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OPTIMIZATION OF LOAD AND HBN CONTENT FOR IMPROVING TRIBOLOGICAL PERFORMANCE OF A Si_3N_4 -HBN CERAMIC COMPOSITE USING TAGUCHI-GREY RELATIONAL ANALYSIS

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Abstract: Silicon nitride (Si_3N_4) is common in various industrial applications and proven its applicability with its high fracture toughness, strength and wear properties. The material has a proven capability in the biomedical field in the context of orthopedic applications. This paper reports the experimental and Taguchi based grey relational analysis for the tribological behavior of Si_3N_4 -hBN ceramic composite sliding against steel (ASTM 316L) in the dry condition. The wear tests were conducted with 0, 4, 8, 12, and 16 % volume of hBN in Si_3N_4 at loading conditions in the range $5\text{N} < \text{Load} < 25\text{N}$, with 5N increments using Pin-on-Disc (PoD) tribometer. A weighted grey relational grade is calculated for minimization of volumetric wear rate and coefficient of friction with aim of improving tribological performance of Si_3N_4 -hBN ceramic composite applicable for various industrial and biomedical applications. Load and % volume of hBN addition are two factors considered for optimization. Analysis of variance (ANOVA) presented % vol of hBN is significant factor followed by load. In the Si_3N_4 -hBN / steel contact pair, the main phenomenon of wear observed was an adhesive type of wear at both low load and at high load.

Keywords: Silicon Nitride (Si_3N_4), hexagonal Boron Nitride (hBN), Steel, Taguchi-Grey Relational Analysis, Wear, Coefficient of Friction (CoF).

1. INTRODUCTION

In 1859, Deville and Wohler developed synthetic Si_3N_4 and in 1950's commercial interest in Si_3N_4 increased with a better understanding of its properties. Properties of Si_3N_4 have made it a highly suitable material for the biomedical field along with various industrial applications and extreme operating conditions, in particular those demanding high strength, low density, and low wear rate [1]. Treatment for the hip, knee, shoulder, and other articulated parts in the body has been

revolutionized with the development of biomaterials. Important characteristics of biomaterials are that they are inert within the body environment and sufficiently mechanically stable to sustain loads consistent with those of the human body. The most common materials used for hip/knee joint replacement are metal (M), Plastic (P), and ceramic (C). The plastic is generally used for cup applications only while other metal and ceramic materials are used for both cup and socket applications. The most common material combinations available in the market

are Metal-on-Metal (MoM), Metal-on-Plastic (MoP), Ceramic-on-Plastic (CoP), Ceramic-on-Ceramic (CoC), and Ceramic-on-Metal (CoM). In the MoM category, titanium alloys and stainless steel are frequently used in total hip replacement (THR). Metal-on-UHMWPE has become preferable to the MoM system, because of the adverse reaction of metal ions release during wear in MoM pair [2, 3]. Si_3N_4 is biocompatible and along with its superior mechanical properties presented itself as an alternative material in the field of orthopedic surgery [4]. Si_3N_4 is suitable in the field of biomedical for developing hip and knee joints and spine disc surgery bearings [5, 6].

Tribological studies of articulating surfaces have focused on friction and wear performance during articulation. The study of articulating surfaces present in the human body or in animals, such as an artificial joint, is referred to as bio-tribology. Bio-tribology is very important in order to understand the performance or lifespan of an artificial joint. Wear in an artificial joint remains a critical issue limiting the lifespan of artificial joints. Although Si_3N_4 is suitable for joint replacement applications the wear performance of Si_3N_4 is still an issue of debate. One of the common opinions on the wear performance of Si_3N_4 is that the absence of material oxidation helps it to minimize friction in the presence of water lubricants [7]. The Amedica Corporation, USA have developed various prototype total hip bearings using sintered Si_3N_4 , and in testing have confirmed the improved strength and fracture toughness over medical grade oxide ceramic alumina (Al_2O_3) [8] and a number of research works have already proved the enhanced wear performance of Si_3N_4 in the presence of water lubrication. The wear performance of Si_3N_4 will be more favorable in the human synovial fluid, as it is an excellent lubricant irrespective of bearing material [9, 10]. One of the unique properties of Si_3N_4 is its ability to be formulated into a porous substrate and hard glassy bearing surface, capable of providing direct bone in-growth. The tribological study of ceramic has shown that the wear mechanism in ceramic depends

upon the contact conditions. Wear in the Si_3N_4 occurs through surface fracture with a load exceeding the threshold value and further wear occurs due to surface oxidation [11].

