"TRIBOLOGICAL BEHAVIOR OF SILICON NITRIDE ON ADDITION OF HEXAGONAL BORON NITRIDE"

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Abstract - Ceramics are the excellent materials for the industrial use as a sliding part, in particulars Si_3N_4 a promising material. A low coefficient of friction for Si_3N_4 slide in distilled water has been reported. The friction coefficient can be further decrease by addition of hexagonal Boron Nitride (hBN). The main objective of this project is to study the tribological behavior of Silicon Nitride (Si_3N_4) – Hexagonal Boron Nitride (hBN) composite on the stainless steel. Unlubricated wear test were carried out with the help of Pin-On-Disc wear tester. The hBN is added in Si_3N_4 in different proportionate with the help of Planetary ball mill. The pins are formed by sintering & same were tested on Pin-on-Disc tester. The result shows:-1) The addition of hBN to Si_3N_4 resulted in sever decrease of friction coefficient. 2) Wear coefficient were below 10^{-4} mm^3 / Nm for all materials decrease sharply with increase in hBN volume friction. The analysis indicates that, without lubrication, Si_3N_4-hBN composites was spalled off during the wearing tests & spalling pits were formed on the wearing surface of Si_3N_4-hBN composites firstly & then the wear debris was embedded into the pits & oxidized, thus a tribofilm containing B_2O_3, SiO_3& Fe_2O_3 was formed on the wearing surface finally. This tribofilm can protect both Si_3N_4-hBN pin & stainless steel disc & lubricate the wear surface

Keyword: Wear, CNT, ANOVA, Pin-on-disc, hardness.

1. INTRODUCTION

Silicon nitride is non-oxide ceramic that is rarely observed in nature, but may occur naturally. Since it has been found in particles of meteorite rock, Synthetic silicon nitride was 1st developed by Deville and Wohler in 1859. But it remained little more than curiosity for nearly a century. Today, industrial use of silicon nitride and its composites are well established and include high performance bearings , turbine blades and glow plugs(i.e application that require a material with high fracture toughness strength and low wear properties) silicon nitride has been used in spinal fusion implants and has been developed for bearing components of prosthetic hip and knee joint Silicon nitride (Si3N4) based ceramics represent a combination of mechanical, tribological ,thermal and chemical properties that makes them suitable for high performance components in severe environments in several industrial applications. These properties, namely hardness, fracture toughness, friction and wear coefficient, are also very important for many high-load medical application of bio-ceramics in the human body. Another desirable property of ceramic-based implants is bioactivity. Hexagonal Boron Nitride (h-BN) is also known as 'White Graphite', has similar (hexagonal) crystal structure as of Graphite. This crystal structure provides excellent lubrication properties. Uses of Hexagonal Boron Nitride (h-BN)

• h-BN powder is used as a lubricant additive and can be dispersed in Lubricating Oil, Grease, Water and Solvents. When mixed with water and binders, it can also be applied as paint (for lubricity coating).

• Due to strong thermal resistance h-BN is used as an additive for high temperature lubrication.

• h-BN powder can be sprayed (similar to sand blasting) or can be sprinkled on hot surfaces (hot forging dies) to provide dry lubricity.

• h-BN being a good thermal conductor, it is used as an additive to various types of heat radiation material.

• Due to Non-Wetting property, h-BN is widely used in glass manufacturing process. A biomaterial is essentially a material that is used and adapted for a medical application. Biomaterials can have a benign function, such as being used for a heart valve, or may be bioactive; used for a more interactive purpose such as hydroxy-apatite coated hip implants. Biomaterials are also used every day in dental applications, surgery, and drug delivery

2. LITERATURE REVIEW

M.N.Rahaman et al. (2012) [1]: Silicon nitride (Si3N4) is a ceramic material used for industrial applications that demand high strength and fracture resistance under extreme operating conditions. Silicon nitride has unique tribological and physical properties like low mass density, low thermal expansion, high hardness, and excellent chemical stability over broad range of temperature makes it suitable for high speed application like bearings. Recently, Silicon nitride (Si3N4) has been used as an orthopedic biomaterial, to promote joint replacement and to developed bearings that can improve wear properties. This paper shows that unlike metals, Si3N4 semitransparent to X-rays and being nonmagnetic, it enables MRI of soft tissues proximal to Si3N4 implants.

