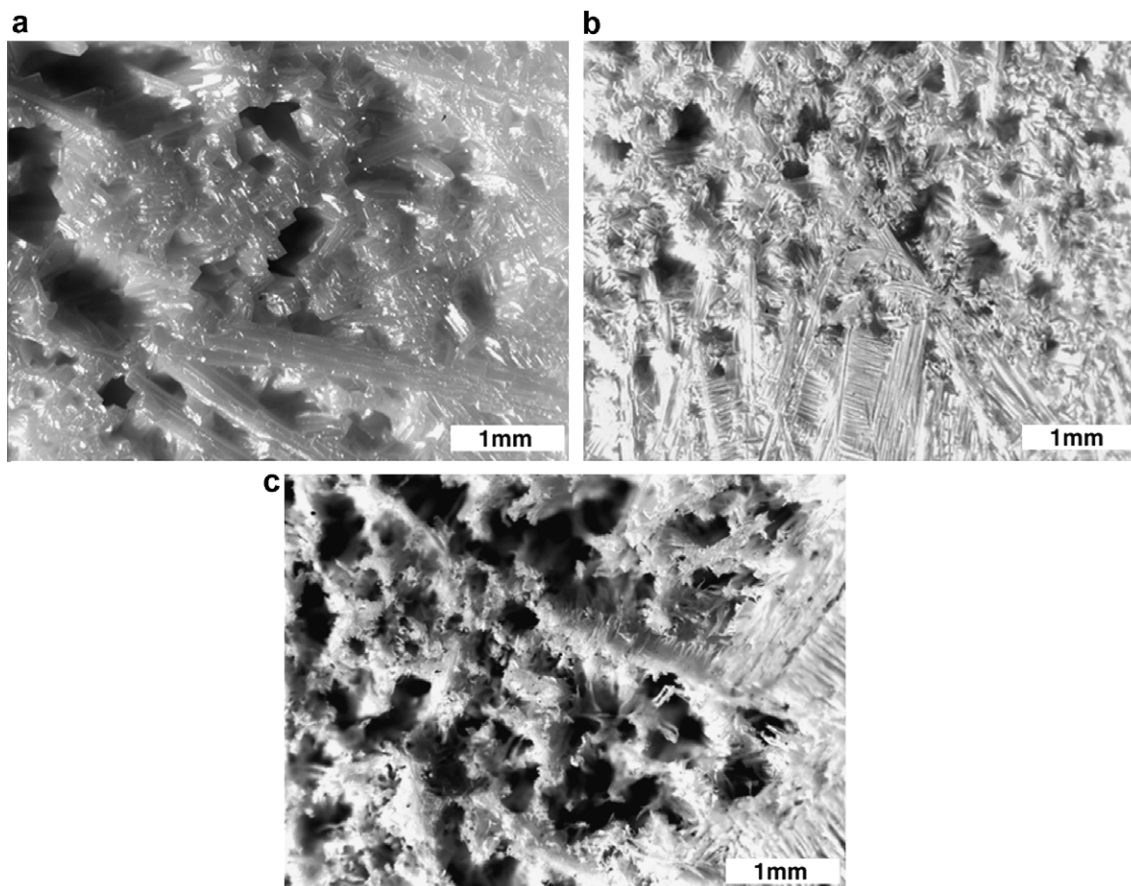


Fig. 1. Photographs of the sulfur sample surfaces: (a) as-cast, (b) after 11 days in vacuum, (c) after 54 days in vacuum. A vacuum level on the order of $3\text{--}7 \times 10^{-6}$ torr ($4\text{--}9.3 \times 10^{-4}$ Pa) was maintained in the chamber.

$\pm 5\%$. In addition, the surface area measurement is strictly a bulk sample consideration. Examination of Figs. 1, 3 and 4 clearly show that the stated surface area does not consider surface irregularities such as ledges and porosity. After vacuum processing the sublimated voids further add to the surface area. Additional scatter in the data could arise from non-uniform distribution of aggregate, more at the surface on a given sample would show less weight loss. It is also assumed that the sublimation rate is uniform on the sample surface and sides; the bottom area in contact with the aluminum pan is ignored. Some insight to this assumption was inadvertently obtained. In Fig. 5 the green¹ diamond datum point representing the sulfur ~ 65 wt% JSC-1 sample after eight days is well out of the band. Examination of the sample showed that after cutting it from the bulk a small “leg” was unintentionally left on the sample (Grugel and Toutanji, 2006). This effectively raised the sample bottom from the pan and exposed additional area from which sulfur could easily sublimate. Furthermore, over time, as the sulfur sublimates more and more of the aggregate is exposed. In this case any now “free” aggregate would sit on the sample surface, effectively

blocking any sulfur underneath from leaving. If this same piece was placed on its side, its large surface area now parallel to a gravity vector, exposed aggregate may well fall off exposing fresh sulfur for sublimation. In summary scatter in the observed data should be expected and is dependent on, at least, aggregate size, shape, distribution, and sample exposure position.

