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STATISTICAL DESIGN STUDY OF LUNAR CERAMIC

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TABLE OF CONTENTS

LIST OF ILLUSTRATIONS

LIST OF TABLES

Introduction

Long-term habitation on the moon will require construction of a lunar base. To facilitate such construction and to minimize its cost, materials available on the lunar surface can be used. The only such material is the lunar regolith or mantle rock, which occurs as powder and monolith. Fabrication of its powdered form into a ceramic is one option for processing lunar soil into a structural material.

A lunar ceramic could be used for a variety of applications. Magoffin and Garvey[1] have used concentrated solar rays to melt lunar simulant to form a glass. Reducing the intensity of light could yield in-situ ceramic foundations for roads or launch and landing pads.[1] For more demanding applications, the regolith would be processed into a ceramic to provide materials for insulation, radiation shielding blocks, and structural components. With the development of continuous and noncontinuous lunar glass fibers, reinforcement of the ceramic by the fibers is yet another option.[2]

To fabricate the ceramic, particle sizing would be necessary to obtain consistent properties. Further alteration to the lunar regolith would be minimal. Since the lunar soil is composed primarily of silica, densification would be accomplished by liquid phase sintering.[3] Therefore, energy would need to be applied in some form to promote sintering. This may be accompanied by pressure and the addition of binders. The present study consisted of milling a simulant lunar powder; processing the lunar simulant into a sintered ceramic; and measuring the strength that resulted from various combinations of processing parameters.

Experimental Procedure

The lunar simulant chosen for this study was Minnesota Lunar Simulant 1 (MLS-1)*. MLS-1 is a low-titanium basalt similar in chemistry to that of Apollo sample 10084.[4] The results of

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* University of Minnesota Space Sciences Laboratory

elemental analysis of MLS-1 and Apollo sample 10084 are given in Table I. The grain size of the MLS-1 is similar to coarser lunar mare basalts (<1mm) but is more equigranular, perhaps due to recrystallization[4]. Lunar soils generally contain varying amounts of glass and agglutinates due to micrometeorite impacts.[5] MLS-1 contains 10 to 30 weight percent glass products produced by processing in an in-flight sustained shockwave plasma reactor (ISSP).[5] This compares to a range of 10 to 80 weight percent found in actual lunar soil samples.

The plasma processed powder was ball milled for approximately 35 hours. This milling time yielded the smallest particle size. Large, mostly metal particles were removed from the powder by using an 80 mesh sieve (180μm). All of the culled particles could be removed from the sieve magnetically indicating that they were metallic.

Binder amount, cold pressing pressure, and firing time were the fabrication variables. The ceramic samples that resulted were evaluated based on their compressive strength. BBN/Catalyst* used these parameters to produce a statistically designed experiment. Once a process study like this has been defined, BBN/Catalyst generates a design which specifies the value of the processing variable for each run. After the experiment is completed, the resulting graphs can reflect linear and quadratic effects and interactions. The present designed experiment consisted of three factors (processing variables) at three levels and one response variable. Five samples were processed for each run. The average value of the strength obtained from the five samples was used as the response for that particular run.

Cylindrical greenware samples were fabricated using beeswax as the binder. Amounts of two, three or four percent wax were added to the lunar simulant. The sample size was chosen in accordance with ASTM C773-88.[6] Mineral oil was used as the releasing agent for the greenware (graphite was used for runs three and five). Before cold pressing, the samples were heated to

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* BBN Software Products, Cambridge, MA.

approximately 90°C to soften the wax. The MLS-1 powder was then subjected to a pressure of 276, 345, or 414 MPa in a cold pressing fixture.

After pressing several samples, a maximum of seven samples would be fired at one time while the samples were standing upright on their ends. This minimized warpage of the ceramic during densification. A layer of alumina powder was placed in each alumina crucible to reduce the friction and thermal gradient between the sample and crucible.