S.Hampshire et al. (2007) [2]: Silicon nitride is one of the major structural ceramics that possesses high flexural strength, good fracture resistance, good creep resistance and high hardness. These properties arise because of the processing route which involves liquid phase sintering and the development of microstructures in which high aspect ratio grains and intergranular glass phase lead to excellent fracture toughness and high strength. This review has examined the development of silicon nitride and the related sialons and their processing into a family of structural ceramic materials with high hardness, strength, fracture toughness, creep resistance and wear resistance. The development of knowledge of microstructure–property relationships in silicon nitride materials has shown the importance of understanding the sintering process and the effects of grain boundary chemistry and structure on mechanical and thermal properties.

R.F. Silva et al. (2002) [3]: The main objective of this paper is to help on the clarification of the lack of consensus in the bibliographic data concerning the tribological behavior of Si3N4–BN composites. Unlubricated sliding tests by pin-on-disk were carried out with three grades of composite materials with 10, 18 and 25 vol% of BN. The addition of BN to the Si3N4 matrix resulted in a slight reduction of the friction coefficient, which decreased from 0.82 for monolithic Si3N4 to 0.67 for Si3N4–10%BN materials. Silicon nitride materials profit from the addition of boron nitride platelets to improve their tribological properties only if low volume amounts (10%) are incorporated. An improvement in wear resistance occurs when the BN platelets have their easy cleavage basal planes oriented parallel to the sliding surface, affording a lubricious effect. No BN hydration was detected in this study, most likely due to the moderate test conditions, which were insufficient to activate such chemical reactions with the surrounding environment.

M. Pettersson et al.(2013)[4]:Total joint replacements currently have relatively high success rates at 10-15 years, however increasing ageing and an active population places higher demands on the longevity of the implants. Si_3N_4 and $Si_xC_yN_z$ coatings have been deposited using HiPIMS in order to explore a wear resistant alternative for bearing surfaces in total joint replacements. This promising mechanical property may be explained by the coating's ability to avoid cohesive cracks. None of the Six CyNz coatings showed wear resistance matching that of Si3N4 or the SixNy. Coatings under the tested conditions Also, there is room for process optimization to increase the adhesion to CoCr substrates. Si_xN_y coatings are of great interest for further studies because of their combination of hardness, elastic modulus, and wear resistance. In particular the SiN coating, which exhibited the highest hardness (21GPa), one of the higher elastic moduli (212 GPa), the lowest wear rate ($1.3 \cdot 10^{-7}$ mm^3/Nm) and also fractured in a homogenous way in the scratch test.

S.G. Ghalme et al. (2013) [5]: The main objective of this paper is to investigate the effect of surface roughness and lubricant viscosity on coefficient of friction in silicon nitride- steel rolling contact. Two samples of silicon nitride with two different values of surface roughness were tested against steel counter face. The test was performed on four ball tester in presence of lubricant with two different values of viscosity. Taguchi technique a methodology in design of experiment implemented to plan the experimentation and same is utilized to evaluate the interacting effect of surface roughness and lubricant viscosity. Analysis of experimental results presents a strong interaction between surface roughness and lubricant viscosity on coefficient of friction in rolling contact. It is clear that surface roughness and lubricant viscosity has strong interacting effect on coefficient of friction in rolling contact. While results of ANNOVA table presents, percentage contribution of lubricant viscosity is about 44.18%, while the combined contribution of surface roughness and lubricant viscosity is about 54.93% on coefficient on friction in rolling contact. Implementation of Taguchi technique made it easy to obtain valid conclusion with only 4 number of experiment, it also proves importance of design of experiment in the field of research and development to obtain valid conclusion.

3. Methodology

3.1 Design of Experiment (DOE):-

The technique of defining and investigating all possible conditions in an experiment involving multiple factors is known as the design of experiments (DOE). DOE refers to the process of planning, designing and analyzing the experiment so that valid and objective conclusions can be drawn effectively and efficiently. Design of experiments (DOE) is a powerful technique used for exploring new processes, gaining increased knowledge of the existing processes and optimizing these processes for achieving world class performance. DOE is implemented in industry for achieving breakthrough improvements in product quality and process efficiency.