Consider now the sublimation data for the individual sample compositions: (1) pure sulfur, (2) sulfur with 65 wt% JSC-1, and (3) sulfur–silica binder (25% sulfur and 20% silica by weight) with 55 wt% JSC-1. The sulfur–silica binder is a commercially available product known as Gilson Rediron 9000 Capping Compound (ASTM C617 and AASHTO T231). Obviously, as seen, the sample with the most sulfur per unit bulk surface area will sublimate away the most. Given that, weight loss per surface area for pure sulfur appears nearly constant with time whereas the other two compositions give the impression of decreasing slightly with time. The evaporation rate of sulfur can be evaluated using the well-known Hertz–Knudsen equation (Hertz, 1882; Knudsen, 1909; Langmuir, 1913a) given below

$$\Gamma = \alpha_v \left(\frac{m}{2\pi RT} \right)^{1/2} (P^* - P). \quad (1)$$

¹ For interpretation of the references to color in this figure, the reader is referred to the web version of this paper.

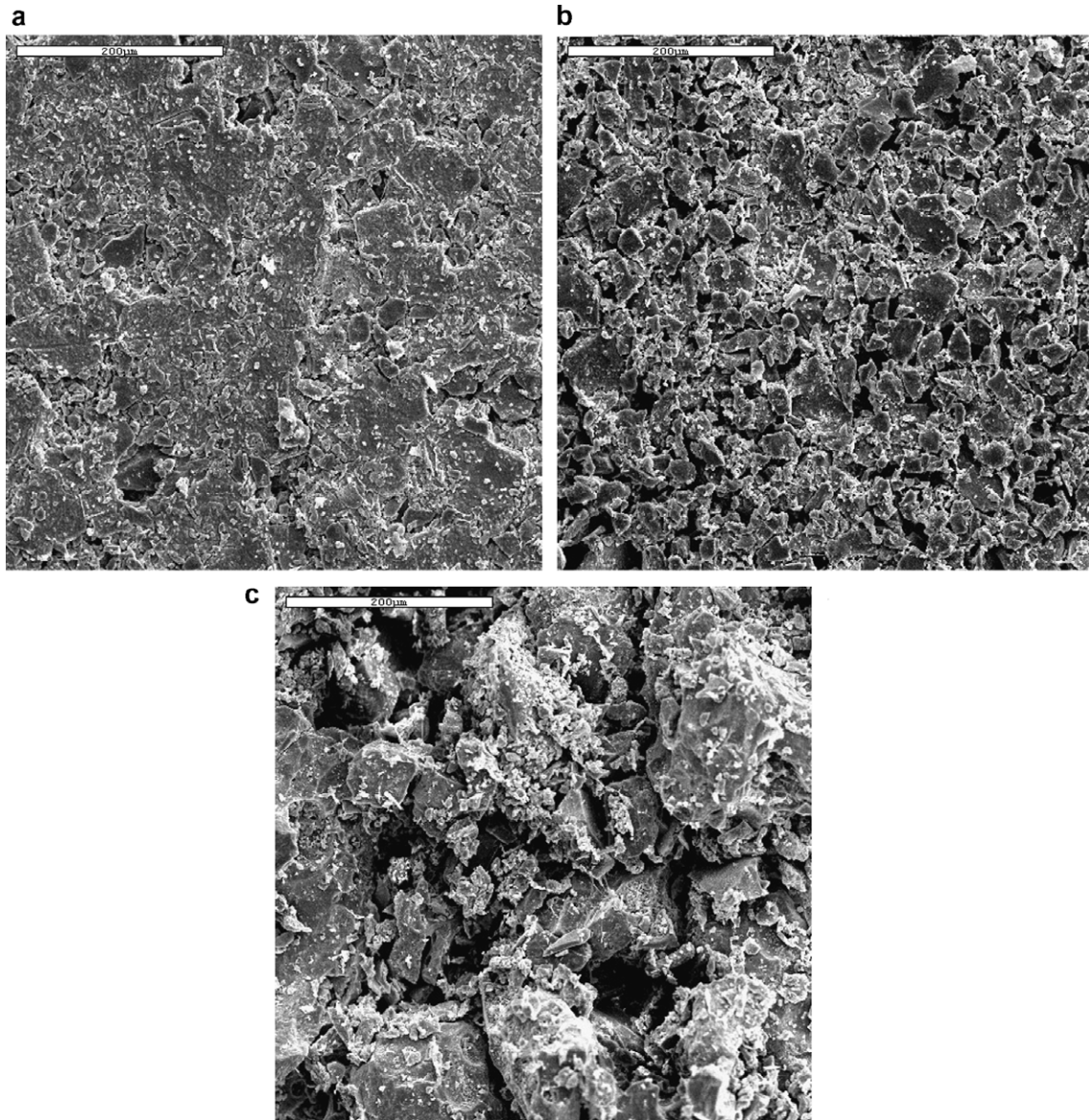


Fig. 2. (a–c) Surface micrographs of the 35 wt% sulfur–65 wt% JSC-1 concrete samples. (a) As-cast, (b) 8 days in vacuum, (c) 58 days in vacuum. Scale bar indicates 200 μm . A vacuum level on the order of $3\text{--}7 \times 10^{-6}$ torr ($4\text{--}9.3 \times 10^{-4}$ Pa) was maintained in the chamber.

Here Γ is the mass evaporation rate, α_v is designated as an evaporation coefficient, m is the atomic mass (32.06 for sulfur), R is the gas constant ($8.31432 \text{ J K}^{-1} \text{ mol}^{-1}$), T is the absolute temperature (293 K for this work), P^* is the partial pressure of the gas phase in equilibrium with its solid (assumed