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# Modeling and optimization of wear characteristics of gelcast fused silica ceramic composites using RSM

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## Abstract

Fused silica ceramic composite with combinations of Boron nitride and Silicon nitride were fabricated using gelcasting, a near net shape fabrication technique. Methylacrylamide (MAM) and N,N1-methylenebisacrylamide (MBAM) were used as monomers. A Mathematical model was developed to determine the effects of load, sliding distance and sliding speed on wear loss. The influence of these parameters are studied with the aid of Response surface methodology using central composite face centered design with six center points. The optimal values for minimum wear loss and parameters have been presented.

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*Keywords:* Fused silica; gelcastin; wear loss; RSM.

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## 1. Introduction

Fused silica is considered to be a better material which is widely used in the applications of aerospace, heat shielding, insulation for electronic equipment, antenna windows and semiconductor manufacturing [1-4]. It has characteristics such as high chemical resistance, outstanding optical qualities, good thermal shock resistance, very low thermal expansion, low dielectric constant and loss tangent [5-7]. Ceramic composites are widely using in many applications. The properties of these are enhanced by the combinations of different materials.

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Nomenclature	
A	load
B	sliding distance
C	sliding speed
RSM	response surface methodology

Advanced ceramics possess some superior properties like higher strength, lower environmental impact, and better reliability. The higher cost and shape difficulties made ceramics delaying further development. A new ceramic forming process called gelcasting was developed by Oak Ridge National Laboratory (ORNL). This process combines the traditional forming process and polymer chemistry. Concentrated slurry is prepared by mixing ceramic powders and monomers [8]. Monomer and cross linker are the main constituents to hold the ceramic particles together by forming the macromolecular network and helps in the formation of pores in ceramic bodies [9]. This slurry was poured in a mould to get the desired shape, size and during heating in-situ polymerization takes place to form a green body. The green body is dried under controlled humidity and binders are burn out on further sintering. The higher solid loading reduces the shrinkage of the green body in drying and sintering. The problems associated with cracking and warping will reduced by lower shrinkage. Higher solid loading will lead to increase in viscosity which makes the slurry to cast into the mould.

Wear is a complex system. Wear of a material depends on several functions such as contact geometry, surface roughness, microstructural features, grain sizes, fracture toughness, speed, load, temperature, duration, environment, and lubrication [10]. Few attempts were made to study the wear of fused silica ceramic composites.

Local algorithms are well suited for optimization problems with many design variables (more than 50), in cases where the analyses are computationally expensive, when numerical noise is not a severe problem, when gradients are readily available, and when local minima is not an issue. B) Global algorithms are well suited for optimization problems with fewer design variables (less than 50), where the analysis is computationally inexpensive, for discrete and combinatorial optimization problems, when numerical noise is severe, in cases where the gradient does not exists, when the objective and/or constraint functions are discontinuous, and when a global optimum is required. RSM is used to determine the factor levels that will simultaneously satisfy a set of desired specification and to determine the optimum combination of factors that yield a desired response and describes the response near the optimum. It also To determine how a specific response is affected by changes in the level of the factors over the specified levels of interest and to achieve a quantitative understanding of the system behavior over the region tested. This paper includes fabrication of ceramics and modeling of dry wear using Response Surface Methodology (RSM).

## 2. Experimental Details

### 2.1 Materials

A commercial fused silica powder of 99.9% purity (M/s. Ants Ceramics Pvt. Ltd., Thane, India) with an average particle size of 1-5  $\mu\text{m}$  having density of 2.2  $\text{g}/\text{cm}^3$  was used in this study. Commercially available dispersant Darvan 821A was used. Methylacrylamide (MAM) and N,N'-methylenebisacrylamide (MBAM) (both Alfa Aesar) as monomer and crosslinker, Polyethelene glycol 400 (PEG-400) and Ammonium persulfate (APS) (both Alfa Aesar) are used as surfactant and initiator. Diluted  $\text{HNO}_3$  and NaOH (both S.D. fine chemicals, India) were used for pH adjustment and distilled water as solvent.

