# Performance of "waterless concrete"

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ABSTRACT: Waterless concrete consists of molten elementary sulfur and aggregate. The aggregates in a lunar environment will be lunar rocks and soil. Sulfur is present on the Moon in Troilite soil (FeS) and, by oxidation of the soil, iron and sulfur can be produced. Sulfur concrete specimens were cycled between liquid nitrogen (~-191°C) and room temperature (~21°C) to simulate exposure to a lunar environment. Cycled and control specimens were subsequently tested in compression at room temperatures (~21°C) and ~-101°C. Test results showed that due to temperature cycling, the compressive strength of cycled specimens was 20% of those non-cycled. This reduction in strength can be attributed to the large differences in thermal coefficients of expansion of the materials constituting the concrete which promoted cracking. Similar sulfur concrete mixtures were strengthened with short and long glass fibres. The lunar regolith simulant was melted in a 25 cc Pt-Rh crucible in a Sybron Thermoline high temperature MoSi2 furnace at melting temperatures of 1450 to 1600°C for times of 30 min to 1 hour. Glass fibres and small rods were pulled from the melt. The glass fibres were used to reinforce sulfur concrete plated to improve the flexural strength of the sulfur concrete. Beams strengthened with glass fibres showed to exhibit an increase in the flexural strength by as much as 45%.

# **1 SULFUR/REGOLITH CONCRETE**

Sulfur, a thermoplastic material, is melted and mixed with an aggregate after which the mixture is poured, moulded and allowed to harden. Sulfur concrete is not concrete in the traditional sense because little chemical reaction happens between the components. It is considered well established as a building material to resist corrosive environments, or in areas where there is high acid or salt content. Sulfur concrete usually contains 12-22 weight % sulfur and 78-88 weight % aggregate in its composition. The sulfur could consist of 5% plasticizers. The aggregate may include coarse and fine particles. Sulfur is generally expected to melt at about 119°C and stiffen above 148°C; therefore, the sulfur and aggregate must be mixed and heated at a temperature between 130°C and 140°C. Thus, the environment in which sulfur concrete is used must not have a temperature greater than the melting point of sulfur (Vaniman et al. 1992).

Commercial use of sulfur "concrete" on Earth is well established, particularly in corrosive, e.g., acid and salt, environments. Having found troilite (FeS) on the Moon raises the question of using extracted sulfur as a lunar construction material, an attractive alternative to conventional concrete as it does not require water. Table 1 is a record of the amount of sulfur found during some of those missions (Gibson & Moore 1973).

Table 1.	Sulfur	abundance	in	Apollo	soil	samples.

Apollo mission	Sulfur abundance µgS/g			
Apollo 14	706-778			
Apollo 15	517-712			
Apollo 16	474-794			

Sulfur can be extracted from lunar soil by heating the lunar soil to over  $1100^{\circ}$ C in a vacuum environment resulting in the release of sulfur in the form of SO<sub>2</sub> and H<sub>2</sub>S. Then by using the Claus process, pure elemental sulfur and water are created, as shown in Equation (1). The Claus process takes the hydrogen sulfide and the sulfur dioxide and passes them through a heated catalyst bed at a temperature of 323°C. The catalyst that is usually used is bauxite (Al<sub>2</sub>O<sub>3</sub>), which has been found in the Apollo lunar soil samples (Lunar Sourcebook 1991)

$$SO_2 + 2H_2S \rightarrow 2H_2O + 3S \tag{1}$$

Test results have shown that the compressive strength of sulfur concrete is higher than that of hydraulic concrete. The addition of silica to sulfur concrete increases the compressive strength by as much as 26% (Toutanji et al. 2006). Mechanically, silica is very similar to the silicate minerals in the lunar regolith. This addition of silica to the sulfur concrete decreases the required sulfur content but also improves the mechanical properties of the system.

#### 1.1 Specimen preparation

Cubes measuring 50.8 mm (2 in) were cast made of sulfur concrete (Fig. 1). Two different sulfur concrete mixtures were made: a) 35% purified sulfur and 65% JSC-1 aggregate by mass and b) 20% silica, 25% purified sulfur and 55% JSC-1 aggregate by mass. To examine the effect of severe environmental conditions on the concrete mixtures, the effect of the addition of silica, which is the main element of the lunar regolith composition, was also studied.