to be 1.8×10^{-6} torr or 2.4×10^{-4} Pa), and P is the ambient (obtainable) pressure of the experimental vacuum chamber (5×10^{-7} torr or 6.67×10^{-5} Pa). The evaporation coefficient, α_v , was introduced and justified by Knudsen as a consequence of experimental results being less than that predicted by Eq. (1) in its basic form. Also known as a “sticking” coefficient, α_v is a measure of the difficulty for atoms to either attach to or be released from a surface, and ascertaining its value is fraught with difficulty (McEachern and Sandoval, 1973). Values of $\alpha_v \approx 1$ were reported for tungsten (Langmuir, 1913b), copper and iron

(Marshall et al., 1937), nickel and nickel oxide (Johnston and Marshall, 1940), and beryllium (Holden et al., 1948). Other values for α_v include 0.17 for Sb at 650 K (Rosenblatt and Lee, 1970) 0.17 for LiF at 1000 K (Howlett et al., 1971), and 0.1–0.4 for the KCl–NaCl system between 913 and 1033 K (Wang et al., 1996). A value of 4.6×10^{-3} was also measured for arsenic at 550 K (Rosenblatt and Lee, 1969). No value of α_v for sulfur could be found so it was, for purposes of calculation, assumed to equal 1.

Using the above values, evaluation of Eq. (1) results in an evaporation rate for pure sulfur of $\Gamma = 2.50808 \times 10^{-8} \text{ g cm}^{-2} \text{ s}^{-1}$ ($2.50808 \times 10^{-7} \text{ mg mm}^{-2} \text{ s}^{-1}$); this ideally translates, at 20 °C, to sublimating away a 1-cm thick layer of sulfur in ~ 955 days. Consequently, assuming a uniform sample surface area of 962.1 mm^2 , one might expect to lose $\sim 2.413 \times 10^{-4} \text{ mg}$ of sulfur per second from the sample. If