### 2.2 Slurry preparation

The flow chart of gelcasting is shown in Fig. 1. Gelcasting of fused silica was carried out with the combination of Boron nitride (BN) and Silicon nitride ( $\text{Si}_3\text{N}_4$ ). Firstly premix solution was prepared by mixing Darvan 821A dispersant (1 wt % of monomer content), PEG (surfactant), monomers MAM and MBAM (10-20 wt% of fused silica) in distilled water by magnetic stirring. After that solid loading of 65 vol % fused silica was added to the premix solution and stirred for about 6 hrs. The slurry was deaired for 15-20 min and then the initiator Ammonium

persulfate APS (1 wt % of monomer content) is added for initiating polymerization. Finally, the slurry was cast into a glass mold and heated at 75-80 °C for 1 h for polymerization to take place. After the polymerization of the polymers, the green samples were removed from mould. The green bodies were then dried in a controlled humidity oven for 24 h. Finally, sintering was performed at a heating rate of 2 °C /min at 600 °C for binder burn out and heating rate 5 °C/min and 2 hour holding time at 1250 °C under nitrogen atmosphere.

### 2.3 Pin on disc tribometer

Wear tests are carried out on a pin-on-disc tribometer in dry conditions. IEICOS make computerized pin on disc wear testing machine with fused silica ceramic composite as pin material and high carbon EN31 steel (HRC60) is used as counter-surface. The rotating movement of the disc can be varied from 30 to 2000 rpm. The disc is cleaned before any test using dry air and the pin is cleaned in an ultrasonic tank using acetone and ethanol. The normal loading is carried out by dead weights. The experimental set up of pin on disc is shown in fig (2) and fig (3).

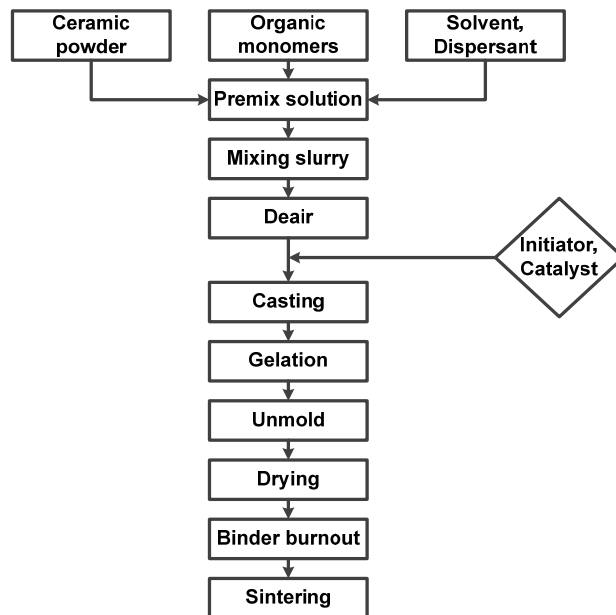


Fig. 1. Flow chart of gel casting process

### 2.4 Modeling with Response Surface Methodology (RSM)

Few modeling techniques were used to find the relation between input variables and output variables. RSM is the latest technique used for the statistical analysis. RSM is a combination of statistical and mathematical tool which has been used for growing, advancing and optimization technique [11]. It gives the quantitative estimates of possible interactions between factors which is a difficult task using other optimization techniques. The responses influenced by multi variables were easily solved using RSM. It can be used for model formulation of process optimization and also prediction of optimal combination of the parameters for a particular response. The number of trails needed to model a response was reduced using RSM [12].

Central composite face centered design with 6 center points is considered in RSM for experimentation. Load, sliding distance and sliding speed were chosen as input parameters and wear loss as response. The levels for all the input variables were fixed based on trial runs. The levels and their factors are given in Table 1. The measured responses after conducting the experiment are given in Table 2. ANOVA has been used to determine the significant parameters and their contribution and to model the response wear loss.

Wear tests were conducted on Fused silica ceramic composite with Silicon Nitride and Boron Nitride. Wear loss is measured by the weight measurement method.