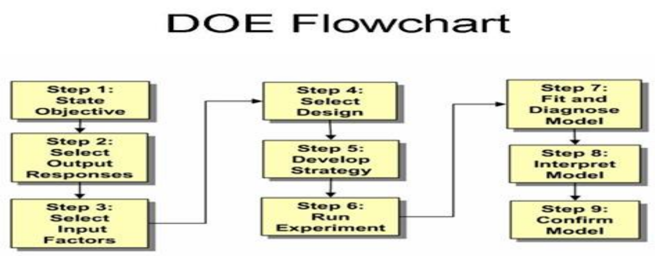

Fig 3.1 Flow chart of DOF

3.2 Taguchi Method:-

Taguchi Method have been widely utilized in engineering analysis and consist of plan of experiments with the objective of acquiring data in controlled way ,in order to obtain information about the behavior of a given process. Taguchi method is a powerful tool for the design of high quality systems, products and processes. It provides simple, efficient and systematic approach to optimize design of experiments for process or product improvement. Taguchi method is efficient method for designing process that operates consistently and optimally over a variety of conditions.

The steps applied for Taguchi optimization in this study are as follows:

- Select noise and control factor
- Select Taguchi orthogonal array.
- Conduct Experiment.
- Measurement of Wear, frictional force and temperature.
- Analyse results, (Signal to Noise ratio)

3.3 Orthogonal array:-

Classical experimental design methods are too complex and are not easy to use. A large number of experiments have to be carried out when the number of process parameters increase. To solve this problem, the Taguchi method uses a special design of orthogonal arrays to study the entire parameter space with only a small number of experiments. Orthogonal arrays (often referred to Taguchi methods) are often employed in industrial experiments to study the effect of several control factors. An orthogonal array is a type of experiment where the columns for the independent variables are "orthogonal" to one another. Orthogonal arrays are highly fractionated factorial designs.

To define an orthogonal array, one must identify:
1. Number of factors to be studied
2. Levels for each factor
3. The specific 2-factor interactions to be estimated
4. The special difficulties that would be encountered in running the experiment
Benefits of using orthogonal array:
1. Conclusions valid over the entire region spanned by the control factors and their settings.
2. Large saving in the experimental effort.
3. Analysis is easy.

4. Experimental Work

4.1 Selection of levels, Load parameters & % of h-BN:-

Silicon nitride (Si3N4) materials profit from the addition of hexagonal boron nitride (h-BN) to improve their tribological properties only if low volume amounts are incorporated. For this reason h-BN is added into silicon nitride in five different proportions as given in table no 3.1 below. Accordingly, five levels of loads are selected corresponding to five proportions.

Table no-4.1 Load & % of h-BN

Level Parameters	Level-1	Level-2	Level-3	Level-4	Level-5
Load (N)	5	10	15	20	25
h-BN (%)	4	8	12	16	0

4.2 Preparation of Test Samples

4.2.1 Mixing of powder:-

In order to prepare test samples in the form of small pin, first powder of silicon nitride (Si3N4) and hexagonal boron nitride (h-BN) is mixed together by percentage in five different proportions. The weight of each powder sample is 100gm (Si3N4+h-BN). Mixing of powder is carried out with the help of ball mill. As highest degree of fineness is required, planetary ball mill is used for mixing these samples.

4.2.2 Sintering:-

Sintering is a method for making objects from powder, by heating the material in a sintering furnace below its melting point (solid state sintering) until its particles adhere to each other. Sintering is traditionally used for manufacturing ceramic objects, and has also found uses in such fields as powder metallurgy.The temperature used for sintering is 1900 ^{0}C below the melting point of the silicon nitride and h-boron nitride. The Sintering of test samples is done at: VB Ceramic Chennai

Fig 4.1:- Sintered Samples

4.3 Selection Orthogonal Array:-

Two factors namely load and % of h-BN are considered as controlling factors. Each parameter has five levels denoted by 1,2,3,4 and 5 respectively. According to the

Taguchi method, for five levels and two parameters L25 orthogonal array should be employed for the experimentation.