The wear performance of Si_3N_4 against steel in the dry lubricant condition is still unclear. Akdogan and Stolarski [12] evaluated the wear performance of Si_3N_4 sliding against steel; they found that main types of wear are abrasive and adhesive wear of the surface. Olofsson *et al.* [13] evaluated the wear performance of a Si_3N_4 and CoCr disc against a Si_3N_4 and Al_2O_3 ball in the presence of a Phosphate Buffered Saline (PBS) and a bovine serum using PoD tribometer. In the case of Si_3N_4 sliding against Si_3N_4 , it was shown the formation of a tribofilm on Si_3N_4 controlled friction and wear in both PBS and bovine serum comparable to other pairs.

Hexagonal boron nitride (hBN) is a bio-compatible, solid situ lubricating material [14-16]. Some researchers proposed the addition of hBN in ceramic materials to improve their wear performance. Incorporation of hBN in silicon nitride leads to the generation of hydrated layers of an oxides (H_3NO_3 and $\text{BN}(\text{H}_2\text{O})_x$) during sliding thereby minimizing the wear coefficient. Li *et al.* [17] investigated the effect of the hBN addition on the friction and wear performance of B4C-hBN ceramics in dry friction conditions. With 20% of hBN content in the Si_3N_4 -hBN ceramic composites, the CoF reached a minimum value of 0.179. Carrapichano *et al.* [18] conducted the sliding wear test on a pin-on-disc tribometer for the Si_3N_4 -BN composite in a self-mated pair, with 10, 18 and 25% vol. of BN in Si_3N_4 . They concluded that the addition of Boron up to 10% improved the tribological properties of Si_3N_4 and further addition affected the mechanical properties of Si_3N_4 . Wei *et al.* [19] analyzed the tribological behavior of Si_3N_4 -hBN (with 0%, 2%, 4%, 6%, 8%, and 10% of hBN) sliding against pure Si_3N_4 . The analysis of the experimental results presented a decrease in CoF with an increase in the hBN content.

Along with the addition of lubricants, various factors including speed and load have a significant effect on the tribological

performance of silicon nitride. In an earlier study, it has been demonstrated that wear loss of Si₃N₄-hBN ceramic composite sliding against steel is a function of the interaction between load and % volume of hBN addition [20]. Chen [21] evaluated the tribological performance of Si₃N₄-based ceramic materials for various loading conditions. At low loading the wear mechanism was abrasive, with an increase in load; the adhesive wear mechanism was prominent. Wei Chen *et al.* [22] evaluated the effect of sliding speed on the tribological performance of Si₃N₄-hBN sliding against steel. The analysis of results revealed that with an increase in speed the degree of abrasive wear decreased leading to a decrease in the coefficient of friction. In another work, Wei Chen [23] evaluated the effect of load on the tribological performance of a Si₃N₄-hBN ceramic sliding against ASS and 45 steel. It was reported that the SN10/ASS sliding pair presented the minimum value of wear rate and CoF. But, with the increasing load for the same pair, the CoF increased with a combined abrasive and adhesive mechanism.

In this study, we evaluated the tribological

performance of Si₃N₄-hBN ceramic composite sliding against ASTM 316L medical grade steel without lubricant. Volumetric wear rate and coefficient of friction (CoF) were evaluated for analysis of tribological performance. Taguchi based grey relational analysis applied with aim of optimization of load and hBN content for simultaneous minimization of wear rate and CoF

2. SPECIMEN PREPARATION AND TEST METHOD

Giving consideration to earlier research works the parameters for our tribological study were selected to be: % volume of hBN in silicon nitride at 0%, 4%, 8%, 12%, and 16% level (SN0 –SN16). Table 1 shows the factors and corresponding levels selected for the tribological study of a Si₃N₄-hBN ceramic composite in a dry environment.

The pin samples were prepared with a 99 % pure powder of Si₃N₄ and hBN of 1- μ m size mixed in five different proportions with the aid of ball mill. The mixed powder with an additive of polyvinyl alcohol was sintered by uniaxial

Table 1. Control parameters and its level values

Control para.	Levels				
	1	2	3	4	5
% Vol. of hBN	0 (SN0)	4 (SN4)	8 (SN8)	12 (SN12)	16 (SN16)
Load (N)	5	10	15	20	25

Table 2. Properties of sintered sample*

Properties	Samples				
	SN0	SN4	SN8	SN12	SN16
Density (gm/cc)	2.04	1.96	1.96	1.93	1.84
Vickers Hardness (MPa)	7484.51	2775.88	2318.17	1741.07	907.96

*Testing at Central Glass and Ceramic Research Institute, Kolkata (India).

