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## DIFFERENTIAL THERMAL ANALYSIS OF LUNAR SOIL SIMULANT

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Science and Engineering Directorate

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## TECHNICAL MEMORANDUM

### DIFFERENTIAL THERMAL ANALYSIS OF LUNAR SOIL SIMULANT

#### INTRODUCTION

The use of lunar regolith for the production of structural materials could greatly reduce the cost for construction and long-term habitation of a lunar colony. One lunar product, fiberglass (both discontinuous and continuous forms), promises ease of manufacture and wide applicability.<sup>1</sup> Continuous fiberglass can be utilized as reinforcement in structural composites, including pressure vessels, glass cables, and woven thermal insulation. Discontinuous (chopped or blown) could be used as insulation or composite reinforcement. The chemistry of lunar soils is similar to that of terrestrial basalt.<sup>2 3</sup> Glass fibers have been produced using a simulated lunar basalt.<sup>4</sup> One aspect of glass fiber production which is critical to fiber strength is suppression of critical nuclei formation during the drawing process. It has been shown that the presence of even a small amount of crystalline material can be deleterious to basalt fiber strength.<sup>5</sup> One method of investigating crystallization is to use differential thermal analysis (DTA). Crystallization events will show up as exotherms on a DTA plot. As part of an ongoing study, DTA was used to test some of the thermal characteristics of a lunar simulant which is being used to produce continuous glass fiber.

#### EXPERIMENTAL AND RESULTS

The simulant used in this study is known as Minnesota lunar simulant-1 (MLS-1). This material was provided by the University of Minnesota Space Sciences Laboratory. The major element chemistry of MLS-1 is shown in table 1 along with a comparison with Apollo sample 10084.

DTA was used to characterize the melting point of MLS-1, as well as the crystallization behavior of glass made from MLS-1. The simulant was heated to 1,100 °C at a rate of 5 °C/min and then to 1,450 °C at a rate of 1 °C/min. Figure 1 shows the resulting curve from this DTA run. An endotherm is exhibited at 1,200 °C which indicates the MLS-1 melting point. The melting point was verified by heating a sample to 1,200 °C in a tube furnace and visually observing the phase transition.

For crystallization studies, MLS-1 was heated in a platinum crucible to 1,450 °C in a box furnace for 24 hours, then poured onto an aluminum plate to form a glass. The glass was then crushed using a mortar and pestle to facilitate loading into the DTA sample holder. The glass sample was then heated to 1,300 °C at a rate of 5 °C/min. Figure 2 is a DTA curve of the MLS-1 glass. The first crystallization exotherm appears at approximately 350 °C and is followed by other exotherms up to 1,000 °C. Figure 3 is a DTA heating curve of E-glass produced for comparison. Note the absence of exotherms in this curve.

The MLS-1 was next doped with silica such that the weight percent of silica was raised from 44 to 55 percent. This was done to aid the glass formation process of MLS-1. The DTA curve for this material is shown in figure 4. One can see that the melting point was lowered to 1,150 °C along with the appearance of two small exotherms at approximately 800 °C and 1,000 °C. These exotherms may be due to reactions of the silica with constituents in the MLS-1. This material was also made into a

glass and then heated in the DTA. Figure 5 shows the DTA curve for the silica doped MLS-1 glass. It can be seen that the crystallization exotherms between 200 °C and 1,000 °C are no longer present. However, there is an exotherm present at approximately 1,280 °C.

## DISCUSSION AND CONCLUSIONS

In order for a normally crystalline material to be spun into a glass fiber, it is necessary to rapidly cool or quench the fiber from at or above the melting point to near room temperature such that recrystallization does not occur. This quench must be extremely rapid, e.g., on the order of hundreds of °C/s, to avoid nucleation events. A true glass, however, may be cooled at slower rates and avoid crystallization. This means that a glass fiber can be formed over a wide range of temperatures, known as the working range of the glass. The materials tested in this study, although capable of forming glass, are not good glass formers. This is evidenced by the DTA results which showed that nucleation occurred readily in the MSL-1 glass. Doping the MLS-1 with silica did suppress the low temperature crystallization events, however, a higher temperature exotherm occurred, which is most likely due to sintering of silica.

Thus, it will be necessary to provide a rapid quench during fiber formation to obtain suitable fiber characteristics (e.g., tensile strength and flexibility). This is particularly important for fiber diameters of less than 20 micrometers, since recrystallized material would serve as comparatively large, high-stress sites for failure.

Further work is planned in order to determine the origin of the exotherms noted in figure 2. This would entail producing standards by mixing known amounts of silica with titania, iron oxide, and alumina and performing DTA analyses on these compounds. Reactions between these materials should provide insight into what is occurring in the MLS-1 glass.

