HIGH TEMPERATURE TRIBOLOGICAL CHARACTERISATION OF SIALON CERAMICS

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Abstract

Advanced structural ceramics are presently used in several tribological applications, such as precision instrument bearings, automotive engine parts and cutting tools inserts. Design and selection of advanced ceramics for these applications require reliable data on the effects of temperature, load and environment on the tribological behaviour of these materials. In our present work, Tribological studies of SiAlON were carried out using reciprocating friction monitor machine. Different experiments were performed at high temperature by changing sliding distance, load time duration and point of contact. Different configurations were adopted by running ceramic pin on SiAlON disc and a ceramic ball on SiAlON disc. Dense micro sized SiAlON samples were fabricated by using different sintering additives to study the influence on friction and wear properties. Sliding wear test were conducted on these graded ceramic composites using Pin-On disc and Ball-On disc method.

Index Terms–Ceramics, SiAlON, Tribology, Wear Volume, Sliding Distance, Ceramic ball, Friction.

INTRODUCTION

Ceramics are inert compounds or combination of non-metallic solids with metallic elements or atoms, which are held together by strong atomic-force. Ceramics has been used for tribological purposes for decades. Although high manufacturing costs have limited their applicability, they have been used in special applications such as high temperature or highly corrosive environments and in situations that require high resistance to wear. The toughened ceramics and fibre reinforced composites that have recently been introduced have opened up new areas of applicability. Silicon nitride ceramic materials are considered among the most promising materials for advanced structural applications, due to their unique combination of excellent properties such as high strength, high hardness, high toughness, good oxidation resistance, good chemical stability, low coefficient of thermal expansion, and low density.

SiAlON is a solid solution of silicon nitride and alumina having the general formula $Si_{6-z}Al_zO_zN_{8-z}$. SiAlON is known for its low density, high strength, superior thermal shock resistance, fracture toughness, and chemical inertness. SiAlON ceramics, in comparison with Silicon Nitride ceramics, have better high-temperature properties and higher hardness due to the smaller amount of intergranular glassy phase [1].

SiAlON is currently used in various applications, such as cutting tools, induction heating, resistance welding, metal forming tools, diesel engine valves, valve guides, roller followers, tappet shims, seals, and hybrid bearings. All these examples show that SiAlON ceramic possesses a high potential for more applications in tribo-elements. In view of the increasing use of SiAlON ceramic for various applications, it is essential to understand the friction and wear behaviour of SiAlON under various conditions of rubbing, i.e. unidirectional and reciprocating sliding, and fretting [2]. Closely related to Silicon Nitride are the SiAlON ceramics, which are substitutional solid solutions with the Si and N elements being replaced by Al and O, respectively. α- and β- SiAlONs are isostructural with α- and β-Si_3N_4, and the two phases are totally compatible, which means that the overall phase constitution is essentially determined by the composition of the starting powder mixtures. Materials of the α phase consist of equiaxed grain structures, whereas the anisotrophic growth rates in β-Si_3N_4 results in micro-structure containing elongated β grains in a fine grained matrix. These differences in microstructure results in a range of ceramics with a wide range of mechanical properties.

Wear Properties

Because of their inherent brittleness, ceramics can wear by chipping. Surface and subsurface cracks form, join, and release small chips of material. A fine powder is produced as this wear debris is ground up in the wear process. Therefore, ceramics are sensitive to high contact stresses or to any contact condition leading to a state of stress that contains tensile components. Metals and plastics can deform plastically to relieve high contact stresses before fracture occurs. Ceramics can deform plastically under the hydrostatic stress associated with concentrated contacts, but the plastic deformation involved is very small, when compared with metals and polymers.

In instances of sliding contact, brittle materials can exhibit deformation-type wear. In cases of either abrasion or erosion, wear behavior is much different. Both abrasive and erosive processes involve tensile components in the contact state of stress. For abrasive wear, the wear rate is proportional to the elastic modulus and inversely proportional to hardness and fracture toughness. However, it has been shown that alumina and silicon carbides also exhibit a sensitivity to microstructure during wear by scratch tests [3]. The grain size and properties of the intergranular material were found to influence wear.

The thermal-shock-induced fracture of ceramics is a serious wear problem. Because of low thermal conductivity, large thermal gradients can develop in ceramics during frictional heating. When quenched, these hot spots develop large tensile stresses and cracks develop. The result is an increase in wear as relatively large pieces break out of the surface. Eventual failure by fracture can occur.

1.4. Mechanical and Physical Properties

The properties of select commercial ceramics that are typical of those used in tribological applications are given in Table1. Mechanical values vary in the literature, because ceramics properties are significantly influenced by manufacturing processes. Thus, one grade of ceramic can vary in hardness and toughness, depending on its manufacturer. Important data, such as thermal shock resistance, flexural strength, creep at elevated temperatures, and oxidation or decomposition data, are available only for certain types of ceramics.

As shown in Table 1, the hardness of ceramics is significantly higher than that of hardened tool steel, whereas ceramic density is lower. The ceramics also have greater strength values than steel. Many of the ceramics listed in the table are also stiffer than steel and therefore should have greater beam strength per square unit cross section.

Ceramics are ideal materials for high-temperature applications that can justify their cost. For example, the hot hardness value of silicon nitride is retained to about 1200°C (2190 °F). However, some ceramics (other than the oxides) can degrade at elevated temperatures. The carbides and nitrides are subject to oxidation. Those that contain silicon, such as silicon carbide and silicon nitride, depend on a film of silicon dioxide to protect their surfaces from oxidation. Above 1500°C (2730°F), the film softens, oxygen diffuses into the bulk material, and unacceptable oxidation of the body occurs. At elevated temperatures, alumina and zirconia, which are fully oxidized, will not oxidize, although the glassy binder in alumina tends to soften, and partially stabilized zirconia undergoes phase changes. Perhaps the greatest drawback to using ceramics in machinery is their brittleness, or low toughness.