Table 3. Typical properties of steel disc

Desig.	Density (gm/cc)	Mod. of elasti. in tension (GPa)	Mean coeff. of thermal expansion from 293 to 873 °K (10 ⁻⁶ /K)	Thermal conductivity at 373 °K (W/m K)	Avg. surface roughness Ra (μ m)
ASTM 316L	7.95	186.4	18.5	16	0.242

hot-pressing in an inert atmosphere at 30 MPa, 1600 °C and 60 min dwell time in the form of a pin of the dimension 10 mm diameter and 15 mm length.

Tables 2 and 3 show corresponding properties of Si₃N₄-hBN ceramic composite and steel disc.

The wear experiments were performed on a Ducom TRLE-PMH400 pin-on-disc tribometer having a maximum normal load capacity of 200 N. Tests were performed according to ASTM F732 standards [24]. During tests the composite was used as the pin specimen loaded through a vertical specimen holder against a flat steel disc as the counterface rotating at a speed of 200 rpm for a 20-min duration.

Wear rate and coefficient of friction recorded online during wear test. Wear measurement- wear volume loss or volumetric wear rate was calculated per meter of sliding distance using the following equation:

$$\begin{aligned} \text{Vol. Wear Rate} \left(\text{mm}^3/\text{m} \right) &= \\ &= \frac{\text{Wear loss} \cdot 10^{-3} \cdot \text{CS Area of pin}}{\text{Sliding distance}} \quad (1) \end{aligned}$$

Where, wear loss is in microns, CS area in mm², and sliding distance in m.

3. METHODOLOGY

3.1 Design of Experiment-Taguchi Method

Design of experiment –Taguchi method is a statistical technique, helps to study a number of parameters simultaneously and economical way. Taguchi method helps to plan experimental layout using Orthogonal Array (OA) to study the effect of control parameter through a minimal number of experiments [25]. Orthogonal Array used to plan experiment for two control parameters such as load and % vol. of hBN addition in silicon nitride. Each control parameter varied through five levels as shown in Table 1. Based on two control parameters and five levels L25 orthogonal array selected with 25 numbers of experiments. To evaluate the process parameters, Taguchi method uses signal-to-

noise (S/N) ratio as an objective function for the desired output. S/N ratio is a measure of robustness helps to identify control factors that reduce variability in a product or process by minimizing the effects of uncontrollable factors. S/N ratio is characterized into three categories: Nominal the best (NB), Lower the better (LB) and Higher the better (HB). Regardless of desired output, the maximum value of S/N ratio is corresponds optimized control parameters for desired output. Taguchi method can't optimize multi-objective optimization problems. To overcome this, Taguchi method is integrated with Grey relational analysis (GRA) to optimize multi-objective problems.

3.2 Grey Relational Analysis

In 1982, Deng proposed Grey system theory to uncertainties in system models and analyze the relation between systems. Grey relational analysis helps to convert multi-response problem into single response problem by calculating grey relational grade (GRG). The various steps followed in the grey relational analysis are as follow:

1. Normalization of data in the range of 0 or 1.

The collected raw experimental data is normalized into 0 or 1, using two criteria wise lower is better (LB) and higher is better (HB). A LB criterion is used to normalize data when the objective is to minimize. Equation 2 is used for LB criteria. A HB criterion is used to normalize data when the objective is to maximize. Equation 3 is used for HB criteria

$$x_i(k) = \frac{\max y_i(k) - y_i(k)}{\max y_i(k) - \min y_i(k)} \quad (2)$$

$$x_i(k) = \frac{y_i(k) - \min y_i(k)}{\max y_i(k) - \min y_i(k)} \quad (3)$$

Where, $x_i(k)$ is the value after the grey relational generation, $\min y_i(k)$ is the smallest value of $y_i(k)$ for the k^{th} response, and $\max y_i(k)$ is the largest value of $y_i(k)$ for the k^{th} response. $i = 1, 2, 3...$ the number of experiments and $k = 1, 2, 3...$ the number of responses.

2. Calculation of Grey Relational Coefficient (GRC)

GRC is calculated to determine the relation between ideal and actual normalized experimental data. GRC (ξ) is calculated using equation 4.

$$\xi = \frac{\Delta_{min} + \psi \Delta_{max}}{\Delta_{oi}(k) + \psi \Delta_{max}} \quad (4)$$

Where $\Delta_{oi}(k) = \|x_0(k) - x_i(k)\|$ = difference of the absolute value of $x_0(k)$ and $x_i(k)$; ψ is the distinguishing coefficient; $0 < \psi < 1$, Δ_{min} is the smallest value of $\Delta_{oi}(k)$ and Δ_{max} is the largest value of $\Delta_{oi}(k)$.