Table 1. Factors and levels

S. No	Parameter	Notation	Unit	Level		
1	Load	A	N	5	10	15
2	Sliding distance	B	m	600	900	1200
3	Sliding speed	C	m/s	1	2	3

Table 2. Design layout and experimental results

Run order	Load (N)	Sliding distance (m)	Sliding speed (m/s)	Wear loss (g)
1	5	600	3	0.008
2	5	600	1	0.032
3	15	900	2	0.0189
4	15	600	1	0.0252
5	10	900	3	0.0177
6	15	600	3	0.013
7	5	900	2	0.014
8	5	1200	1	0.04
9	15	1200	3	0.03
10	15	1200	1	0.042
11	10	900	2	0.021
12	10	900	2	0.0215
13	10	900	2	0.022
14	5	1200	3	0.014
15	10	600	2	0.0144
16	10	900	2	0.023
17	10	900	2	0.0235
18	10	1200	2	0.027
19	10	900	1	0.0405
20	10	900	2	0.024

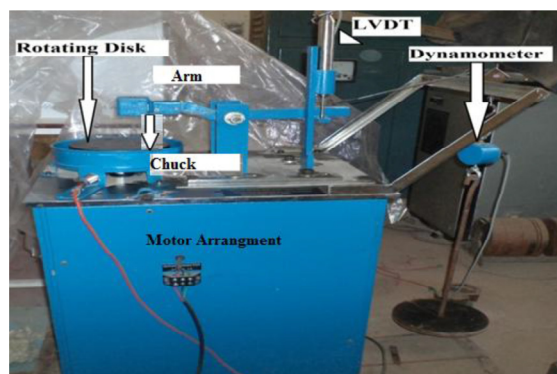


Fig. 2. Pin-on-Disc Tribometer



Fig. 3. Pin-on-disc Tribometer digital display unit

### 3. Results and Discussions

ANOVA has been applied to find the significant parameters that are given in the Table 3.

Table 3. ANOVA results for Wear loss

Source	Sum of Squares	DOF	Mean Square	F Value	p-value Prob > F	
Model	0.00169	9	0.000187927	80.78426405	< 0.0001	Significant
A-Load	4.5E-05	1	0.000044521	19.1382659	0.0014	
B-sliding distance	0.00036	1	0.000364816	156.8236476	< 0.0001	
C-sliding speed	0.00094	1	0.0009409	404.4651824	< 0.0001	
AB	4.9E-05	1	0.000049005	21.06580536	0.0010	
AC	8.3E-05	1	0.000083205	35.76737752	0.0001	
BC	4E-07	1	4.05E-07	0.174097565	0.6853	
A <sup>2</sup>	5.6E-05	1	5.55751E-05	23.89007917	0.0006	
B <sup>2</sup>	1.7E-07	1	1.65682E-07	0.071221731	0.7950	
C <sup>2</sup>	0.00018	1	0.000182866	78.60856771	< 0.0001	
Residual	2.3E-05	10	2.32628E-06			
Lack of Fit	1.6E-05	5	3.25256E-06	2.32325974	0.1882	not significant
Pure Error	7E-06	5	0.0000014			
Cor Total	0.00171	19				

From Table 3, it has been observed that load, sliding distance, sliding speed, load and sliding distance, and load and sliding speed, sliding distance and sliding speed, load<sup>2</sup>, sliding speed<sup>2</sup> are significant model terms. The interactions are shown in Fig. 4 (a), Fig. 4 (b), Fig. 4 (c), Fig. 5 (a), Fig. 5 (b) and Fig. 5 (c).