Property	Al2O3	SiC	Si3N4	PSZ	SiAlON	TiC	WC/Co	Pyrex
Density, g/cm3	3.9	3.1	3.3	5.8	3.2	4.9	3	2.5
Thermal conductivity, W/m.K	35.6	126	27	1.8	21	35	1.6	1.5
Coefficient of thermal expansion, m/m · °C	7.1	4	2.3	10.1	3	8	6	56
Heat capacity, J/kg · K	880	2800	810	400	620	600		500
Hardness, HV · kgf /mm2	1500	2700	1300	1600	1780	3000	1500	
Young's modulus, Gpa	370	410	276	200	290	430	552	70
Tensile strength, Mpa	262	100	524	700	450	896	896	
Compressive strength, Mpa	2600			1850	3500			
Fracture toughness		4.6	4.5	9.5	7.7			

Typical mechanical and physical properties of generic ceramics

OUR WORK

SiAlON is a solid solution of Silicon Nitride and alumina having the general formula $Si_{6-z}Al_zO_zN_{8-z}$ where z varies from 0 to 4. In the present case the SiAlON was prepared by (CGCRI powder) AlN and Al_2O_3. The starting powders used to prepare the α- SiAlON ceramics were α-Si_3N_4 (H.C. starck, Germany d=0.38μm), Al_2O_3 (H.C. starck, Germany d=0.38μm), AlN (H.C. starck, Germany d=2μm) and Y_2O_3 (H.C. starck, Germany d=0.75μm). The powder mixtures of SiAlON composition were first planetary milled for 4hours in 2-butanol, using Al_2O_3 jars and Si_3N_4 balls, dried and sieved through an 80μm sieve. Then the powders were dispersed in 60-vol% methylethylketon and 40-vol% ethanol (Merck, Germany) followed by planetary milling for 4hours to prepare a stable suspension. These green bodies were then consolidated by slip casting by pouring suspension into Teflon rings set on absorbent plaster blocks. Pressureless sintering was achieved in a graphite furnace under pure Nitrogen atmosphere. They were initially heated to 1200°C at a rate of 20°C/min and then to 1750°C at a rate of 4°C/min, followed by holding for 2hours. After sintering, the samples were cooled down to room temperature at a rate of 20°C/min. Table1 shows the chemical composition of α- SiAlON ceramics.

AdvancedCeramic	Si_3N_4	AlN	Y_2O_3	Al_2O_3
α- SiAlON	71.88	16.26	9.33	2.53

Chemical Composition of α- SiAlON

Experimental setup

The reciprocating friction monitor tester was adopted for tribological studies and its schematic diagram is shown in fig. It consists of a lower test specimen on which an upper specimen oscillates under controlled loading. The frequency and amplitude can be set as per test requirements. There is also provision for heating of the lower specimen for conducting tests at elevated temperature. A piezoelectric force transducers employed to measure friction during tests. The pin on flat disc and a ball on flat disc configuration was adopted. The ceramic ball of Silicon Nitride 9mm in diameter was rubbed against SiAlON disc (ϕ20mm×6mm thick) and pin was rubbed against SiAlON disc.

Test Procedure

Initially both the specimen were ground and polished to a mean surface roughness of 0.1μm and then were ultrasonically cleaned and degreased in acetone bath for 5mins. After this the samples were dried in an Oven at 50^{o}C and were weighed repeatedly on a balance accurately. The pins were fixed to the load arm with a chuck. The pins stay over a disc (SiAlON) with two degrees of freedom, a vertical one, which allows normal load application by direct contact with the surface of the disc, and a horizontal one, for friction measurement. The ceramic ball was also rubbed against SiAlON disc and then after the test ball scar diameter was measured on travelling microscope. After the test, the process was repeated to measure the weight loss of SiAlON disc and pins. A constant plot of friction coefficient was taken throughout the experiments.

Wear testing of the SiAlON was conducted in dry air at a constant temperature of 500^{o}C. Sliding was reciprocating and continuous, the only interruptions being periodic measurement of weight loss due to wear. The test was run for maximum sliding distance of 90m at a sliding velocity of 0.08m/s. In all the experiments ceramic ball and a pin was rubbed against SiAlON disc for a series of prescribed time intervals such that wear of SiAlON, ceramic ball and pins could be evaluated as a function of sliding distance for a given contact load and sliding velocity.

For sliding velocity test, the SiAlON were worn against the ceramic ball for a constant and sliding distance to evaluate the variation of wear volume as a function of sliding velocity. Similarly the SiAlON were worn against titanium pin to evaluate the wear volume as a function of contact load.

Wear volume of ceramic disc was calculated from cumulative weight loss and density. The wear volume of ceramic ball was calculated from wear scar diameter by using following equations:

$$\text{Wear volume} = \pi h^2 (r-h/3)$$

$$\text{Scar depth (h)} = \frac{(d/2)^2}{r + \sqrt{r^2-(d/2)^2}}$$

r is radius of ball;

d is diameter of wear scar;

The coefficient of specific wear commonly used for comparison was calculated by the following equation:

$$\text{Coefficient of wear (k)} = \frac{\text{wear volume}}{\text{Load} \times \text{sliding distance}}$$

RESULTS

1. Friction behaviour as a function of sliding distance, load and temperature

The coefficient of friction as a function of sliding distance, load and temperature for different ceramic materials studied are shown in fig 1- 8. Fig 1 & fig 3 represents coefficient of friction, against load of SiAlON with TiN+Si_3N_4 pin and ceramic ball for sliding velocity of 0.08m/s.. Fig 2 and fig 3 represents coefficient of friction against sliding distance of SiAlON / TiN+Si_3N_4 and SiAlON / ceramic ball under a contact load of 20N. A constant temperature of 500^{0}C was maintained throughout these tests. Fig 5 represents the coefficient of friction of SiAlON / ceramic ball against different temperatures under contact load of 20N for sliding velocity of 0.08m/s.

It is evident from fig 1 and fig 3, that coefficient of friction increases with increase in load at constant temperature of 500^{0}C for SiAlON / TiN+Si_3N_4 pin and SiAlON / ceramic ball contact. It is because of large number of asperities are coming in contact as the load is increased. As the number of asperities are increasing, coefficient of friction will also increase. It is also evident from fig 2 and fig 3, that the coefficient of friction is decreasing with increase in sliding distance at constant temperature of 500^{0}C. It is also evident from figure 5 that slight decrease in coefficient of friction was observed at temperatures 200^{o}C to 400^{0}C from 0.315 to 0.301 and then at 500^{o}C coefficient of friction decreases from 0.301 to 0.118 with same pair of SiAlON / ceramic ball at 20N load.