3. Calculation of Grey Relational Grade (GRG)
The analysis of multiple outputs characteristic is based on grey relational grade. The GRG (γ) is an average sum of GRC and calculated using equation 5. Its value lies between 0 and 1.

$$\gamma_o = \frac{1}{n} \sum_{k=1}^n \xi_i(k) \quad (5)$$

Where, n is a number of process responses.

4. RESULTS AND DISCUSSION

The output characteristic wise, the coefficient of friction recorded online and volumetric wear rate calculated using equation 1. The collected experimental data is further processed following steps 1 to 3 for calculation of GRG. To improve the tribological performance of Si3N4-hBN ceramic composite, both CoF and volumetric wear rate needs to be minimized. The experimental data is normalized with LB criterion using equation 2. After calculating GRC, the weighted GRG is calculated with 70% weightage to volumetric wear rate and 30% weightage to CoF.

$$\text{Weighted GRG}(\gamma) = \frac{1}{2} (0.7 \cdot \xi_i(\text{VWR}) + 0.3 \cdot \xi_i(\text{CoF})) \quad (6)$$

Table 4, shows experimental data processed into a weighted grey relational grade.

Figure 1 shows the column map for a grey relational grade of Si3N4-hBN ceramic composite sliding against steel under dry

conditions. From Table 4 and Figure 1 it is clear that the maximum value of GRG- 0.467 observed at 5N load and 12% volume of hBN in silicon nitride. The experiment number 3 corresponding to 5N load and 12% volume of hBN presents optimal condition for minimum volumetric wear rate and coefficient of friction simultaneously.

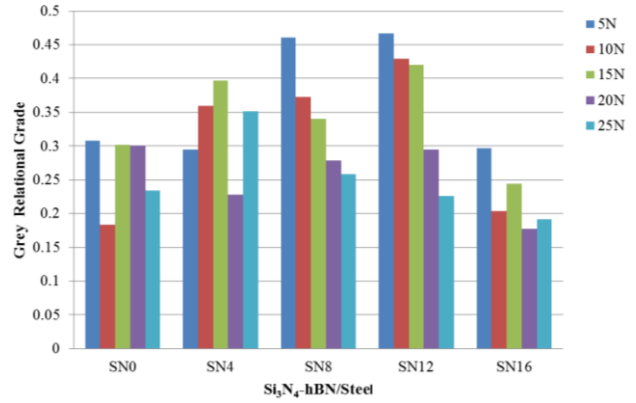


Figure 1. Column Map for GRG of Si3N4-hBN/Steel Pairs under Different Loads

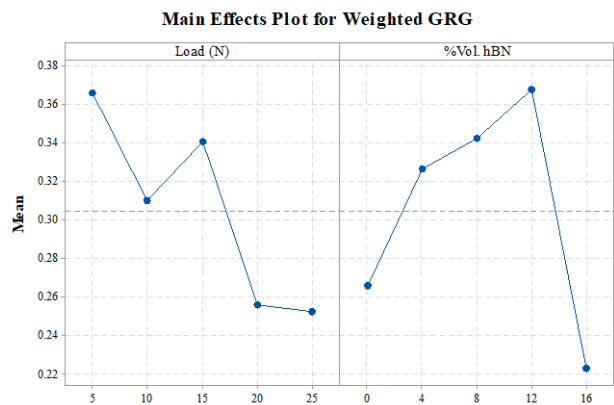


Figure 2. Main Effect Plot for Weighted GRG

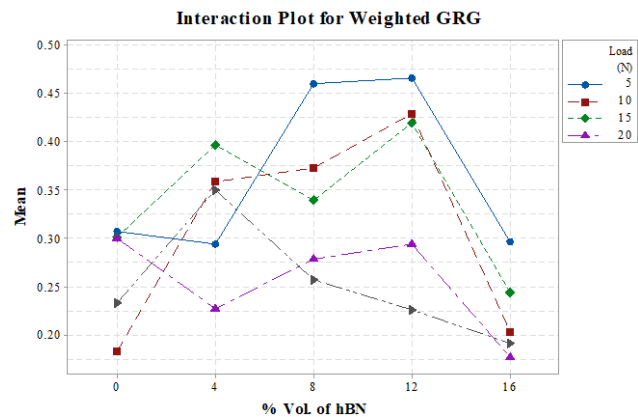


Figure 3. Interaction Plot for Weighted GRG

The main effect plot and interaction plot of load and % vol. of hBN on weighted grey relational grade is presented in Figure 2 and 3.