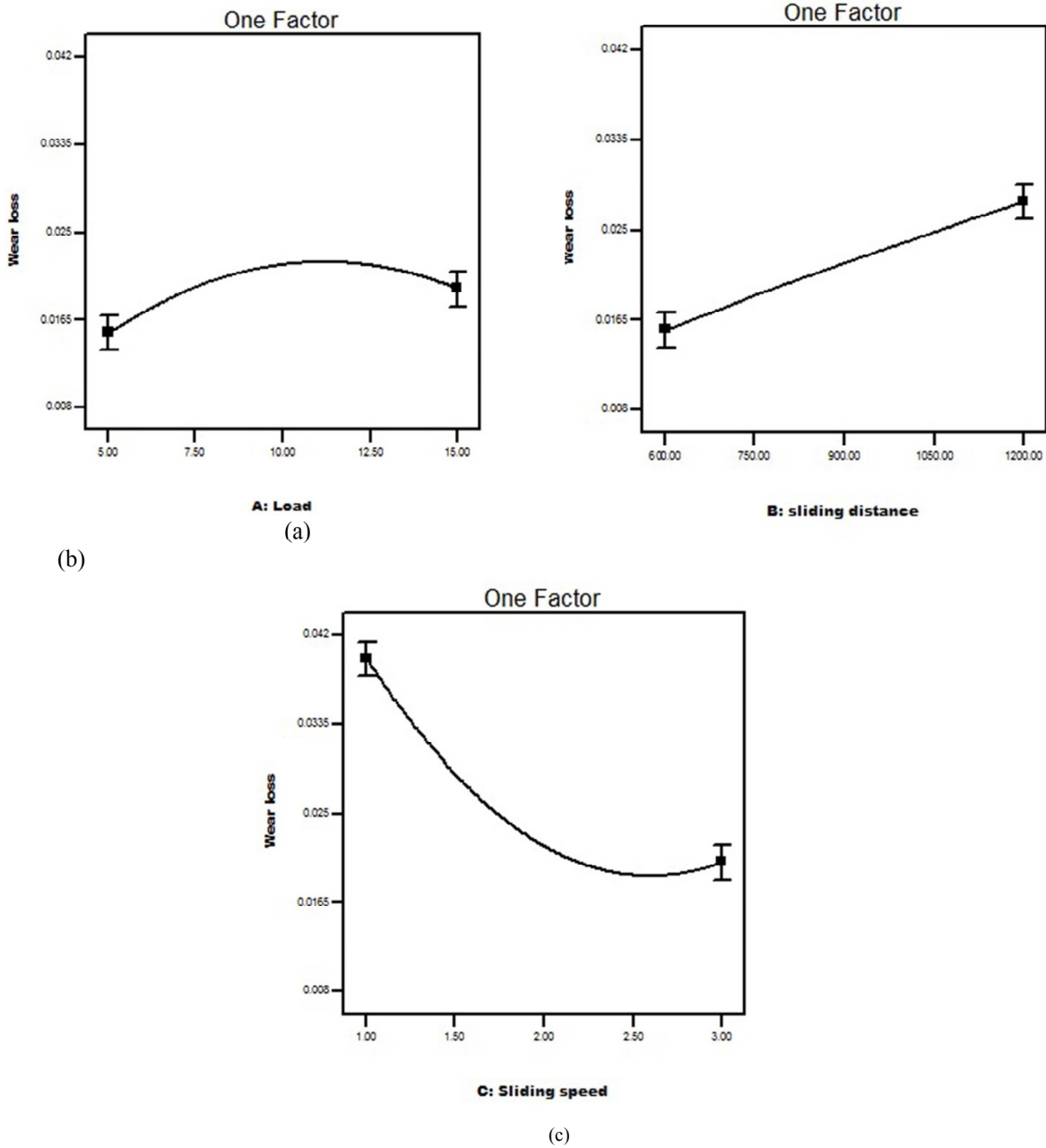


Fig. 4. (a) Effect of load on wear loss; (b) Effect of sliding distance on wear loss; (c) Effect of sliding speed on wear loss