2. Wear behaviour as a function of sliding distance, load and temperature

The wear as a function of sliding distance, load and temperature for different ceramic materials studied are shown in fig 8- 19. Fig 6- fig 9 represents wear against load of SiAlON with TiN+Si_3N_4 pin for sliding velocity of 0.08m/s. Fig 10 and fig 11 represents wear against sliding distance of SiAlON / TiN+Si_3N_4 under a contact load of 20N. Fig 12- fig 13 represents wear against load of SiAlON with ceramic ball for sliding velocity of 0.08m/s. Fig 14 and fig 15 represents wear against sliding distance of SiAlON / ceramic ball under a contact load of 20N. A constant temperature of 500^0C was maintained throughout these tests. It is evident from figure 6 and figure 12, that wear increases linearly under a contact load variation of 10N to 50N at constant temperature of 500^0C. However higher wear was observed in case of titanium silicon nitride pin and ceramic ball as compared to SiAlON as in fig 9 and 13. It is also evident from figure 11 & 15, wear volume increases linearly in SiAlON /ceramic ball contact and SiAlON /titanium silicon nitride pin as sliding distance gets increased. High wear resistance was offered by SiAlON and ceramic than SiAlON and titanium silicon nitride pin. High wear was also observed in case of SiAlON rubbing against titanium silicon nitride as compared to SiAlON rubbing against ceramic ball at 500^oC under contact load of 20N as can be seen from fig18 and fig 19.

3. Specific Wear rate as a function of sliding distance, load and temperature

The specific wear rate as a function of sliding distance, load and temperature for different ceramic materials studied are shown in fig 20- 26. Fig 20 & fig 22 represents the specific wear rate against load of SiAlON with $TiN+Si_3N_4$ pin and ceramic ball for sliding velocity of 0.08m/s. Fig 21 and fig 23 specific wear rate against sliding distance of SiAlON / $TiN+Si_3N_4$ and SiAlON /ceramic ball under a contact load of 20N. A constant temperature of 500^0C was maintained throughout these tests. Fig 24 represents the coefficient of friction of SiAlON /ceramic ball against different temperatures under contact load of 20N for sliding velocity of 0.08m/s. It is evident from figure 20 and 22 that specific wear decreases with the increase in load at constant temperature of 500^0C for SiAlON / $TiN+Si_3N_4$ pin and SiAlON /ceramic ball contact. It is also evident from figure 21 and 23, that wear rate decreases with increase in sliding distance at constant temperature of 500^0C under contact load of 20N. Specific wear of SiAlON rubbing against ceramic ball is higher than the specific wear of SiAlON when rubbed against titanium silicon nitride pin at temperature of 500^0C under contact load of 20N. But in both cases specific wear decreases linearly at constant temperature of 500^0C under contact load of 20N.

Conclusion

Due to high temperature, thermal and mechanical stability of ceramics and their ability to resist oxidation, these ceramics are going to be the future materials for use at high temperatures. Therefore it was essential to study high temperature performance of ceramics. In this study non lubricated tests were performed at low temperature of 200^0C and high temperature of 500^0C on SiAlON /ceramic ball contact and SiAlON /$TiN+Si_3N_4$ contact.

1. It was seen that the coefficient of friction at constant load of 20N at constant sliding distance of 48m, decreases from 0.315 to 0.118 with increase in temperature from 200 -500^0C.
2. SiAlON / ceramic ball and SiAlON /$TiN+Si_3N_4$ tests were then conducted at high temperature of 500^0C with varying loads from 20N to 40N load and constant sliding distance of 48m. It is evident from figure 7 the coefficient of friction against varying loads at constant temperature of 500^0C for SiAlON /ceramic ball contact is comparatively lesser than the coefficient of friction at constant temperature of 500^0C at constant sliding distance of 48m for SiAlON /$TiN+Si_3N_4$ contact.
3. It is also evident from figure 8, the coefficient of friction against sliding distance at constant temperature of 500^0C at contact load of 20N for SiAlON /ceramic ball contact is comparatively lesser than the coefficient of friction at constant temperature of 500^0C at contact load of 20N for SiAlON /$TiN+Si_3N_4$ contact.
4. The wear volume at different temperatures varying from 200-500^0C at constant contact of load of 20N at constant sliding distance of 48m, increases with increase in temperature. SiAlON / ceramic ball and SiAlON /$TiN+Si_3N_4$ tests were then conducted at high temperature of 500^0C with varying loads from 20N to 40N load and constant sliding distance of 48m. It is evident from figure-18 that wear volume against load, for SiAlON /ceramic ball contact is comparatively lesser than the wear volume for SiAlON /$TiN+Si_3N_4$ contact at constant temperature of 500^0C at constant sliding distance of 48m.
5. It is also evident from figure 19, wear volume against sliding distance at constant temperature of 500^0C at contact load of 20N for SiAlON / ceramic ball contact is comparatively lesser than the wear volume at constant temperature of 500^0C at contact load of 20N for SiAlON / $TiN+Si_3N_4$ contact.
6. The specific wear rate at different temperatures varying from 200-500^0C at constant contact load of 20N at constant sliding distance of 48m, increases with increase in temperature. SiAlON / ceramic ball and SiAlON /$TiN+Si_3N_4$ tests were then conducted at high temperature of 500^0C with varying loads from 20N to 40N load and constant sliding distance of 48m. It is evident from figure-25 that wear rate against load for SiAlON /ceramic ball contact is comparatively lesser than the wear rate for SiAlON /$TiN+Si_3N_4$ contact at constant temperature of 500^0C at constant sliding distance of 48m.
7. It is also evident from figure 26, wear volume against sliding distance at constant temperature of 500^0C at contact load of 20N for SiAlON /$TiN+Si_3N_4$ contact ball contact is comparatively lesser than the wear volume at constant temperature of 500^0C at contact load of 20N for SiAlON /ceramic ball contact.

