

Developing Cementitious Materials Using Lunar Soil Simulant

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Summary: An organic-inorganic nanohybrid of high flexure strength and low permeability is developed using lunar soil simulant and polymer-silicate interphase. The interphase consists of a continuous polyamide 6 phase, exfoliated silicate nanolayers, and dispersed silicate tactoids intercalated by polyamide 6 oligomers. The lunar soil simulant is strongly bonded by the interphase through a two-staged heating and mixing process, forming a multiscale structure with the characteristic lengths ranging from nanometer level to sub-millimeter level. This technique has great potential in developing high-performance space infrastructural materials using locally harvestable resources. A complete report has been published elsewhere.^[1]

Introduction: One of the long term goals of sending human beings to the Moon and the Mars is to stay there, for which temporary outposts and permanent bases must be constructed.^[2] Due to the tight constraint of space transportation capacity, it is highly desirable that the outposts and bases can be built by using locally harvestable resources, such as lunar soils. For instance, it is, theoretically, possible that as lunar soils of different chemical compositions are appropriately mixed together, through complicated heating and curing procedures “lunar cements”, materials that can react with water or other liquid agents to form load-bearing components, can be obtained.^[3] However, to achieve this, massive and energy-consuming “cement plants” must be built on the Moon, before the “lunar cements” are available. Even if this could be done, the “lunar cements”, as any other ordinary cementitious materials, are of low flexure strengths. Their long-term reliability, especially in the vacuum or high/low-temperature environments, is also problematic. Moreover, the availability of water or reactive chemicals necessary for the cementing process is quite limited.

In view of these issues, over the years a few alternative techniques such as direct sintering of lunar soils^[4] and water-free sulfur cements^[5] have been proposed. The main issues related to these techniques include the lack of systematic testing data, the relatively high energy consumption, the relatively poor material properties, and/or the limited availability of resources. Therefore, they are still far from being directly useful for space construction.

Materials Development: In the current study, JSC-1 lunar soil simulant was employed as a close analog to lunar soils. It was developed at The Johnson Space Center based on the analysis of Apollo lunar soil samples, taking into consideration the major chemical

chemical and physical properties such as the chemical composition, the grain size, the inter-particle friction and cohesion, etc. The nanointerphase was prepared through a simplified “one-pot” procedure. At room temperature, monomers, acid, silicate, and deionized water were uniformly mixed together. A trace amount of accelerator was also added. The silicate was used as the precursor. The mixture was thermal- in nitrogen environment at 260°C for 6 hours, during which the layer stacks in silicate expanded and eventually individual nanolayers were exfoliated. The nanointerphase was then cooled down to room temperature, ground into pellets with the size of about 0.5 mm, thoroughly washed by warm water, and finally dried in air. It was characterized using a Bruker AX8 x-ray diffractometer, and the result is shown in Fig.1.

The nanointerphase was then softened, followed by the addition of lunar soil simulant. The nanointerphase content was 15%. The simulant and the nanointerphase were mixed at 120 rpm to reach uniform dispersion and then at 10 rpm to minimize defect density. The resultant material, which will be referred to as simulant-based polymer intercalated/exfoliated (SBP) lunar cement in the following discussion was cooled down to room temperature in air.

To evaluate flexure properties, the SBP lunar cement was hot pressed into smooth sheets with the thickness of 3.2 mm by using a type 3912 Carver hydraulic compression molding machine. The pressure was set to 27.5 MPa and the temperature was 270°C. Flexure specimens, with the width of 10.2 mm and the length of about 24.2 mm, were cut from the sheets using a fresh razor blade. The flexure experiment was performed in a three-point bending setup using a type 5569 Instron machine. The loading rate was 1 mm/min. Altogether 5 samples were tested. The average flexure strength $\gamma = (3/2) \cdot PL / bt_0^2$ was measured to be 74.1 MPa, and the standard deviation was 6.7 MPa, where P is the maximum center-point loading, L is the support distance, b is the sample width, and t_0 is the sample thickness. The fracture surfaces were observed in a FEI Quanta 200 environmental scanning electron microscope (see Fig.2).