No. of Experiments = Level Factor

Considering 2 factors and 5 levels:

No. of Experiments = $(Level)^{Factor} = 5^2 = 25$

Hence L25 Orthogonal array is selected for experimentation.

Expt. No	Load	% h-BN
1	5	4
2	5	8
3	5	12
4	5	16
5	5	0
6	10	4
7	10	8
8	10	12
9	10	16
10	10	0
11	15	4
12	15	8
13	15	12
14	15	16
15	15	0
16	20	4
17	20	8
18	20	12
19	20	16
20	20	0
21	25	4
22	25	8
23	25	12
24	25	16
25	25	0

4.4 Testing Parameters:-

Loading condition: 5N – 25N

Speed: 250 rpm.

Test duration: 20 Min.

Temperature: Environmental

4.5 Sample Specifications:-

Pin Diameter : 10mm

Pin Length : 15mm

Disc Diameter : 165mm

Disc Thickness : 8mm

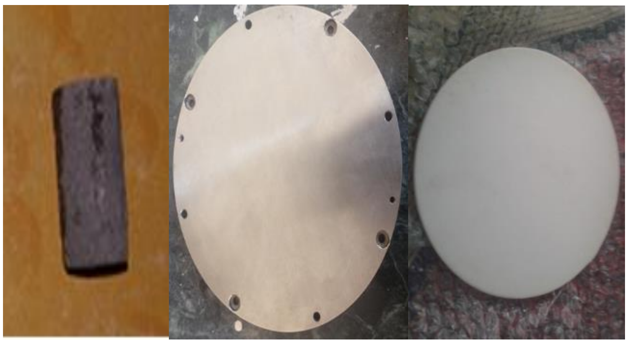

Fig.4.2 Pin Sample, Steel disc & Alumina disc

5. Analytical procedure

5.1 S/N Ratio:-

In Taguchi"s design method the design parameters (factors that can be controlled by designers) and noise factors (factors that cannot be controlled by designers, such as environmental factors) are considered influential on the product quality. The Signal to Noise (S/N) ratio is used in this analysis which takes both the mean and the variability of the experimental result into account. The S/N ratio depends on the quality characteristics of the product/process to be optimized. Usually, there are three categories of the performance characteristics in the analysis of the S/N ratio; that is, the lower is better (LB), the higher is better (HB), and the nominal is better (NB).

In this case wear volume loss is expected to be minimum, so S/N ratio is calculated for lower is better. The S/N ratio for each response is computed differently based on the category of the performance characteristics and hence regardless of the category the larger S/N ratio corresponds to a better performance characteristic. Once all of the S/N ratios have been computed for each run of an experiment, Taguchi advocates a graphical approach to analyze the data. In the graphical approach, the S/N ratios and average responses are plotted for each factor against each of its levels. From the graphs, lower the better values were selected and the confirmation tests were conducted.

S/N ratio can be calculated by using formula:

For Smaller is Better:

5.2 S/N Ratio for alumina Disc:-

$$SN_S = -10\log\left(\frac{\sum_{i=1}^{n} y_i^2}{n}\right)$$

Table no-5.1

Expt. No	Vol. loss (mm3/m)	S/N ratio for WVL	K (mm3/Nm)	S/N ratio for K
1	0.3022	10.3941	0.06044	24.3735
2	0.1990	14.0229	0.0398	28.0023
3	0.2444	12.2379	0.04888	26.2174
4	0.0337	29.4319	0.00675	43.4113
5	0.5055	5.9262	0.10109	19.9057
6	0.2007	13.9499	0.02006	33.9499
7	0.0156	36.1263	0.00156	56.1264
8	0.1438	16.8466	0.014377	36.8466
9	0.0204	33.7861	0.002045	53.7861
10	1.0867	-0.7222	0.10867	19.2778
11	0.3140	10.0614	0.02093	33.5832
12	0.0111	39.0935	0.00074	62.6154
13	0.1029	19.7517	0.00686	43.2735
14	0.0953	20.4163	0.00635	43.9381
15	1.3473	-2.5893	0.08982	20.9325
16	0.2063	13.7100	0.01031	39.7306
17	0.5002	6.0166	0.02501	32.0372
18	2.2799	-7.1583	0.11399	18.8623
19	0.2035	13.8287	0.01017	39.8493
20	0.3169	9.98155	0.01584	36.0022
21	0.5142	5.7774	0.02057	33.7362
22	2.1100	-6.4856	0.08440	21.4732
23	0.4144	7.6516	0.01658	35.6104
24	0.1551	16.1877	0.00620	44.4166
25	4.1178	12.2933	0.16471	15.6655