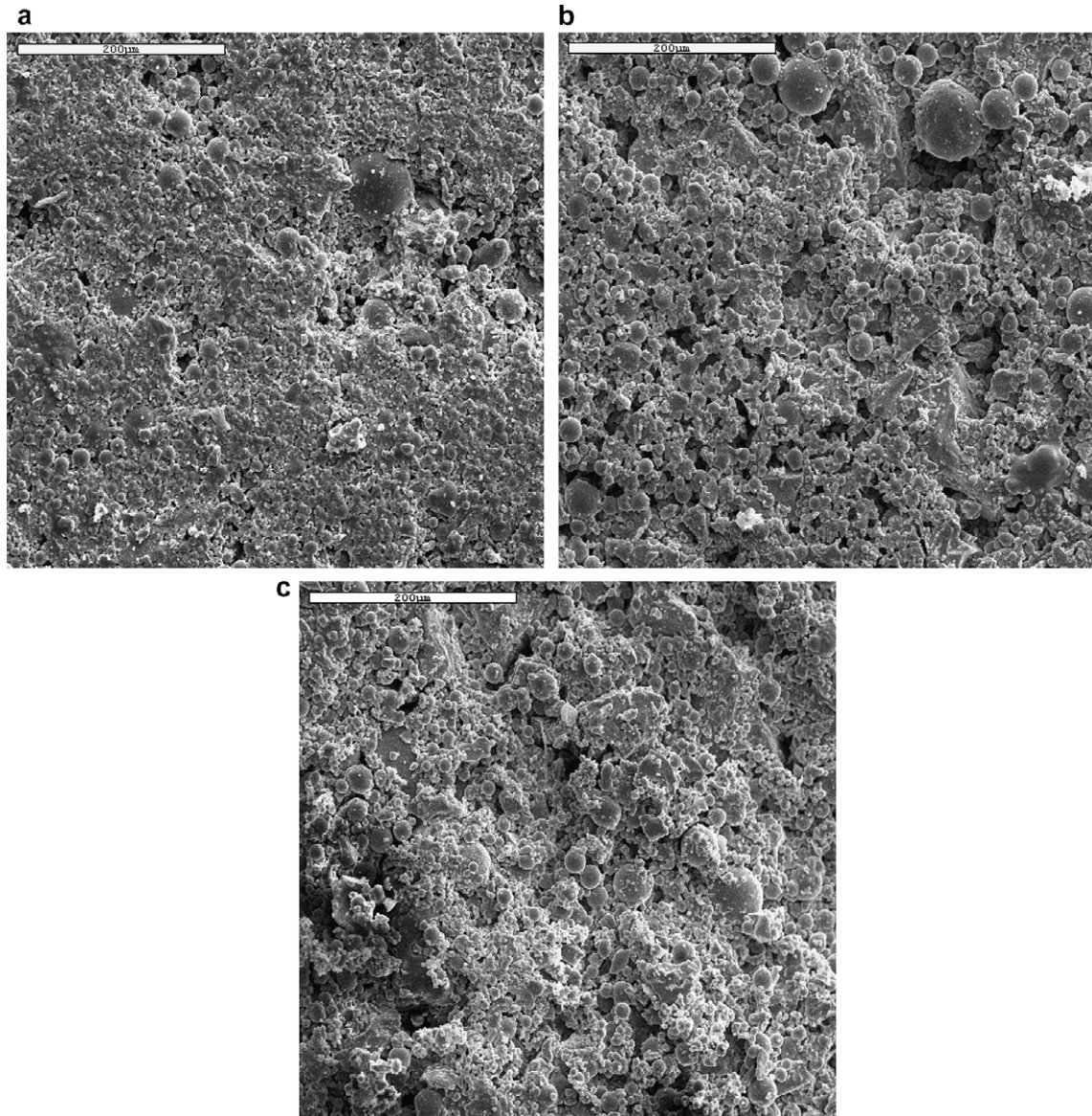


Fig. 3. (a–c) Surface micrographs of the 45 wt% sulfur–silica binder and 55 wt% JSC-1 concrete samples. (a) As-cast, (b) 8 days in vacuum, (c) 58 days in vacuum. Scale bar indicates 200 μm . A vacuum level on the order of $3\text{--}7 \times 10^{-6}$ torr ($4\text{--}9.3 \times 10^{-4}$ Pa) was maintained in the chamber. Spherical particles are the silica grains.

this value remained constant for the length of the experiment (60 days at 18–20 °C) 1.251 g of sulfur would sublimate. Experimentally, the pure sulfur sample that was exposed to vacuum for 60 days lost 0.9329 g.