Figure 1. Waterless lunar simulant specimen.

The specimens were subjected to light freezethaw exposure (room temp. to  $\sim$ -27°C) and severe freeze-thaw exposure (room temp. to  $\sim$ -191°C). The specimens were tested in compression after each exposure using a Universal Hydraulic Testing Machine. The size of the light exposed specimens were cubes measuring 50.8 x 50.8 x 50.8 mm and were subjected to 50 cycles. For the severe freeze-thaw specimens, the cubes measured 25.4 x 25.4 x 25.4 mm were subjected to 80 cycles. A minimum of four specimens were tested for each test.

#### 1.2 Results

The cubic blocks that were 50.8 cm on each side were cut into eight 25.4 cm cubes. Cubes were packaged with a k-type thermocouple in sets of eight and put into a Styrofoam container, into which liquid nitrogen (LN2) was poured and allowed to cool and evaporate. LN2 decreased the temperature from room temperature (RT), at about 21°C to about -191°C. The temperature cycle was repeated 80 times. Compression testing took place at a constant crosshead speed of 0.127 cm/minute. Samples of both compositions that were cycled and non-cycled underwent compression testing, one set happening at room temperature, about 21°C, and the other set at about -101°C.

The average maximum compression strength for the non-cycled samples was 35 MPa and 7 MPa for the cycled samples, a 5 times difference. An explanation for the cycled samples' failure at a load 5 times less than the non-cycled samples could be that the de-bonding of the particles of aggregate with sulfur that occurred during the cycling left cracks which weakened the sample before compression testing. There was no difference in behavior between samples tested at -101°C and at 21°C. Samples cycled to -191°C were weaker than at other temperatures, as shown in Figure 2. The concrete did in fact show a transition when it was cycled to -191°C but what is not known is whether -191°C is the definitive temperature or if the transition could have occurred at different temperatures. Although, it is not definitively determined, the rate of de-bonding may have been related to the number of cycles in the plastic range, as well as the heating and cooling rates. Sulfur and aggregate have vastly different coefficients of expansion that causes de-bonding at unknown temperatures, unknown because many properties of sulfur are still unknown such as exactly where sulfur transitions from elastic to plastic behavior.



Figure 2. The max compressive strength exhibited by the cycled and non-cycled samples.

## 2 GLASS FIBRE-REINFORCEMENT

Glass fibre and glass rebar reinforcement are used in a large number of terrestrial civil infrastructure applications. Glass fibres and glass rods are also ideal candidates for use in lunar construction.

Utilization of the lunar regolith for production of fibreglass and glass rods will require knowledge of the glass forming capability of this material. Some work has been performed with lunar simulants. Tucker & Ethridge (1998) studied the fibre forming characteristics of Minnesota Lunar Simulant-1 (MLS-1) and Minnesota Lunar Simulant-2 (MLS-2). MLS-1 simulated the mare composition and MLS-2 the lunar highlands. It was found that MLS-2 was easier to draw into a fibre. However, both simulants led to "fragile" glasses which tended to crystallize quite readily. Addition of boric oxide (8 wt. %) extended the viscosity such that continuous fibres were easily drawn using a fibre pulling apparatus. Glass formation appears to be easier with JSC-1 simulant.

## 2.1 Specimen preparation

JSC-1 lunar soil simulant was melted in a high temperature furnace at temperatures between 1450°C and 1600°C for times of 30 minutes to 1 hour using a 25cc platinum crucible. The crucible containing the melt was placed on a refractory brick and glass fibre was hand drawn directly from the melt using an alumina rod. The fibre was drawn through two felt pads containing a polyamide solution to provide a protective coating for the glass fibre. The coated fibres were then placed in a low temperature furnace and the polymer coating was cured at 200°C for 12 hours. The coating acts as a barrier to atmospheric moisture, which is known to degrade glass strength (Tucker et al. 2006). The drawing process and drawn fibres are shown in Figures 3 and 4. er et al. 2006). The perform mechanisms and low temperature furnace were replaced with a high temperature furnace. The furnace windings are Pt/Rh wire, which gives the furnace a capability of reaching 1600°C. The fibre diameter is controlled by the furnace temperature and/or the take-up reel rotation rate which determines the draw rate. Fibre diameter is controlled primarily through take-up speed, although temperature changes of the glass viscosity can also be used. Coating is achieved by running the fibre through a small cup containing a polymer solution. The polymer is then UV cured before winding.