Therefore by all these results we can find that SiAlON disc when rubbed against Ceramic ball is comparatively better than that, when SiAlON disc is rubbed against $TiN+Si_3N_4$ pin under same conditions in all respects of wear volume, coefficient of friction and wear rate.

Graphs and Plots

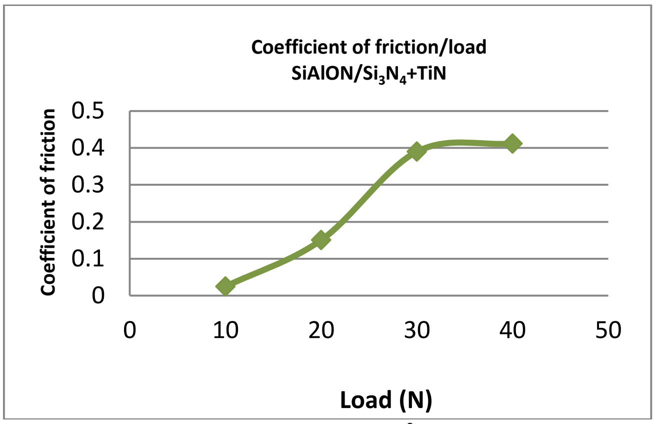


Fig 1:Vs=0.08m/s, T=500⁰C,Sd=48m

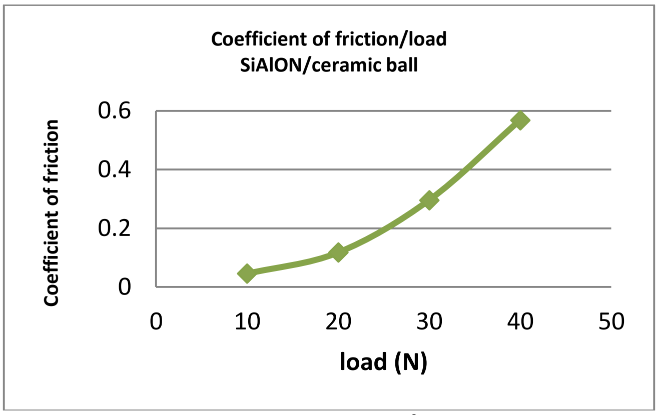


Fig 3: Vs=0.08m/s, T=500⁰C,Sd=48m

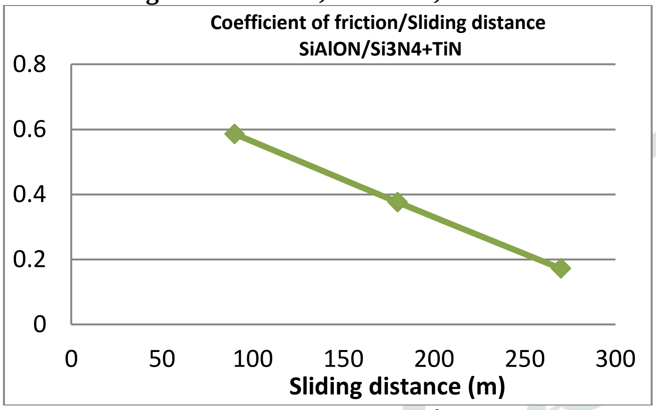


Fig 2: F=20N, T=500⁰C

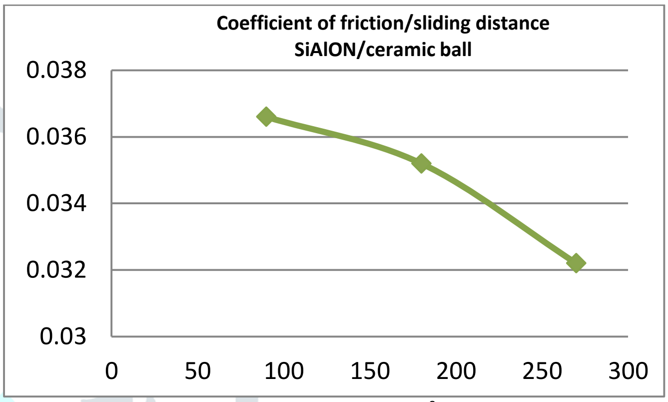