The Hertz–Knudsen equation for pure sulfur and the experimental conditions is plotted in Fig. 5. Examination reveals good agreement, assuming of $\alpha_v = 1$, for the first 10 or so days after which increasing deviation is seen. The higher initial rate of sulfur sublimation can be inferred from Fig. 1a–c. This as-cast material has the greatest surface area, Fig. 1a, from which to draw atoms. The surface is essentially comprised of a dendritic network, the arms in-filled with the last material to solidify giving it a glazed appearance. This in-filled surface material would contain the majority of any impurities, have a slightly lower melt-

ing temperature, and possibly have greater volatility. This postulate is supported by Fig. 1b which shows emergence and clear definition of the primary (highest melting point) dendritic structure. The surface seen in Fig. 1b has also been subjected to vacuum for 11 days, approximately the time where the sublimation rate is seen to begin decreasing, Fig. 5. This decrease in sublimation rate can be attributed to, at least, two factors. The cavities would still contain the residual solidification product but their deepening now increases the difficulty for sulfur atoms to leave. Second, the surface now consists of well exposed, but randomly oriented, dendrites that grow in specific crystallographic directions. The exposed dendrite arms constitute a mesh that could obstruct released atoms, and it is further expected that the release rate of atoms will differ (e.g. Inaba et al.,

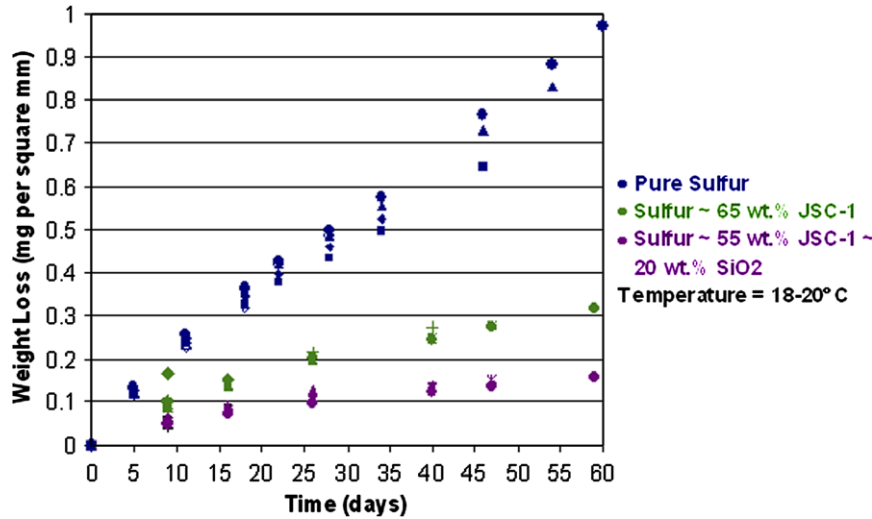


Fig. 4. Plot of the sulfur weight loss for the exposed surface area (mg mm^{-2}) as a function of time.

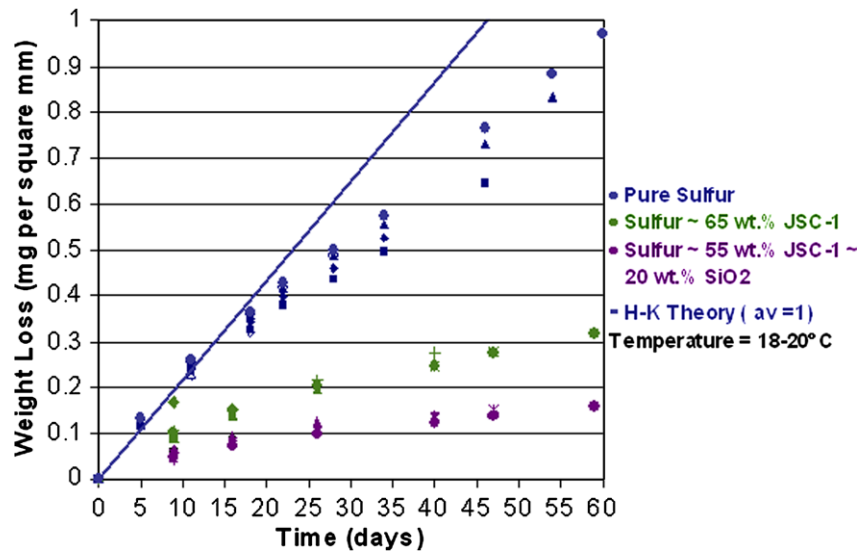


Fig. 5. Comparison of measured weight loss for pure sulfur with the Hertz–Knudsen equation, $\alpha_v = 1$.

2001) for each crystallographic orientation. Thus it is prudent to note that the assumption of $\alpha_v = 1$ may have been fortuitous and that an appropriate evaluation of its true value would entail specifically designed equipment in conjunction with a sulfur single crystal of known orientation. Finally, Fig. 1c (54 days in vacuum) shows increasing size of the shrinkage cavities and further detail of the dendrite structure; the evaporation rate continues to slowly decrease.