The samples were initially heated to 110°C and held for two hours to remove moisture from the greenware. The temperature was then raised to 600°C and held for four hours to remove the wax. Continuing, the samples were raised to a temperature of 1100°C standing upright on their ends in alumina crucibles. A temperature of 1100°C was chosen because it was approximately the largest achievable temperature the lunar ceramic could sustain without melting. All heating and cooling rates were 3°C/minute. The samples were fired for 12, 18, or 24 hours in air. During firing, the color of the greenware changed from light gray to reddish brown.

The ceramic's ends were cut on a lathe to obtain the proper length to diameter ratio and to ensure that the ends were parallel in accordance with ASTM C773-88. The compression test was performed on an Instron 8500.

Results and Discussion

A particle size distribution of the milled simulant appears in Figure 1. The average particle size was found to be approximately 16 microns.

A photomicrograph was taken using a Hitachi S-4000 Field Emission Scanning Electron Microscope (SEM) using secondary electrons. The average distribution of porosity and grain structure in the ceramic is shown in Figure 2.

The compressive strength data collected is presented in Table II. The minimum and maximum standard deviations of the sixteen runs were 4.7 and 24.2 MPa respectively. The average standard deviation for all sixteen runs was 20.4 MPa. Considering the lunar ceramic was fabricated by the cold pressing method, this is a reasonable value for the average standard deviation. BBN/Catalyst's Model Graph shown in Figure 3, represents the data shown in Table II. The error bars on the plots account for the variability in the process. The root mean square (RMS) error value is one-half the length of the error bar.

Much information can be gained by analyzing the graphs. The plots of pressure and wax percentage versus strength shows a strong interaction in Figure 3. This interaction is due to significantly different slopes (one positive and one negative) for the two lines. The interaction between time and strength is less than the wax and pressure plots because the slope of one line is positive and the other is zero. Parallel lines would indicate a weak interaction. The plot of wax percentage and strength also have a slight quadratic effect. The magnitude of the quadratic effect is determined by the intensity of the curve.

To examine how the range of each variable influenced the strength, BBN/Catalyst's Interpret Graph is used as shown in Figure 4. According to the strength and pressure plot, a pressure lower than 276 MPa could yield a greater strength. However, since the slope of the line is small, a significant increase in strength would not be expected. Looking at the strength and wax percentage plot in Figure 4, a further decrease below two percent wax could yield a greater strength. Several attempts were made to fabricate greenware with one percent wax. However, cracking of the greenware samples occurred due to friction between the particle and die-wall during extraction of the greenware from the fixture. Therefore, the only other possible increase in strength would be by increasing the firing time. Yet due to the energy and economics that would be required, increasing the sintering time may not be worthwhile, especially in a lunar environment. The greatest compressive strength obtained from the Interpret Graph was 247

MPa (with an RMS error value of ±14 MPa). This value matches exactly with the value obtained in run seven (see Table II).

In other experiments, the plots of the processing and response variables may result in a strong quadratic effect. If the plot had a minimum then both ends of the plot would have to be inspected. This is what the slider (vertical) bars are used for that appear at the maximum points of each plot in Figure 4. The slider bars would simply be moved from the minimum value of a variable to the maximum value of that same variable or vise versa. BBN/Catalyst would then adjust the other factors' effect on the response variables according to the new value of the chosen variable. Since no intense quadratic effects existed in Figure 4, these adjustments were not necessary.

Conclusion

The results indicate that a lunar ceramic cold-pressed at 276 MPa and fired at 1100°C for 24 hours yielded the greatest compressive strength of 247 MPa. This result is greater than those obtained by Meek *et al.*[7] via microwave sintering at similar temperatures.

The compressive strengths obtained are recognized to be considerably less than other terrestrial ceramics (i.e., 2620 MPa for alumina at 99.5 % theoretical density).[8] To increase the strength, a reduction or elimination of the binder, residual stresses, and other cold pressing limitations would have to occur. This increase in strength would be accomplished by hot isostatic pressing or some other advanced process. Of course, operation of these advanced processes will require more electrical energy. This increased use of electrical energy may be off-set by the reduction of manual labor required by cold pressing in a lunar environment.

Further investigations acquiring data on the properties of ceramics and lunar composites fabricated by advanced methods are recommended. Also, determination of the optimal sintering temperature of a lunar ceramic is needed. There is a good chance certain elements will be extracted before a lunar ceramic is processed. Data with these alterations to the lunar soil taken into account is another area recommended for evaluation of a lunar ceramic.