It can be concluded from this work that MLS-1, though capable of forming a glass fiber, is not a good glass former. Doping with silica will be necessary to reduce the crystallization events and increase the working range of the glass.



Table 1. Major element chemistry of MLS-1 and Apollo 10084.

Element (%)	Average	Maximum	Minimum	10084
SiO <sub>2</sub>	43.86	45.90	41.70	42.55
TiO <sub>2</sub>	6.32	7.43	4.82	7.71
Al <sub>2</sub> O <sub>3</sub>	13.68	15.60	11.76	13.47
FeO	13.40	14.40	12.00	15.16
Fe <sub>2</sub> O <sub>3</sub>	2.60	4.10	0.90	
MgO	6.68	8.44	5.57	7.98
MnO	0.198	0.218	0.182	0.208
CaO	10.13	11.48	9.04	11.99
Na <sub>2</sub> O	2.12	2.27	1.97	0.445
K <sub>2</sub> O	0.281	0.348	0.167	0.147
P <sub>2</sub> O <sub>5</sub>	0.20	0.45	0.02	0.140
CO <sub>2</sub>	0.0015	0.0018	0.0014	
p/m	Average	Maximum	Minimum	
Be	1.00	1.22	0.87	
Zn	122	141	97	
Cu	445	706	214	
Sc	50	61	41	
Co	64	84	53	
Pb	18	20	10	
Ni	97	163	53	
Th	14	15	13	
Cr	173	366	89	
Rb	4	10	1	
V	761	952	506	
Zr	47	113	19	
Y	27	28	13	
Ba	95	117	62	
Sr	212	253	173	

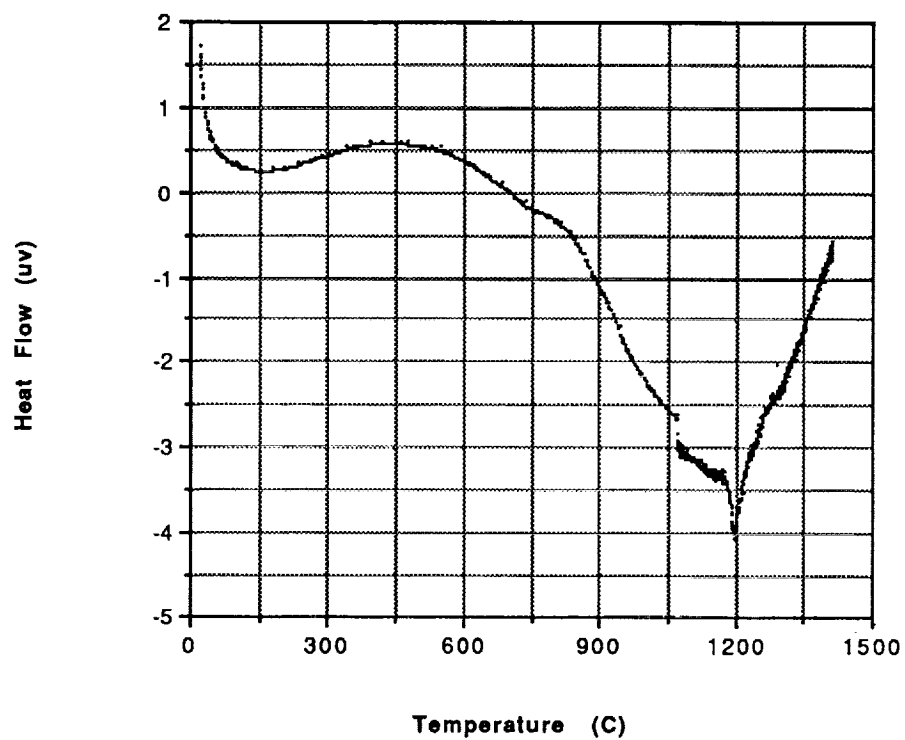


Figure 1. MLS-1 DTA curve.