5.3 S/N Ratio for Steel Disc:-

Table no-5.2

Expt. No	Vol. loss (mm3/m)	S/N ratio for WVL	K (mm3/Nm)	S/N ratio for K
1	0.6671	3.5166	0.1334	17.4959
2	0.0652	23.7127	0.0131	37.6921
3	0.0381	28.3920	0.0076	42.3714
4	0.4209	7.5162	0.0842	21.4956
5	0.4983	6.0503	0.0996	20.0298
6	0.2214	13.0945	0.0221	33.0944
7	0.1480	16.5923	0.0148	36.5923
8	0.0655	23.6733	0.0066	43.6733
9	0.8590	1.3196	0.0859	21.3197
10	1.5294	3.6906	0.1529	16.3094
11	0.0621	24.1322	0.0041	47.6540
12	0.1869	14.5641	0.0125	38.0859
13	0.0126	37.9893	0.0008	61.5111
14	0.4835	6.3112	0.0322	29.8331
15	0.2615	11.6490	0.0174	35.1708
16	0.5498	5.1947	0.0274	31.2153
17	0.3556	8.9807	0.0177	35.0013
18	0.2116	13.4880	0.0106	39.5086
19	0.9441	0.4993	0.0472	26.5199
20	0.2061	13.7197	0.0103	39.7402
21	0.1153	18.7642	0.0046	46.7230
22	0.5194	5.6888	0.0208	33.6475
23	0.4758	6.4505	0.0190	34.4093
24	0.7868	2.0827	0.0315	30.0452
25	0.4672	6.6092	0.0187	34.5680

To obtain optimum proportion of h-BN in Si_3N_4 that gives optimum wear volume loss for each level of the factors, the S/N ratio for smaller-is-better is calculated.

1) From table no-5.1 below points are observed

Minimum wear volume loss - 0.0111 mm³/m

S/N ration for WVL - 39.0935

Minimum wear coefficient -0.00074 mm³/m

S/N ratio for wear - 62.6153

Hence from table it is clear that, the optimum proportion is obtained at 8% h-BN and 15N load.

2) From table no-5.2 below points are observed

Minimum wear volume loss - 0.0126 mm^3/m

S/N ration for WVL - 37.9893

Minimum wear coefficient -0.00084 mm^3/m

S/N ratio for wear - 61.5111

Hence from table it is clear that, the optimum proportion is obtained at 12% h-BN and 15N load.

6. Analysis using MINITAB-17

6.1 Analysis of variance (ANOVA):-

The ANNOVA is performed to investigate the design parameters and to indicate which parameters are significantly affecting the output parameters. This statistical significance of the factors can be evaluated through the analysis of variance (ANOVA). A statistical ANOVA is performed to find the contribution of each factor for attaining the process outcome. It is a mathematical technique which based on the least square approach. The experimental results are based on the analysis of average and analysis of variance. The analysis of variance and also Taguchi design is carried out by using Minitab 17 software which gives the graphical of various parameters.

6.2 Analysis of Alumina disc:-

The wear volume loss & S/N ratio for alumina disc is given in table no-6.1

Table No-6.1

Expt. No	Load	% h-BN	Vol. loss (mm3/m)	S/N ratio for WVL
1	5	4	0.3022	10.3941
2	5	8	0.1990	14.0229
3	5	12	0.2444	12.2379
4	5	16	0.0337	29.4319
5	5	0	0.5055	5.9262
6	10	4	0.2007	13.9499
7	10	8	0.0156	36.1263
8	10	12	0.1438	16.8466
9	10	16	0.0204	33.7861
10	10	0	1.0867	-0.7222
11	15	4	0.3140	10.0614
12	15	8	0.0111	39.0935
13	15	12	0.1029	19.7517
14	15	16	0.0953	20.4163
15	15	0	1.3473	-2.5893
16	20	4	0.2063	13.7100
17	20	8	0.5002	6.0166
18	20	12	2.2799	-7.1583
19	20	16	0.2035	13.8287
20	20	0	0.3169	9.98155
21	25	4	0.5142	5.7774
22	25	8	2.1100	-6.4856
23	25	12	0.4144	7.6516
24	25	16	0.1551	16.1877
25	25	0	4.1178	-12.2933

6.2.1 Main Effect Plot for Volume loss:-

The Taguchi experiment which was conducted can be used to analyze the effects of the selected process parameters on wear characteristics. In order to analyze the main effects, main effect plots were drawn using MINITAB 17 software.