Acknowledgments

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8. Coors Ceramic Company Data Sheet, 1974.

Table I. Major Element Chemistry of Minnesota Lunar Simulant-1 and Apollo Sample #10084.

Element	MLS1 % Ave	10084
SiO_2	43.86	42.44
TiO_2	6.32	7.71
FeO	13.4	15.16
Fe_2O_3	2.6	--
MgO	6.68	7.98
Mn0	0.198	0.208
CaO	10.13	11.99
Na_2O	2.12	0.455
K_2O	0.281	0.147
P_2O_5	0.2	0.14
CO_2	0.0015	--

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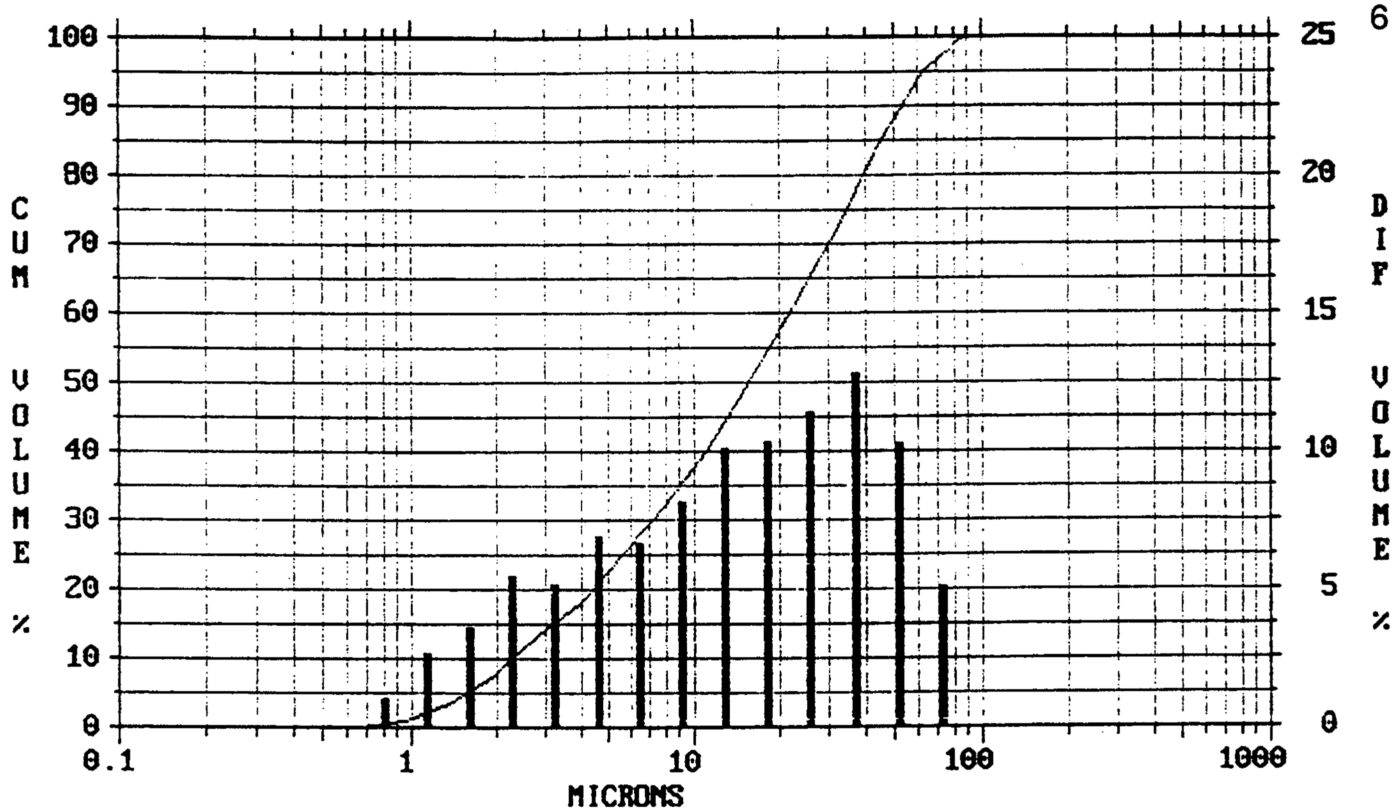

Figure 1. Particle size distribution of the MLS-1 after ball milling.