Obviously, as they contained less sulfur, sublimation rates were expectedly less for the aggregate mixtures, Figs. 4 and 5. The densities (ρ) of sulfur and SiO_2 are, respectively, 2.07 and 2.32 g cm^{-3} (Weast, 1976–1977); the density of JSC-1 is 2.9 g cm^{-3} (McKay et al., 1994). Converting the sulfur ~65 wt% JSC-1 composition and sulfur ~55 wt% JSC-1 ~20 wt% SiO_2 composition samples to volume percentages finds the former to be 57.0 vol %

aggregate (43% S) and the latter 69.55 vol % aggregate (30.45% S). To a first approximation, reasonably noting that the aggregate material does not sublimate, volume fraction factors, v_f , (0.43 and 0.3) can be placed in conjunction with the evaporation coefficient; the Hertz–Knudsen equation with this correction factor included is plotted for the two mixtures in Fig. 6. Again, early fit with the data are seen but the theory soon over predicts the experimental results. This is expected, particularly in view of Fig. 5. Initially the sample surface is relatively smooth with little, if any, of the aggregate exposed. More and more of the aggregate is exposed as sublimation proceeds. This effectively reduces the amount of exposed sulfur, assuming the aggregate does not fall away, which over time leaves less to sublimate through an increasingly difficult path. Fig. 7 shows the surface of two samples that were exposed to vacuum for 60 days. On the left is the sulfur ~65 wt% JSC-1

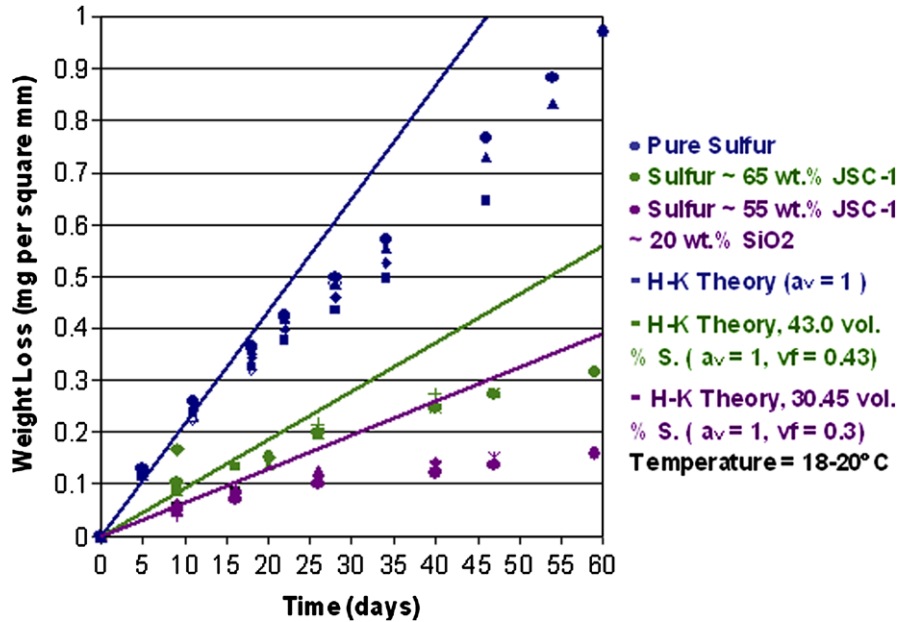


Fig. 6. Comparison of the measured weight loss for pure sulfur and the sulfur concrete samples with the Hertz–Knudsen equation, $\alpha_v = 1$ and with volume fraction, v_f , correction factors.



Fig. 7. Photograph of two samples exposed to vacuum for 60 days. (Left) Sulfur ~35 wt% JSC-1. (Right) Sulfur ~55 wt% JSC-1 ~20 wt% SiO₂.

sample and on the right is sulfur ~55 wt% JSC-1 ~20 wt% SiO₂. The surface discoloration is a consequence of mechanically removing the vacuum affected material. Although the sulfur sublimed the aggregate particles remained cohesively in place, e.g. Figs. 2 and 3. However, they could be easily removed by a disturbance as mild as gentle tapping. The vacuum exposed surface of the sulfur ~65 wt% JSC-1 sample (Fig. 7, left) is the reddish-brown triangular shape located at the central right. The darker brown region surrounding it is the material underneath and was easily exposed by a small air jet. The lighter stripe along the left side denotes the region where the brown material was gently scraped away exposing hard and intact material. The exposed surface of the sulfur ~55 wt% JSC-1