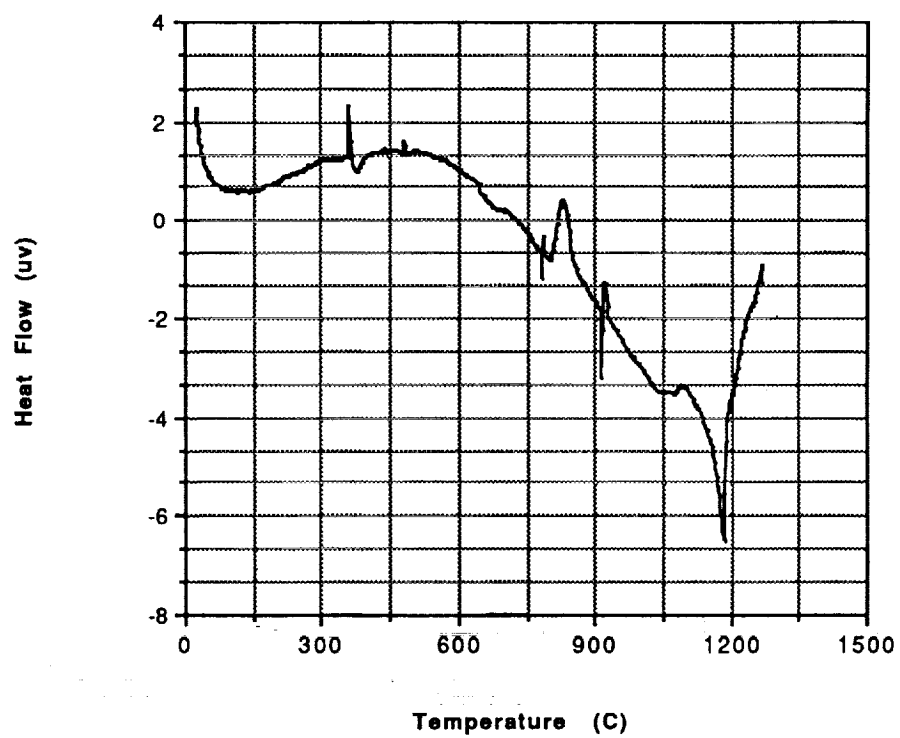


Figure 2. MLS-1 glass.

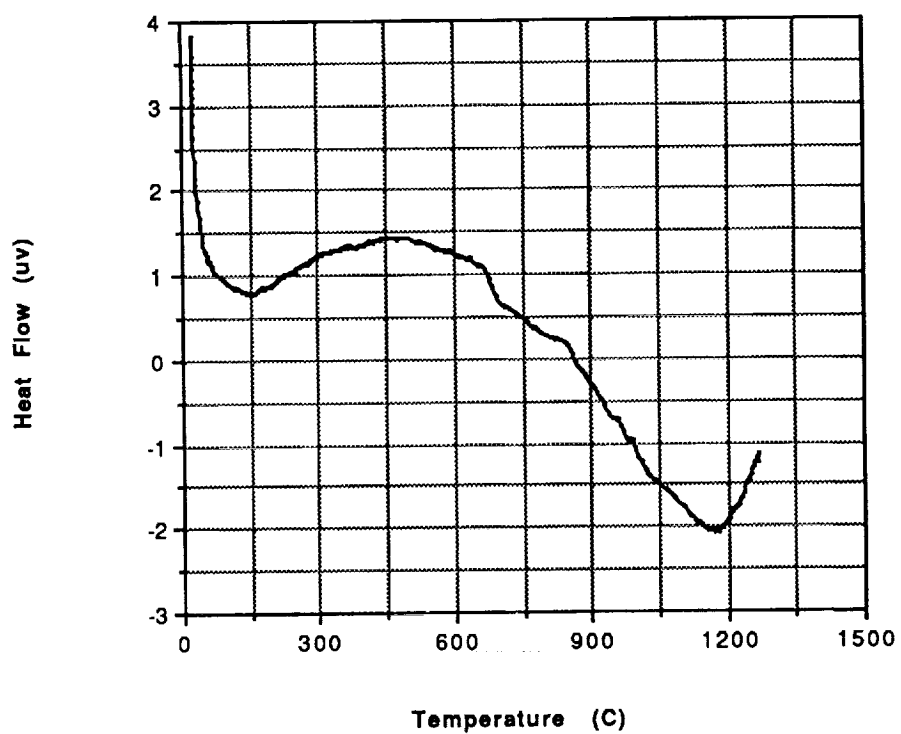


Figure 3. E-glass DTA curve.