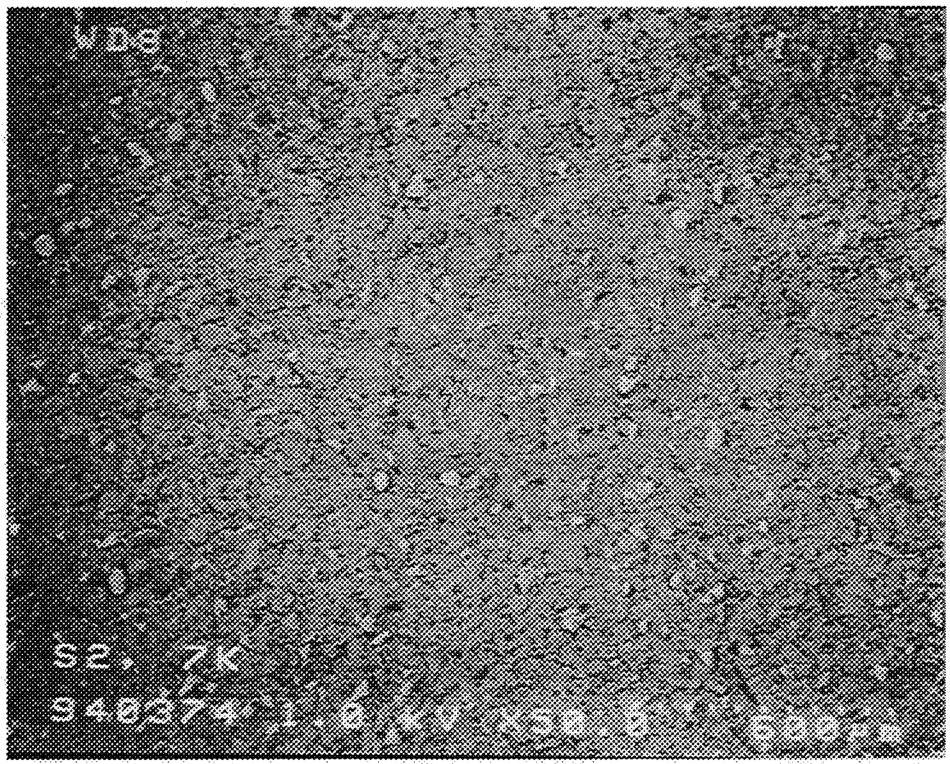

Figure 2. SEM photomicrograph of the lunar ceramic at 50X.

Table II. Summary of Compression Test Data.

Run No.	Pressure MPa	Binder Amt %	Firing Time hours	Strength MPa
1	276	2	12	212
2	414	4	12	211
3	414	2	24	227
4	276	4	24	226
5	414	2	12	190
6	276	4	12	227
7	276	2	24	247
8	345	3	12	212
9	345	2	18	241
10	276	3	18	222
11	414	4	24	239
12	414	4	18	218
13	345	4	24	208
14	414	3	24	241
15	276	4	18	202
16	345	4	12	209