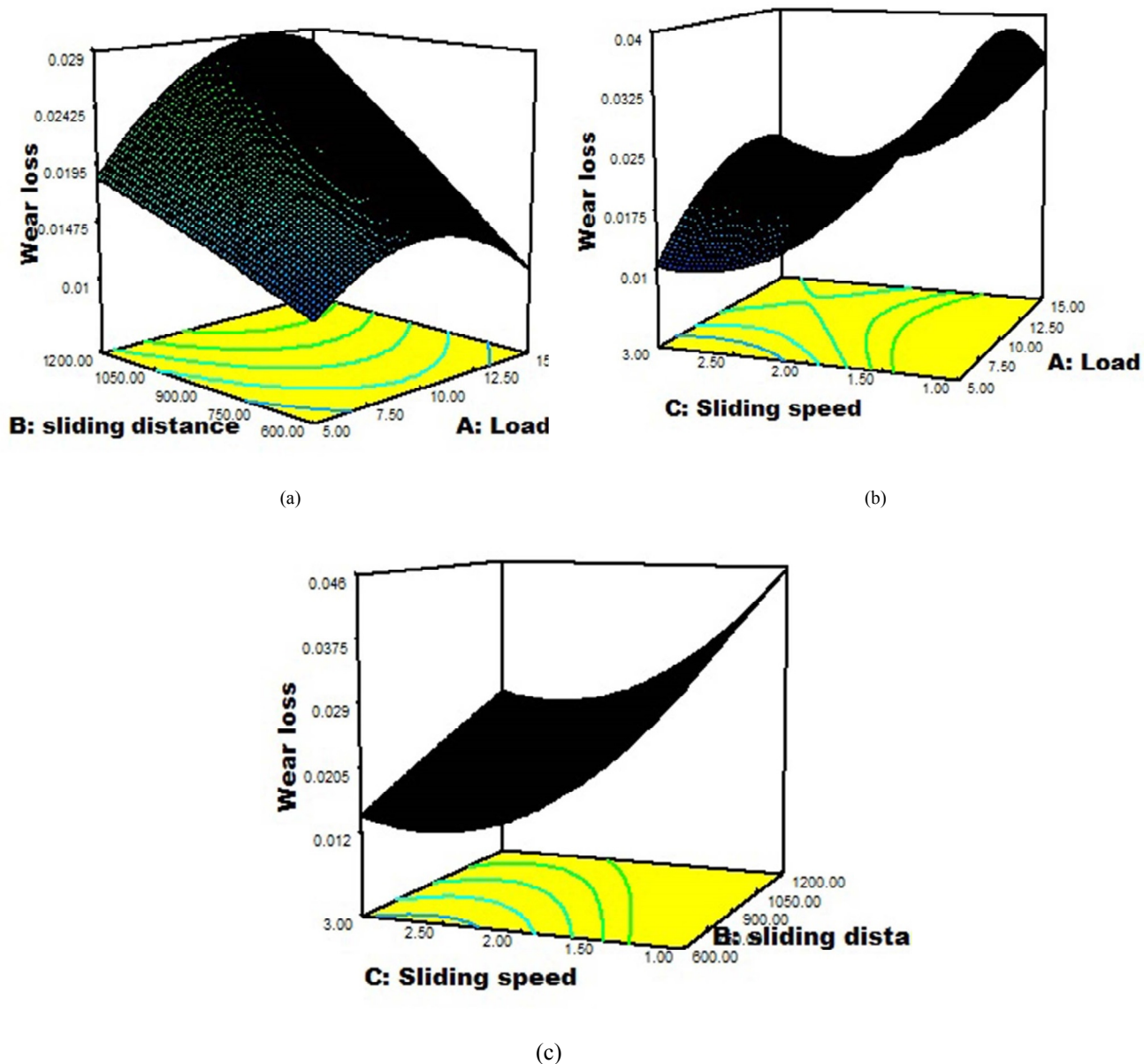


Fig. 5. (a) Effect load and sliding distance on wear loss; (b) Effect of load and sliding speed on wear loss; (c) Effect of sliding speed and sliding distance on wear loss

The wear loss increases as the load and sliding distance increases. As the sliding speed increases, wear loss decreases due to contact of the pin material with the disc and also increase in the temperature of the surface of pin material at the contact with the disc.

A mathematical regression model generated for wear loss is given in Eq.1.

$$\text{Wear loss} = 0.022 + 2.11 \times 10^{-3} * A + 6.04 \times 10^{-3} * B - 9.7 \times 10^{-3} * C + 2.475 \times 10^{-3} * A * B + 3.225 \times 10^{-3} * A * C - 2.25 \times 10^{-4} * B * C - 4.495 \times 10^{-3} * A^2 - 2.455 \times 10^{-4} * B^2 + 8.155 \times 10^{-3} * C^2 \tag{1}$$

From ANOVA, the R-square, adjusted R-square and predicted R-square values were found to be 98.64%, 97.42% and 93.23% respectively.

The optimum value for minimum wear loss is found to be 0.00699249 grams from RSM. The optimal values corresponding to optimal parameters are presented in the Table 4.

Table 4. Optimal results obtained from RSM

Response	Optimal Value	Optimal parameter	RSM
Wear loss (grams)	0.00699249	Load	5.07
		Sliding distance	619.10
		Sliding speed	2.87

## Conclusions

Fused silica ceramic composites with combination of Boron nitride and Silicon nitride were fabricated using gelcasting technique. A mathematical model using response surface methodology was obtained for wear loss that contains load, sliding distance and sliding speed as input parameters. The influences of parameters have been studied by ANOVA for wear loss. It was found that the wear loss of fused silica ceramic composites is influenced by load, sliding distance and sliding speed. The optimal value for minimum wear loss obtained from RSM was 0.00699249 grams.

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