Fig 4: F=20N, T=500⁰C

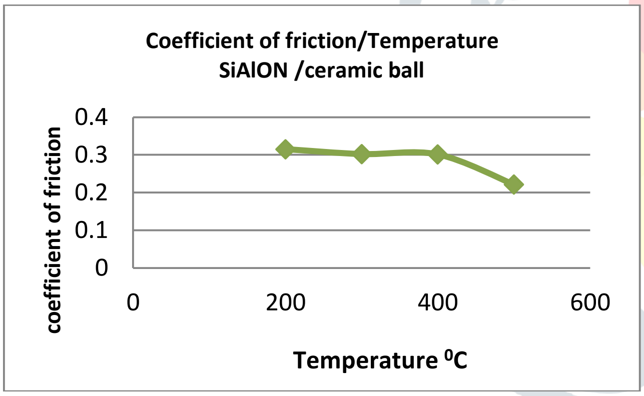


Fig 5: F=20N, Sd=48m,Vs=0.08m/s

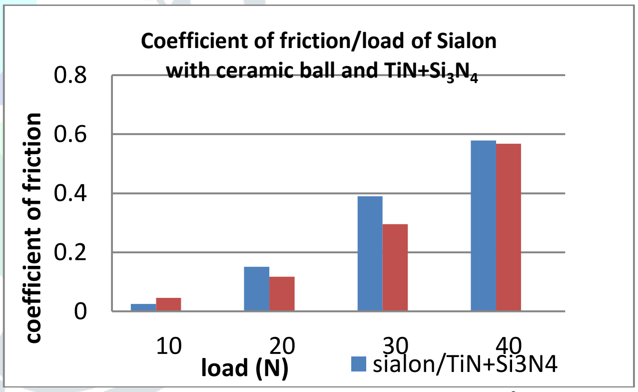


Fig 7: Sd=48m,Vs=0.08m/s, T=500⁰C

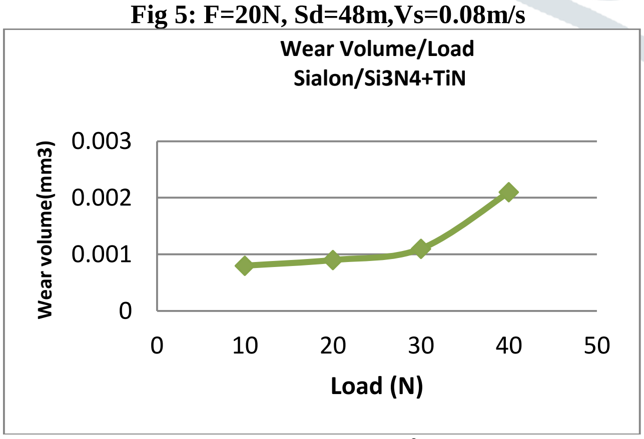


Fig 6: Vs=0.08m/s, T=500⁰C,Sd=48m

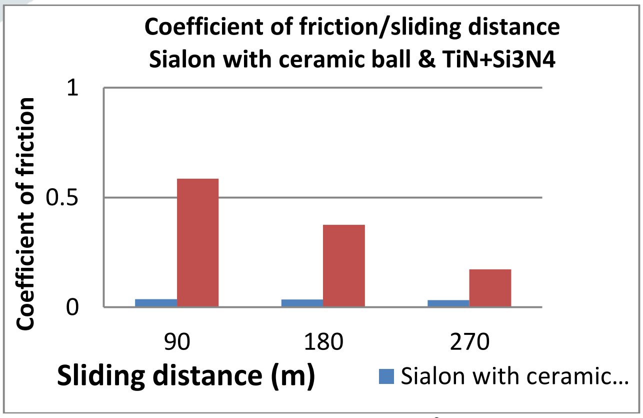


Fig 8: F=20N, T=500⁰C

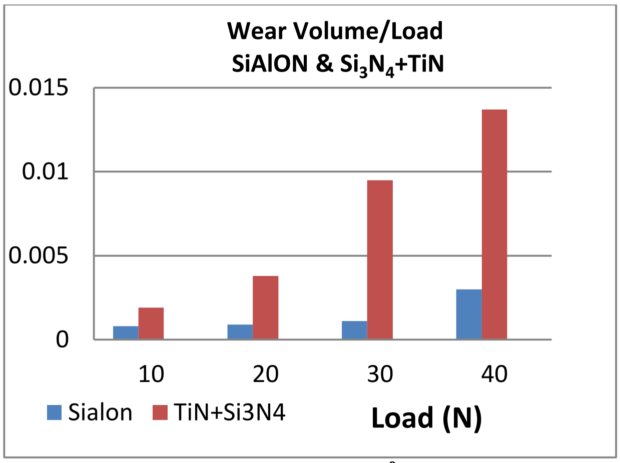


Fig 9: Vs=0.08m/s,T=500⁰C,Sd=48m

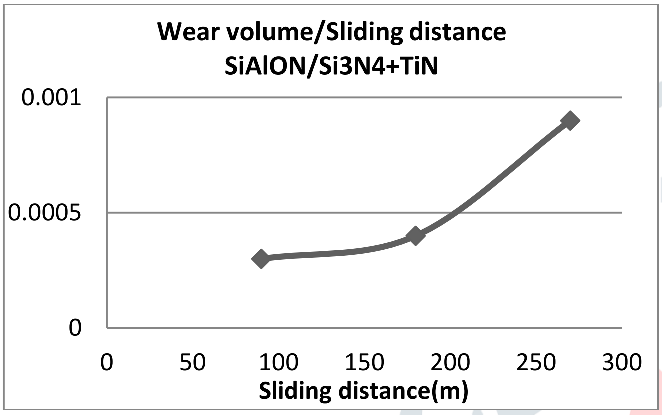


Fig 11:F=20N, T=500⁰C

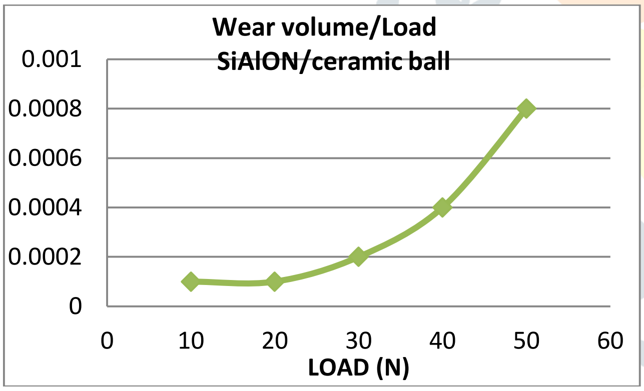
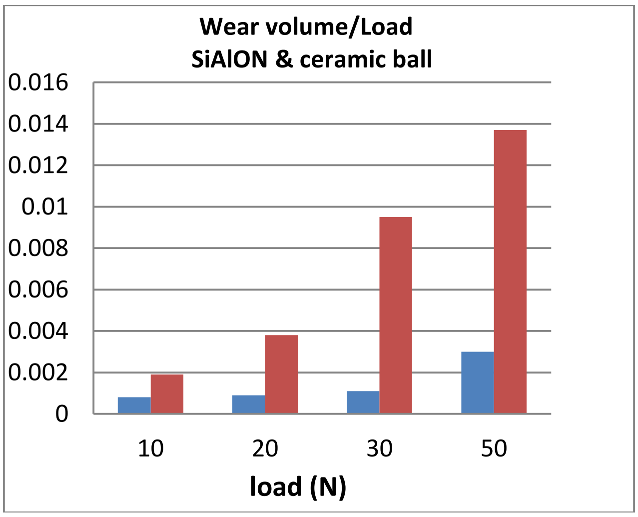