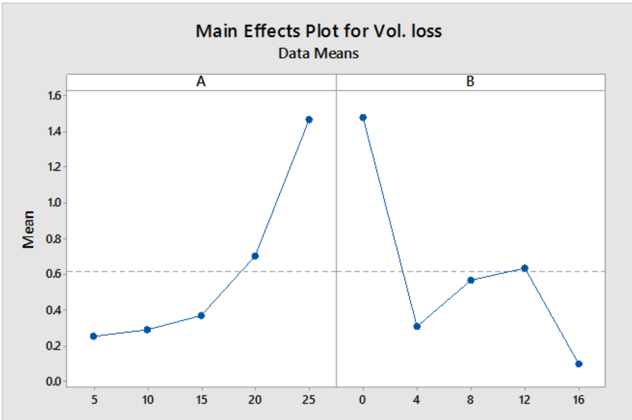

Fig. no. 6.1 Main effect plot for wear volume loss

From the main effects plot, it is clear that

1) With the increase in load, volume loss goes on increasing

2) Addition of h-BN in Si3 N4 leads to reduction in volume loss up to certain extent (8% h-BN) and further it goes on increasing.

6.2.2 Main effect plot for Wear coefficient (K):-

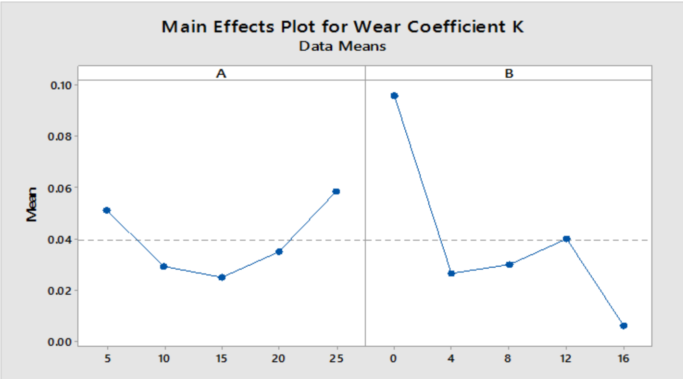

Fig. No. 6.2 Main effect plot for wear coefficient k

From the main effects plot for wear coefficient, it is clear that

1) With the increase in load wear coefficient goes on increasing.

2) Addition of h-BN in Si3 N4 leads to reduction in wear coefficient upto certain extent (8% h-BN) and further it goes on increasing.

6.3 Analysis of Steel disc:-

The wear volume loss & S/N ration for Steel disc is given in table no-6.2

Table No-6.2

Expt. No	Load	% h-BN	Vol. loss (mm3/m)	S/N ratio for WVL
1	5	4	0.6671	3.5166
2	5	8	0.0652	23.7127
3	5	12	0.0381	28.3920
4	5	16	0.4209	7.5162
5	5	0	0.4983	6.0503
6	10	4	0.2214	13.0945
7	10	8	0.1480	16.5923
8	10	12	0.0655	23.6733
9	10	16	0.8590	1.3196
10	10	0	1.5294	3.6906
11	15	4	0.0621	24.1322
12	15	8	0.1869	14.5641
13	15	12	0.0126	37.9893
14	15	16	0.4835	6.3112
15	15	0	0.2615	11.6490
16	20	4	0.5498	5.1947
17	20	8	0.3556	8.9807
18	20	12	0.2116	13.4880
19	20	16	0.9441	0.4993
20	20	0	0.2061	13.7197
21	25	4	0.1153	18.7642
22	25	8	0.5194	5.6888
23	25	12	0.4758	6.4505
24	25	16	0.7868	2.0827
25	25	0	0.4672	6.6092