~20 wt% SiO₂ sample (Fig. 7, right) is the wider strip of brownish-orange on the right side. The lighter strip on the left is where the loosely cohesive material was gently scraped away. The surface area of the samples is approximately 6 × 11 mm. Over a period of 60 days, from Fig. 6, the samples should have, respectively, left and right, lost 0.02178 and 0.01056 g of sulfur. For a surface area of 66 mm² these values translate to sulfur thicknesses of 0.159 and 0.0773 mm. Recall that the sulfur only accounts for ~43.0 and ~30.45 vol % in these samples. When the aggregate is included the overall volume fractions of the affected sample are, respectively, 24.47 and 16.75 mm³. This translates to thicknesses of 0.37 and 0.254 mm. In summary, the calculated thicknesses of the vacuum affected zones are reasonably representative of what was experimentally seen. What has been verified, at least for these samples and conditions, is that the affected depth is a function of the aggregate volume fraction. Put another way, assuming that the sulfur loss rate for the concrete samples becomes constant as suggested by Fig. 6, it would take ~4.4 years to sublimate to a depth of 1 cm in a sulfur ~65 wt% JSC-1 brick versus ~6.5 years for the sulfur ~55 wt% JSC-1 ~20 wt% SiO₂ composition. This, again, is drawn from data where the bulk of the exposed aggregate remained on the sample surface.

The experimental results and analysis presented above were confined to room temperature, i.e., ~20 °C. Lunar temperatures vary considerably from this (–230 to 130 °C) and can significantly affect sublimation rates. Utilizing the Hertz–Knudsen equation in conjunction with the vapor pressure versus temperature curve (Honig and Kramer, 1969), the calculated curves shown in Fig. 8 plot the time to sublimate away a 1-cm thick sulfur layer.

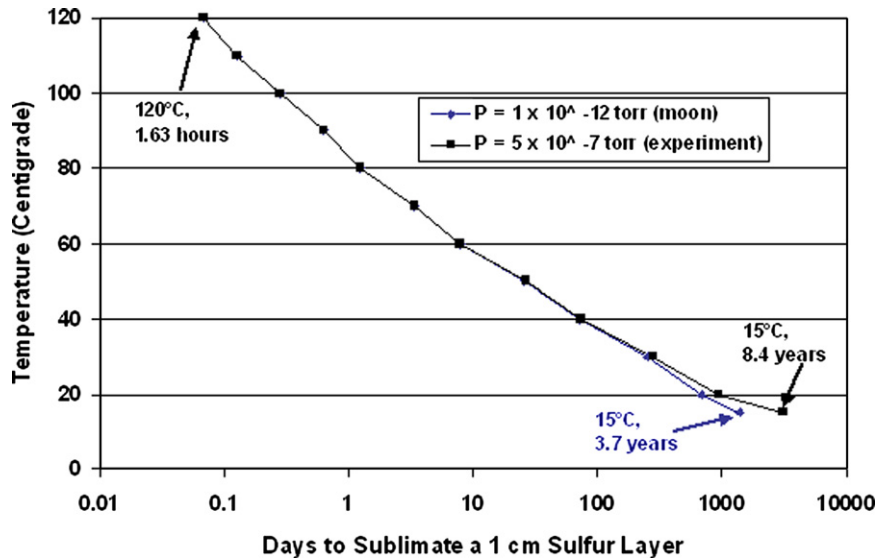


Fig. 8. Calculated plot showing the effect of temperature on the time needed to sublimate a 1-cm thickness of pure sulfur.

Two pressures (moon and experimental chamber) are considered between temperatures ranging from 15 to 120 °C, the latter being near sulfur’s melting point. At ~15 °C the effect of a much lower lunar pressure is seen and a 1-cm layer is calculated take 3.7 years to evaporate in comparison to 8.4 years which is calculated for the ground-based experimental chamber. Above ~30 °C the two curves are essentially indistinguishable. However, the consequence of increasing temperature becomes obvious. What might be tolerable at 15 °C is clearly not at ~120 °C where it is calculated to take less than 2 h to sublimate a centimeter layer.