Fig 12: Vs=0.08m/s, T=500⁰C,Sd=48m



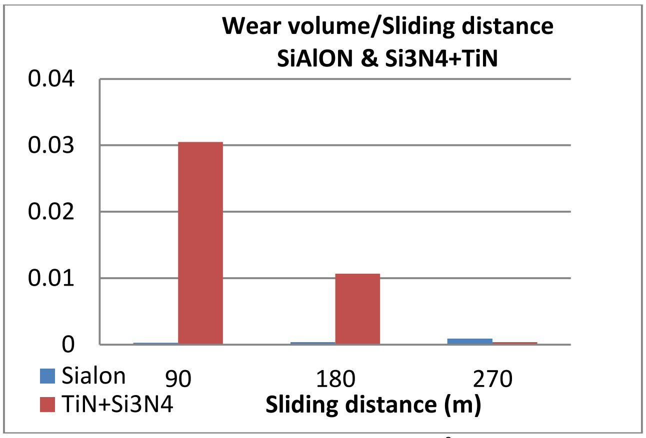


Fig 10: F=20N, T=500⁰

Fig 13:Vs=0.08m/s, T=500⁰C,Sd=48m

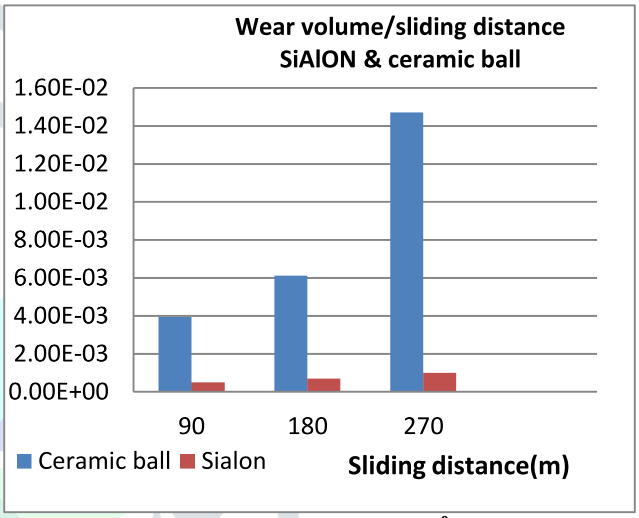


Fig 14: F=20N, T=500⁰C

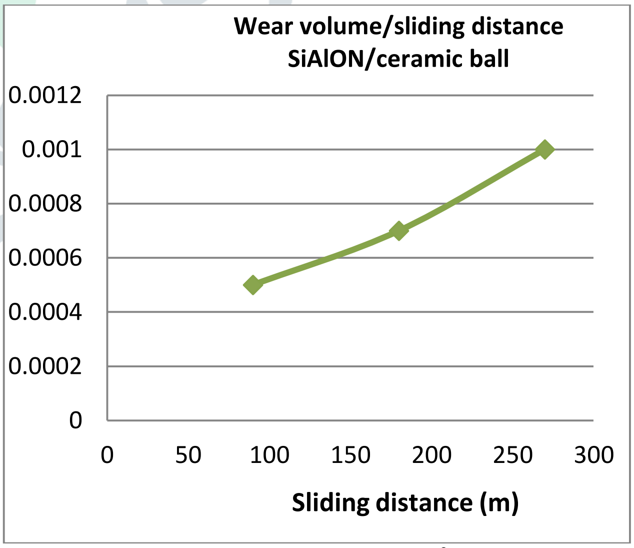


Fig 15: F=20N, T=500⁰C

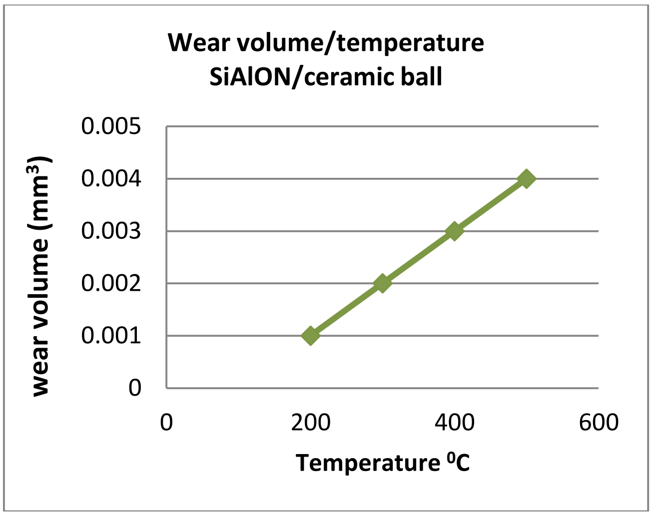


Fig 16:F=20N, Sd=48m,Vs=0.08m/s

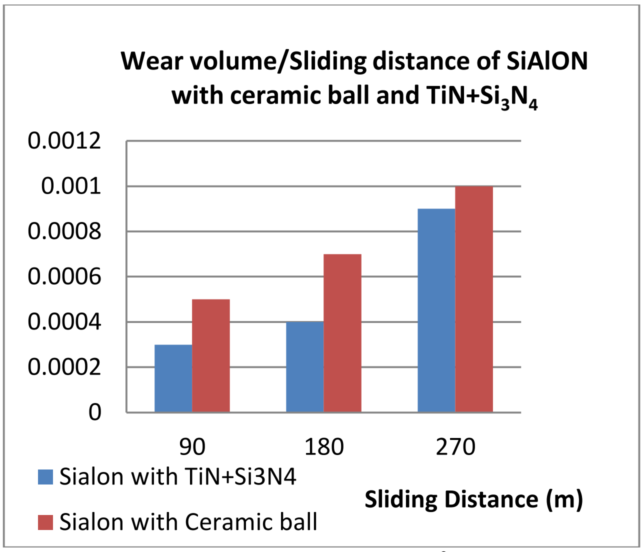


Fig 19: F=20N. T=500⁰C

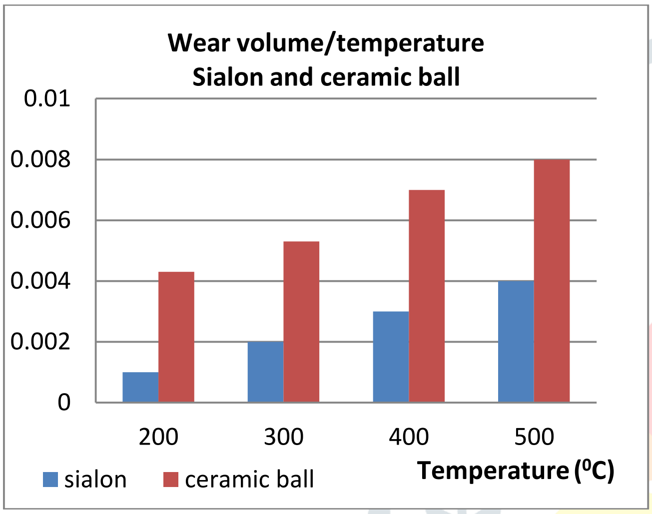


Fig 17: F=20N, Sd=48m, Vs=0.08m/s

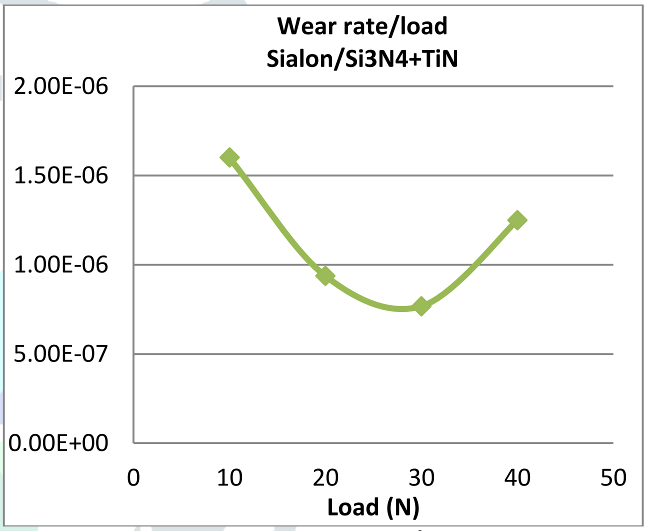


Fig 20: Vs=0.08m/s, T=500⁰C,Sd=48m

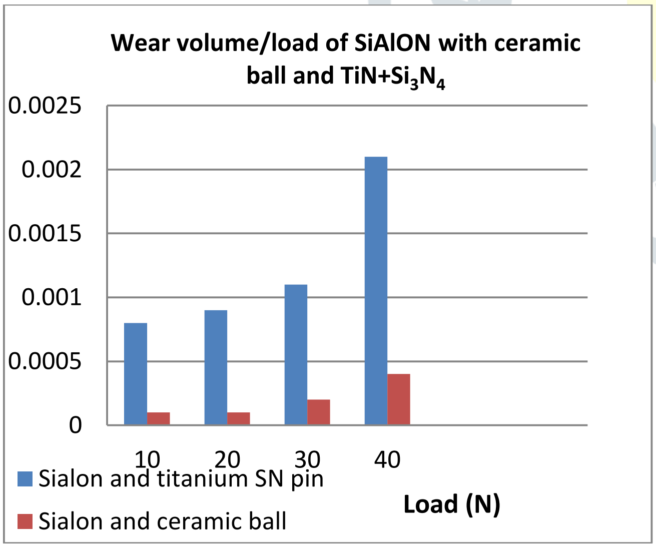


Fig 18: Sd=48m,Vs=0.08m/s, T=500⁰C

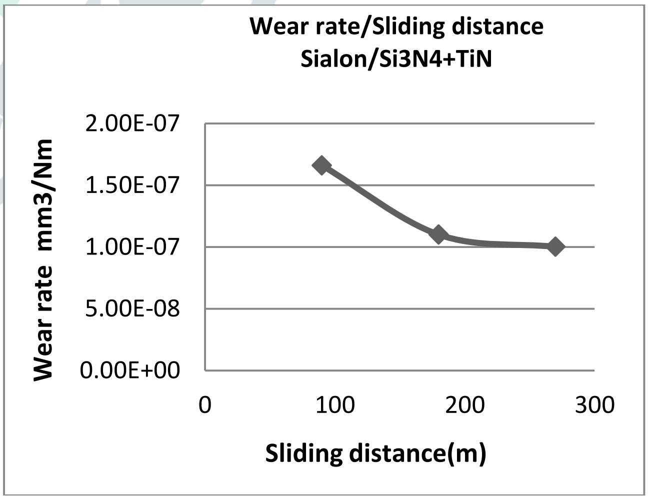


Fig 21: F=20N, T=500⁰C

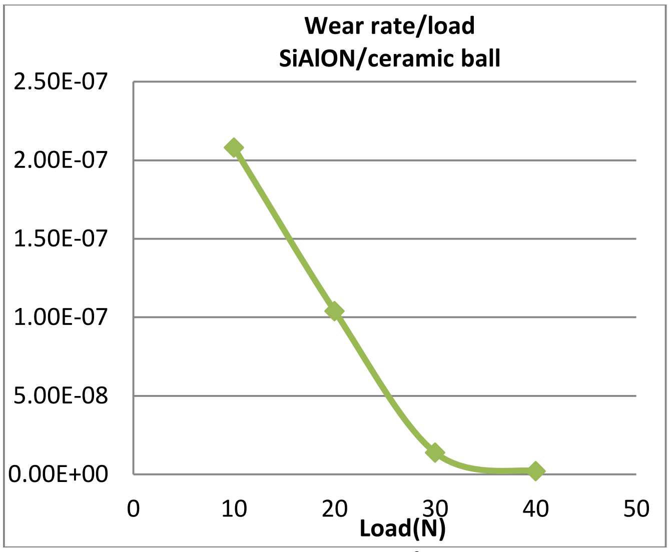


Fig 22:Vs=0.08m/s, T=500⁰C,Sd=48m

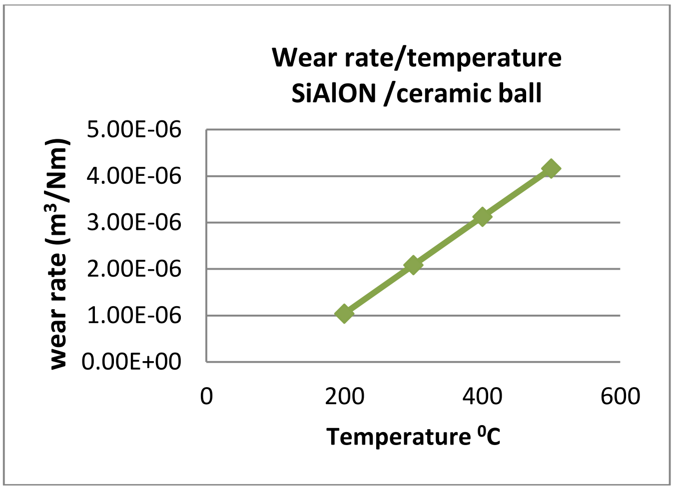