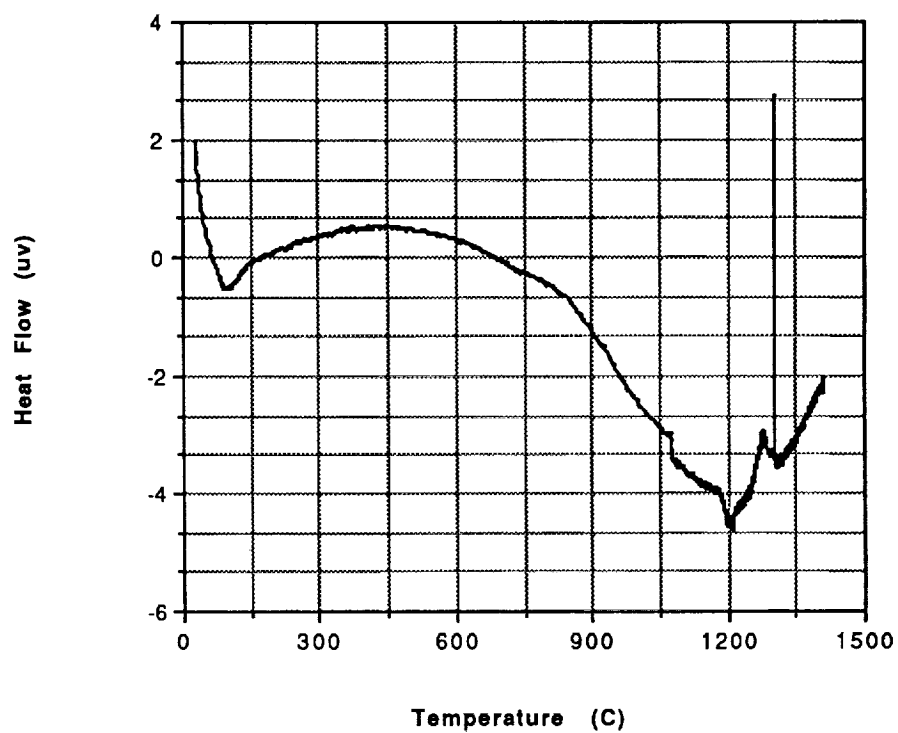


Figure 4. MLS-1 55-percent SiO<sub>2</sub>.

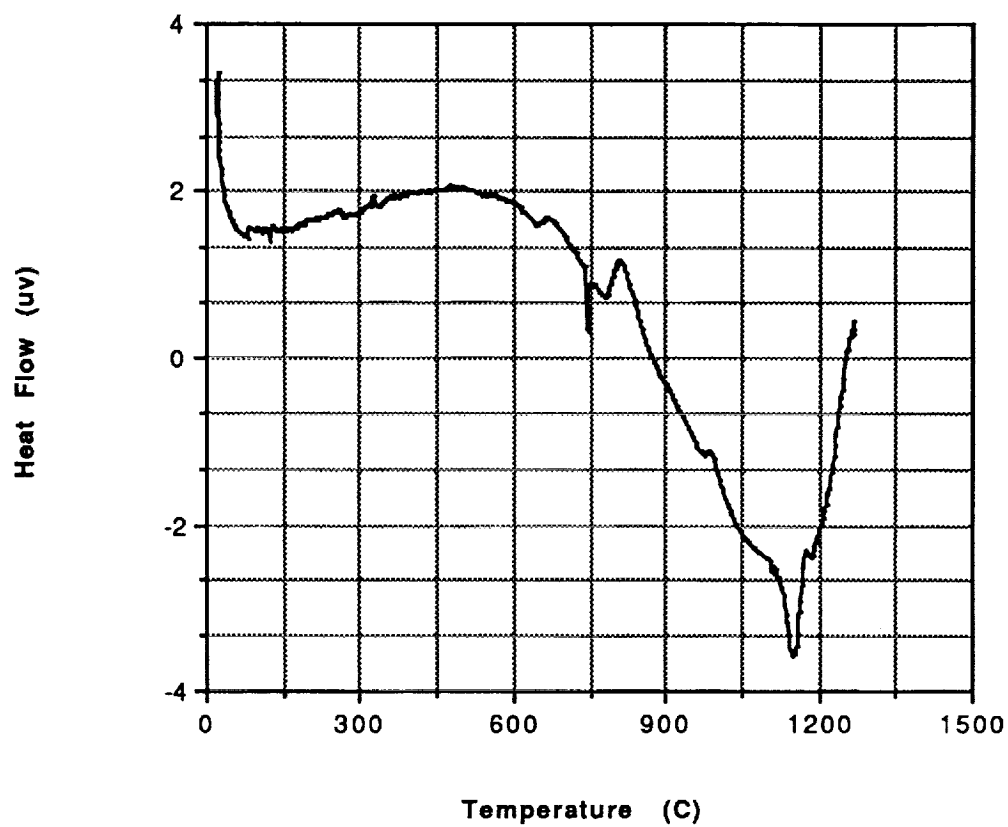


Figure 5. MLS-1 55-percent  $\text{SiO}_2$  glass.

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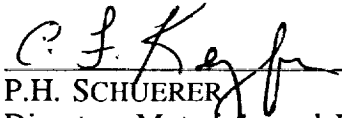
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## APPROVAL

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The information in this report has been reviewed for technical content. Review of any information concerning Department of Defense or nuclear energy activities or programs has been made by the MSFC Security Classification Officer. This report, in its entirety, has been determined to be unclassified.

  
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