Discussion: At the microscopic scale, the material consists of close-packed simulant grains and continuous nanointerphase, as shown in Fig.2. The nanointerphase can be regarded as polymeric membranes in between the simulant grains, bonding them together. The membrane thickness, t , is at the level of a few μm . Note that t is dominated by the nanointerphase content,

c. If the nanointerphase content is high, the simulant-nanointerphase mixture is quite flowable at an elevated temperature, and thus the mixing procedure is easy to control. However, in order to reduce the amount of components that need to be prepared on the Earth, for a lunar cement c must be minimized. According to the characterization results of PIE cements,^[6] as c is lower than 8%, the interphase is insufficient to wet all the inorganic particles. The un-wetted particles would form macrodefects of loosely packed grain clusters, which leads to a significant decrease in flexure strength. Furthermore, due to the changes in rheological properties, a larger inorganic content usually results in a higher energy requirement for heating and mixing, which must be carefully taken into consideration as the power supply for lunar base/outpost construction is limited. It is also important to keep a leeway for the relatively un-investigated space environments; that is, for safety purpose, the nanointerphase content should be higher than the theoretical “optimum” value. In the current study, c is set to 15%. Under this condition, the handling and placing of softened simulant-nanointerphase mixture are straightforward, and the flexure strength is more than one order of magnitude higher than that of ordinary portland cements, which is quite satisfactory for construction applications even for adverse conditions.

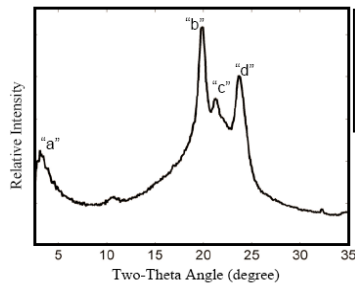


Fig.1 The x-ray diffraction result of the nanointerphase.

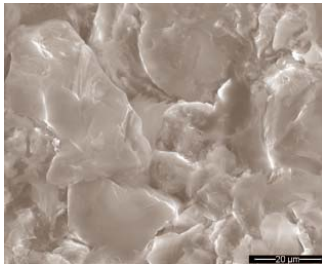


Fig.2 SEM microscopy of the simulant-based PIE lunar cement.

It is possible to further decrease the nanointerphase content without lowering the flexure strength by appropriately control the size gradation of simulant grains. For instance, by using gap gradation protocols, the interphase content can be reduced by nearly 50%. If more accurate continuous gradation protocols are used, c can be even smaller. However, as most of concrete structures on the Earth, the developed lunar cements can be employed as the binding phase for aggregates, such as lunar rocks or coarse lunar soil

grains, to form “lunar concretes”. In a concrete material, the cement binder content is often around 20%, which, for the SBP lunar cement developed in the current study, results in a low nanointerphase content of 3%. That is, to produce 100 parts of space construction materials, only 3 parts of nanointerphase need to be transported from the Earth. Further lowering c would lead to only marginal benefits.

At the nanometer scale, the nanointerphase can be regarded as a polyamide 6 matrix nanocomposite, with the reinforcements of both intercalated silicate tactoids and exfoliated silicate nanolayers. As shown in Fig.1, the nanointerphase is characterized by the x-ray diffraction peaks at the two-theta angles of 4.3° (“a”), 20.5° (“b”), 21.5° (“c”), and 24° (“d”). Peaks “b–d” reflect the semi-crystalline structure of polyamide 6, among which “b” and “d” are for α phase and “c” is for γ phase. In α phase, chain slippage is relatively difficult due to the contoured configuration of monoclinic unit cells. In γ phase, amide groups are twisted from methylene group planes. Their initiation and growth are mainly determined by the cooling condition and the degree of exfoliation of silicate nanolayers, which is reflected by peak “a”. A high peak “a” indicates a high degree of intercalation, and if all the silicate tactoids are delaminated, this peak would entirely disappear. In the current material, the full exfoliated is not achieved, which can actually be beneficial since the multiscale reinforcement mechanism improves strength and toughness simultaneously. The exfoliated nanolayers promote the formation of γ phase and lower the chain mobility; the intercalated tactoids can act as either craze initiation sites or stoppers, bridge stretched fibrils, and increase energy dissipation. Therefore, the mechanical properties of the nanointerphase is much better than that of the neat polyamide 6. These effects are further enhanced as the nanointerphase is compressed into membranes in between the simulant grains. As the characteristic length becomes smaller, the chain alignment is more pronounced, and crazing and shear banding are depressed. As a result, the strength rises. Moreover, the thermal stability and the air/water-proofness can also be significantly improved. The simulant grains also hinder the fracture process by promoting crack bifurcation, causing jerky fracture surfaces, as shown in Fig.2, somewhat similar to the role of the inorganic phase in a nacre.

References: [1] Qiao Y, Chen J, Han A (2007). *Adv. Eng. Mater.*, in the press. [2] Toklu YC (2000). *Proc. Space 2000*, Reston, VA, pp.822. [3] Cox RM (2000). *Proc. Space 2000*, Reston, VA, pp.805. [4] Taylor LA, Meek TT (2005). *J. Aero. Eng.* **18**, 188. [5] Toutanji H, Schrayshuen B, Han M (2006). *Proc. Earth&Space*, League City, TX. [6] Qiao Y, et al. (2007). *Mater. Res. Bull.*, in the press.