Table 4. Data Processing of Each Performance Characteristic (Grey Relational Analysis)

Expt. No.	Control Parameter		Performance Characteristic		GRC (ξ)		GRG (γ)	Rank
	Load (N)	% Vol. hBN	Vol. Wear Rate (mm^3/m)	CoF	Vol. Wear Rate (mm^3/m)	CoF		
1	5	4	0.6671	0.01	0.415	1	0.295	15
2	5	8	0.0653	0.014	0.898	0.978	0.461	2
3	5	12	0.0381	0.03	0.948	0.899	0.467	1
4	5	16	0.4209	0.074	0.533	0.737	0.297	13
5	5	0	0.4983	0.028	0.49	0.909	0.308	10
6	10	4	0.2215	0.06	0.69	0.782	0.359	7
7	10	8	0.1481	0.096	0.775	0.677	0.373	6
8	10	12	0.0656	0.065	0.898	0.766	0.429	3
9	10	16	0.8591	0.17	0.355	0.53	0.204	22
10	10	0	1.5294	0.097	0.235	0.674	0.183	24
11	15	4	0.0621	0.164	0.903	0.539	0.397	5
12	15	8	0.1869	0.146	0.727	0.569	0.34	9
13	15	12	0.0126	0.217	1	0.465	0.42	4
14	15	16	0.4835	0.216	0.498	0.466	0.244	19
15	15	0	0.2615	0.194	0.652	0.495	0.302	11
16	20	4	0.5499	0.243	0.464	0.436	0.228	21
17	20	8	0.3556	0.179	0.576	0.516	0.279	16
18	20	12	0.2116	0.369	0.701	0.334	0.295	14
19	20	16	0.9441	0.276	0.333	0.404	0.177	25
20	20	0	0.2061	0.335	0.706	0.356	0.301	12
21	25	4	0.1153	0.252	0.819	0.427	0.351	8
22	25	8	0.5195	0.129	0.479	0.602	0.258	17
23	25	12	0.4759	0.37	0.502	0.333	0.226	18
24	25	16	0.7868	0.278	0.376	0.402	0.192	23
25	25	0	0.4672	0.3085	0.506	0.376	0.234	20

From main effect plot, it is clear that 5N load and 12% volume of hBN has the maximum value of weighted GRG, presenting optimum parameter level for load and hBN addition. Interaction plot presents strong interaction of load and hBN addition on weighted GRG or on tribological performance of silicon nitride.

4.1 Analysis of Variance (ANOVA)

The analysis of variance (ANOVA) is implemented to evaluate the significant factor affecting the tribological performance of silicon nitride. The ANOVA is performed for GRG considering load and % volume of hBN as two factors. The ANOVA is carried out at 99% of confidence level [25]. Table 5 shows ANOVA for the weighted grey relational grade.

Table 5. ANOVA for Weighted Grey Relational Grade

Source	DF	Adj. SS	Adj. MS	F-Value	P-Value
Load (N)	4	0.05075	0.012687	3.63	0.028
% Vol. hBN	4	0.07014	0.017534	5.01	0.008
Error	16	0.05599	0.003500		
Total	24	0.17688			

The last column of Table 5 showing P-value indicating the significance of control parameters on the tribological performance of silicon nitride. % volume of hBN has P-value 0.008 is less than alpha value 0.05, indicating it is a more significant parameter affecting the tribological performance of silicon nitride.

5. CONCLUSION

In this study, an attempt has been made for simultaneous minimization of wear rate along with the coefficient of friction in SN-hBN/steel sliding pair under dry lubricating conditions so as to improve its overall tribological performance. Load and addition of hBN are two factors selected for optimization in order to improve tribological performance of Si₃N₄-hBN ceramic composite. Summarizing the experimental results, the following conclusions can be drawn:

1. Among all SN-hBN/steel sliding pairs, the best tribological performance was observed with the SN12/ steel pair under a load of 5N condition. This is the optimal condition of load and % volume of hBN for minimization of wear rate and coefficient of friction in Si₃N₄-hBN ceramic composite sliding against steel.
2. There may be a formation of a film or hydrated layers on the surface of the pin at 5N in the SN12 pin, due to the incorporation of hBN. The tribo-chemical reaction leads to the formation of layers on the pin surface protecting the pin surface from wear.
3. Under the all loading condition, the 12% volume of hBN was a reasonable amount for the formation of a protecting film on the surface. The protecting film could not be formed for SN0/steel, SN4/steel, and SN8/steel pairs under the same loading condition and this may be because of low hBN content. A high value of wear rate was observed in the SN16 /steel pair because of its poor physical and mechanical properties.
4. Based on ANOVA results, the most effective parameter is the addition of hBN in silicon nitride improving its tribological performance.

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