6.3.1 Main effect plot for wear volume loss:-

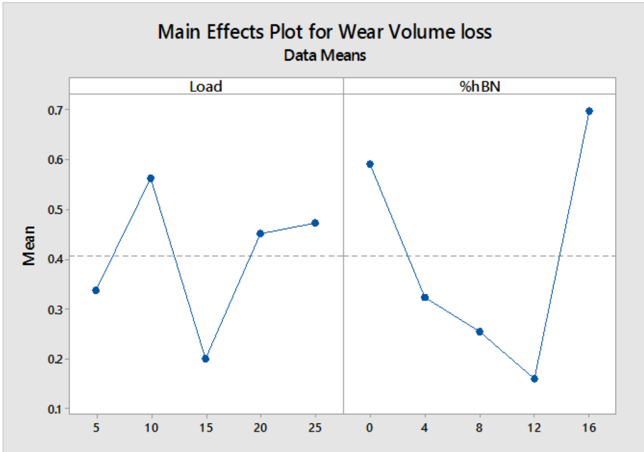

Fig. no. 6.3 Main effect plot for Volume loss

From the main effect plot, it is clear that

1. As the load increase the volume loss also goes on increasing.
2. Addition of h-BN in Si_3 N_4 leads to reduction in volume loss up to certain extent (12% h-BN) and further it goes on increasing.

6.3.2 Main Effects Plot for wear coefficient (K):-

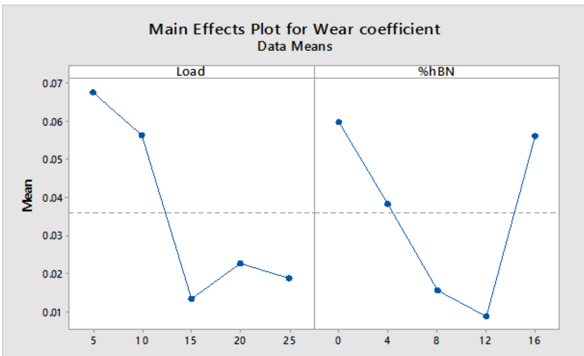

Fig. no 6.4Main effect plot for Wear Coefficient

From the main effect plot, it is clear that

1) As the load increase the Wear coefficient also goes on increasing.
2) Addition of h-BN in $Si_3 N_4$ leads to reduction in wear coefficient up to certain extent (12% h-BN) and further it goes on increasing

7. CONCLUSIONS

After conducting above experimentation the following conclusions are drawn:

The present investigation aimed at optimization of wear properties of Si3N4 and h-BN against Alumina counterface. This analysis was carried out based on L25 orthogonal array in Taguchi optimization technique. Main effect plots and interaction plots were drawn using Minitab 17 software which draws the following conclusions.

1. Wear properties Si3N4 and h-BN shows highly non-linear relationships with load and percentage of h-BN.

2. **Alumina disc:-** Experimental results shows minimum volume loss due to wear is 0.0111mm3/m. This minimum volume loss is observed at 8% of h-BN in silicon nitride. Analysis of variance for wear volume loss shows h-BN percentage has major contribution of 25.48% as compared to load having contribution of 23.51%. Also analysis of variance for wear coefficient (K) shows h-BN percentage has major contribution of 47.13% as compared to load having contribution of 8.66%. This is the optimum proportion of silicon nitride and hexagonal boron nitride to minimize wear against Alumina. This optimum proportion is the suitable alternative for Hip/Knee joint replacement.

3. Steel Disc:- Experimental results shows minimum volume loss due to wear is 0.01260473mm3/m. This minimum volume loss is observed at 12% of h-BN in silicon nitride with 15N Load. This is the optimum proportion of silicon nitride and hexagonal boron nitride to minimize wear against Steel.

This project illustrates the application of parameter design (Taguchi method) in the optimization of wear properties. The following conclusions can be drawn based on the above experimental results of this study. Taguchi‟s method of parameter design can be performed with lesser number of experimentations as compared to that of full factorial analysis and yields similar results. It is found that the parameter design of the Taguchi method provides a simple, systematic and efficient methodology for optimizing the process parameters.

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