Fig 24:F=20N, Sd=48m,Vs=0.08m/s

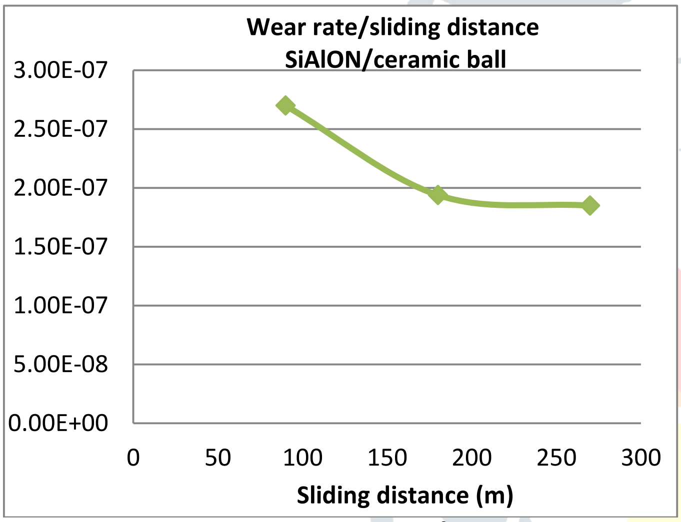


Fig 23: F=20N, T=500⁰C

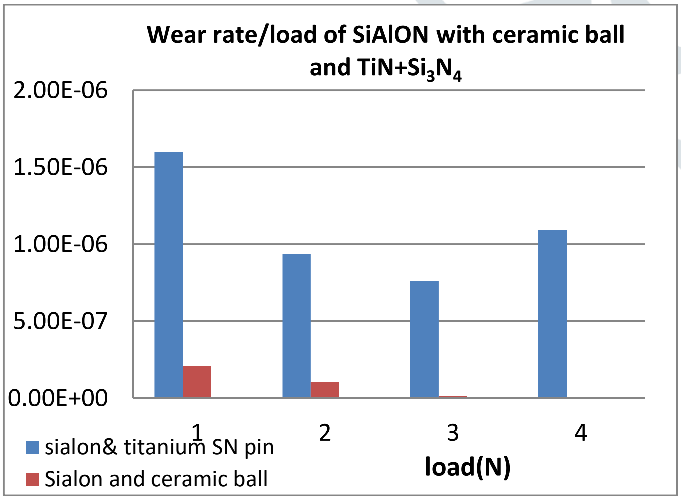


Fig 25: Sd=48m,Vs=0.08m/s, T=500⁰C

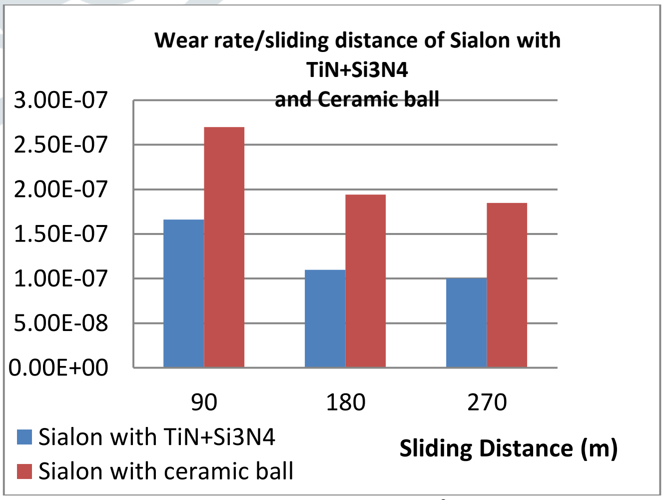


Fig 26: F=20N, T=500⁰C

load(N)	coefficient of friction	Wear volume(mm3)	Wear rate(mm3/Nm)
10	0.0252	0.0008	1.6E-6
20	0.151	0.0009	9.375E-7
30	0.390	0.0011	7.6E-7
40	0.598	0.0021	1.093E-6

Table 2: Coefficient of friction, wear volume, wear rate at temperature 500^{0}C with different varying loads of SiAlON/titanium silicon nitride contact.

Sliding distance	μ	Wear volume	Wear coefficient
90	0.586	0.0003	1.66E-7
180	0.376	0.0004	1.1E-7
270	0.172	0.0009	1.0E-7

Table 3: Sliding distance test at temperature 5000C with contact load of 20N of SiAlON/titanium silicon nitride contact.

Load	Coeff of friction	Wear volume	Wear rate
10	0.0462	0.0001	2.08E-7
20	0.118	0.0001	1.04E-7
30	0.296	0.0002	1.38E-8
40	0.568	0.0004	2.083E-9

Table 4: Coefficient of friction, wear volume, wear rate at temperature 5000C with different varying loads of SiAlON/ceramic ball contact.

Sliding distance	μ	Wear volume	Wear rate
90	0.0366	0.0005	2.7E-7
180	0.0352	0.0007	1.94E-7
270	0.0322	0.0010	1.85E-7

Table 5: Sliding distance test at temperature 5000C with contact load of 20N of SiAlON/ceramic ball contact.

Temperature	μ	Wear volume	Wear rate
200	0.315	0.001	1.04E-6
300	0.302	0.002	2.083E-6
400	0.301	0.003	3.125E-6
500	0.118	0.004	4.16E-6

Table 6: Coefficient of friction, wear volume, wear rate at constant load of 20N at different varying temperatures of SiAlON/ceramic ball contact.

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