The above work considers vacuum effects over time on pure sulfur and two sulfur concrete compositions at a temperature of about 20 °C. Other temperatures, or even

cycles, can easily be implemented on the system to mimic lunar conditions and ascertain evaporation rates. What is difficult to reproduce is the lunar pressure, which is on the order of 10^{-12} torr (1×10^{-10} Pa). This raises the question of whether or not the results presented here, for the given temperature, are applicable to a lunar environment. Recall that the partial pressure of sulfur (P^*) in equilibrium with its solid (at 20 °C) was assumed to be 1.8×10^{-6} torr (2.4×10^{-4} Pa) and that the vacuum chamber was able to reach an ambient level (P) of $\sim 5 \times 10^{-7}$ torr (6.67×10^{-5} Pa). For a constant P^* and T the evaporation rate (Γ), Eq. (1), should increase as P decreases. Thus, is it reasonable to assume that a laboratory chamber capable of 5×10^{-7} torr is representative of the lunar environment, which is smaller by some five orders of magnitude? The cal-

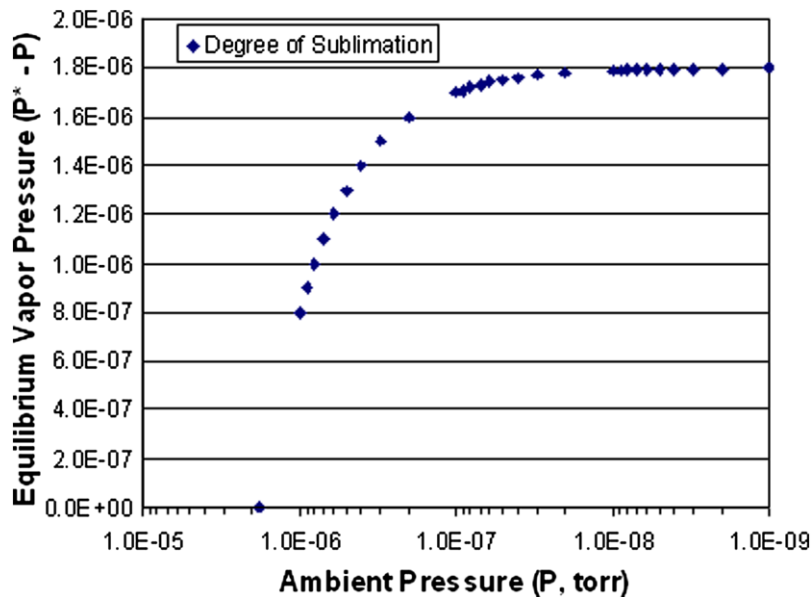


Fig. 9. Calculated plot showing the role the ambient pressure plays in regard to the equilibrium vapor pressure.

culated results shown in Fig. 9 gauge the role vacuum level plays on the sublimation rate by plotting $(P^* - P)$ as a function of the environmental (ambient) pressure. At $P = 1.8 \times 10^{-6}$ torr $(P^* - P)$ is zero but as P decreases the designated degree of sublimation rapidly increases, being upper bound by a maximum of 1.8×10^{-6} torr when P equals zero. For an environmental pressure of 5×10^{-7} torr this corresponds to a $(P^* - P)$ value which is 72.2% of that achievable. Decreasing the pressure to 1×10^{-7} torr accounts for 94.4% and 1×10^{-8} torr relate to 99.4%. While it might be prudent to increase the measured sublimation rates by $\sim 28\%$ it is apparent that the much lower lunar atmosphere of 10^{-12} torr would only negligibly contribute.

5. Conclusions

Pure sulfur and two sulfur concrete mixtures were prepared and placed in a vacuum environment (capable of 5×10^{-7} torr) at $\sim 20^\circ\text{C}$ for 60 days. Periodic weighing of the samples revealed a continuous weight loss due to the sublimation of sulfur. The sublimation rate was evaluated with the Hertz–Knudsen equation assuming an evaporation coefficient of 1.0. Reasonable agreement over ~ 10 days was found for pure sulfur and for the concrete mixtures when the volume fraction of added aggregate was considered. Subsequent discrepancies were attributed to non-uniform surfaces, impurities, and continual exposure of aggregate material. The difference in volume fraction of aggregate between the two concrete samples (57% and 69.6%) was reflected in the depth of affected (sublimated) material. Here, for the given conditions, it was predicted that, respectively, 4.4 and 6.5 years would be needed to sublimate away a 1-cm deep layer from the concrete samples. Sulfur sublimation rates were predicted to change dramatically over a temperature range from 15 to 120°C . Finally it was shown that the much lower vacuum on the moon would contribute only slightly more to the sublimation rates determined from the ground-based experiments.

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