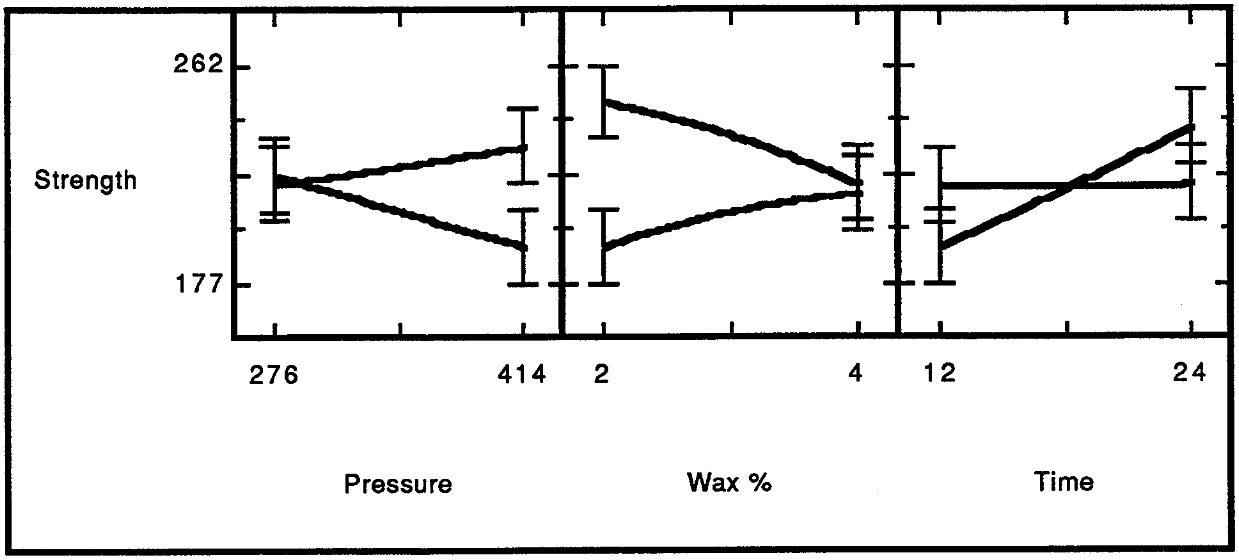

Figure 3. Data from Table II modeled according to BBN/Catalyst.

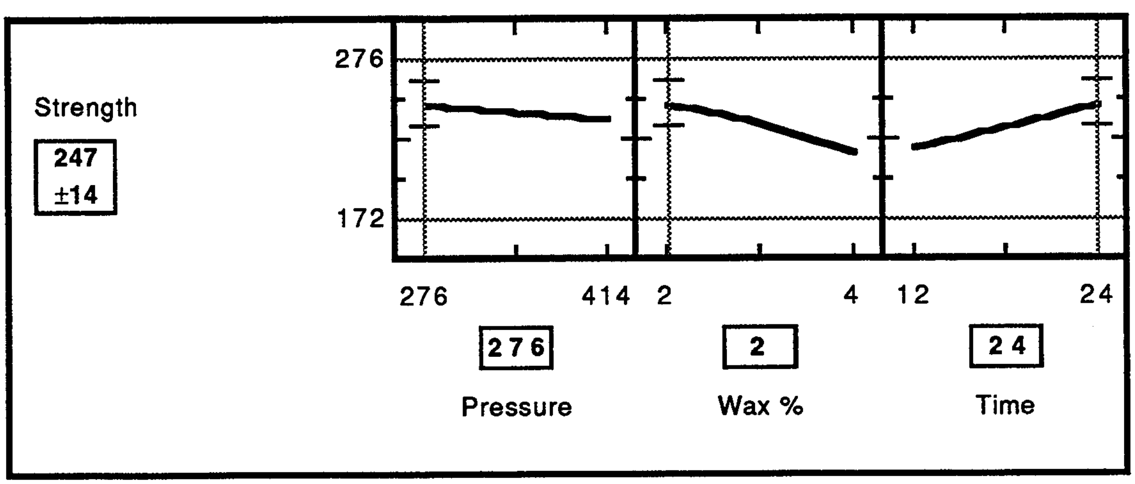

Figure 4. Processing variables that yielded the greatest predicted strength from the statistically designed experiment.

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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13. ABSTRACT *(Maximum 200 words)*
Fabrication of a lunar ceramic was conducted according to a statistically designed experiment. The method of cold pressing was used since the consumption of electrical energy is kept to a minimum, a priority in the lunar environment. This traditional fabrication technique also provides an initial data source on which further investigations can be based. Results obtained from using two percent binder, a cold pressing pressure of 276 MPa, and 24 hours sintering time yielded the greatest compressive strength of 247 MPa. Analysis of each variable's influence on the compressive strength is also presented.

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