Plates measuring 101.6 mm x 254 mm x 12.7 mm were cast, as shown in Figure 5, consisting of sulfur powder and JSC-1. By mass the mixtures were 35% sulfur and 65% JSC-1. Long and short glass fibres produced from lunar regolith simulant, were used to reinforce the sulfur concrete. The percentage of the glass fibre in the mix was about 1% by mass. The diameters of the short fibres ranged from 3 to 20 micrometers and those of the long fibres were between 0.50 mm and 1 mm. Every plate was divided into three small beams, measuring 33.8 mm x 254 mm x 12.7 mm. Plain sulfur-regolith concrete and the sulfur-concrete reinforced with glass fibres were tested for load and deflection, using a four-point bending test setup shown in Figure 6.



Figure 3. Hand-drawing glass fibres.







Figure 4. Glass fibres drawn from lunar regolith simulant. To aid in drawing continuous glass fibre, a KC135 fibre-drawing apparatus has been refurbished (Tuck-



Figure 6. Four-point bend test setup.

### 2.2 Results

Adding glass fibre, derived from lunar regolith simulant, significantly increased the overall strength of the concrete as shown in Figure 7. As compared to the control specimens (SC), specimens strengthened with long hand drawn glass fibres (SCLGF) and specimens strengthened with short glass fibres (SCSGF) have exhibited an increase of more than 40%. This is a preliminary data and more tests are currently conducted to study the effect of the glass fibres on the ductility and strain energy capacity of the sulfur concrete.





#### 3 CONCLUSIONS

To determine the structural integrity of sulfur concrete, sulfur concrete specimens were subjected to cycling between room temperature to -191°C. Results showed a significant reduction in strength. compared to non-cycled samples. This reduction in strength can be attributed to the fact that sulfur and aggregate have vastly different coefficients of expansion that cause de-bonding at different temperatures. Regolith derived glass fibres can also be used with sulfur concrete, also made with in-situ regolith. The addition of fibre showed a significant improvement in the strength. Both short glass and long glass fibres were shown to increase the strength of sulfur concrete by as much as 40%. More tests are currently being conducted to study the effect of the glass fibres on the ductility and strain energy capacity of the sulfur concrete.

### **4** ACKNOWLEDGEMENTS

The authors are grateful to the Marshall Space Flight Center and the In-Space Fabrication and Repair Element for their support of this work and the financial support of NASA grant NNM05AA22A. The authors also would like to thank Drs. Ed. Ethridge and Dennis Tucker of NASA-Marshall Space Flight Center for providing the glass fibres.

# **6** REFERENCES

- Gibson, E.K. Jr, Moore, G.W. 1973. Carbon and sulfur distributions and abundances in lunar fines. *Proceedings of the Fourth Lunar Science Conference*, Vol 2: 1577-1586.
- Lunar Sourcebook, 1991. A user's guide to the Moon. (ed). Heiken G.H, Vaniman, D.T. & French, B.M. (eds), Cambridge University Press, New York.
- Toutanji, H. et al. 2006. Glass fiber reinforced sulfur concrete for habitat structures. Proceedings: ASCE Habitation 2006: Conference on Habitation Research and Technology Development. Orlando, FL.
- Tucker, D.S. and Ethridge, E.C. 1998. Processing glass fiber from Moon/Mars resources. Space 98.
- Tucker, D. et al. 2006. Production of glass fibers for reinforcement of lunar concrete. Proceedings: 43<sup>rd</sup> American Institute of Aeronautics and Astronautics (AIAA). Reno, NV.
- Vaniman, D. et al. 1992. Uses of lunar sulfur. Second Conference on Lunar Bases and Space Activities of the 21<sup>st</sup> Century, Wendell Mendell (ed), Lunar and Planetary Institute: 429-435.