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Wear of Nitrogen Ceramics and Composites in Contact with Bearing Steel Under Oscillating Sliding Condition

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Abstract: The wear of nitrogen based ceramics in contact with bearing steel balls (SAE 52100) was investigated under reciprocating sliding conditions at a velocity of 0.1 m/s, and 20 N, 40 N and 100 N load. Three types of ceramics were studied: viz. hot pressed silicon nitride (HPSN) sintered with selected liquid in the system yttria–aluminium nitride–silica and composites of HPSN with BN and TiC; SiAlON formulated with different amounts of alumina and silica; and alumina–titanium nitride composite having 60 mol% TiN. Among the HPSN composites, HPSN 15 vol% TiC had the lowest average wear factor (K) of 2.0×10^{-5} $mm^3/m/N$. The SiAlONs, in general, had the highest K and this increased with increasing O concentration. The lowest K value for SiAlON ceramic was 6.9×10^{-5} $mm^3/m/N$. The high K value of SiAlON is attributed to O substitution, which promotes adhesive wear resulting in mutual material transfer. The lowest wear of all the ceramics tested was found for the alumina–titanium nitride composite, the wear factor being of the order of 4.4×10^{-7} $mm^3/m/N$, which is one order of magnitude lower than the other nitrogen ceramics. In the case of HPSN and alumina–titanium nitride composites, abrasive wear and wear due to microfracture were the main wear mechanisms. Wear of the steel ball was lowest when in contact with HPSN–TiC composites, being almost equal to that of the ceramic, whereas the alumina–titanium nitride composite wore away the steel almost 20 times faster than the ceramic and will thus be unsuitable as a counterface material for 52100 steel. © 1997 Elsevier Science Limited and Techna S.r.l.

1 INTRODUCTION

Nitrogen ceramics, mainly those based on silicon nitride, are useful engineering materials suitable for applications at high temperatures where even the best metal alloys give poor performance. Because of their high strength at high temperatures, excellent creep properties, low density, high hardness and resistance to corrosion and oxidation, silicon nitride based ceramics are useful materials for use as bearings and wear parts. Amongst other nitride ceramics, titanium nitride has been widely used as a seize resistant coating on cutting tools. Wear of reaction bonded Si_3N_4 and HPSN were first studied by Godfrey and Taylor,[1] Chiu and Dalal[2] and Dalal *et al.*[3] A Si_3N_4–steel sliding pair was studied by Sutor[4] and a Si_3N_4–TiC composite by Blanchard-Ardid and Page.[5,6] Wear of Si_3N_4 at elevated temperature[7] and in different environments[8–10] has been studied. Little data, however, exist on the wear and friction properties of TiN composites. In previous communications, the role of sintering aids in dense Si_3N_4 and the degree of substitution in SiAlON on the wear and friction properties were reported.[11,12] In this work, the wear of Si_3N_4 and its composites with BN and TiC, and of SiAlON and Al_2O_3–TiN composites sliding against 52100 bearing steel were studied.

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Table 1. Properties of nitrogen ceramics

Ceramics	Density (g/cm^3)	MOR (MPa)	Creep strain rate (/h)	K_{IC} (MPa·m$^{1/2}$)	VMH (kg/mm^2)	E (GPa)
HPSN	3.12	575	0.45×10^{-6} (1250°C, 300 MPa)	8.52	1854	390
HPSN+15 vol% TiC	3.70	408	—	6.59	1818	437
SiAlON x=0.5	3.05	315	—	3.40	1500	—
SiAlON x=1.0	3.13	320	0.70×10^{-6} (1200°C, 175 MPa)	3.40	2149	200
Al_2O_3–TiN	4.34	430	—	4.70	1800	353

2 EXPERIMENTAL

The experiments were carried out under oscillating sliding conditions at 50 cycles per second, with an amplitude of 1 mm. This is a very severe condition under which fatigue failure is likely to occur, resulting in a high rate of wear. An SRV Optimol friction tester (Optimol Instrument GMBH, D-8000 Munchen-80, Germany) was used. The sliding pair consisted of a ceramic disc (ϕ25 mm×8 mm thick) as the fixed lower specimen and a bearing steel ball (SAE 52100) which formed the upper oscillating specimen. The speed and distance travelled could be changed by adjusting the frequency, amplitude of oscillation and duration of test. A piezoelectric force transducer was used to determine the coefficient of friction. The test could be carried out under different atmosphere and the temperature could be varied up to 250°C. All wear tests were carried out at a constant temperature of 50°C and in dry air containing around 5 vpm moisture unless otherwise stated. The sliding pairs were cleaned ultrasonically in benzene for 7 min. and dried at 50°C for 15 min. The diameter of the wear scar was measured using a microscope capable of reading 0.1 mm. The wear volume (V_w) of the ceramic disc was obtained by measuring the weight loss in a microbalance (after carefully removing any adhering particles) and from the density. The wear of the ball was calculated based on the scar diameter.

The ceramic discs were prepared by hot pressing in a BN coated graphite die at 28 MPa and at 1750°C for Si_3N_4 based materials and at 1600°C for the Al_2O_3–TiN composite. The ceramic samples were ground and polished with diamond grits, finishing with 4 and 2 μm diamond spray on a polishing cloth. A typical surface finish was: average height of peaks or valleys = 0.60 μm; average maximum peak height over mean = 0.60 μm; and maximum peak to valley height = 1.04 μm.

2.1 Types of ceramic samples

A complete list of ceramics studied is given in Table 1. The Si_3N_4 samples were pressure sintered with 11.8 wt% liquid in the system Y_2O_3–AlN–SiO_2, having the composition $Y_{7.5}Si_{13.2}Al_{20.7}O_{37.7}N_{20.8}$.[13] The composites were prepared with this composition as the base by adding 10, 15 or 20 vol% of TiC or hexagonal BN and hot pressing using identical conditions. It may be noted that the proportion of liquid sintering aid was consequently lowered.

The SiAlON, which is a solid solution of Al_2O_3 in Si_3N_4, has the general formula $Si_{6-x}Al_xO_xN_{8-x}$, where x may vary from 0 to 4.2 and is based on the β-Si_3N_4 structure. SiAlONs having values of x of 0.5, 1.0 and 2.0 were prepared[14] from end components by adding excess SiO_2 to the extent of 1.5%, 0.75% and 0.2% for the three SiAlONs, respectively. The O content in the three SiAlONs varied from 2.5 at% to 14 at%.

The TiN based composites were prepared by hot pressing an intimately mixed powder Al_2O_3 and 60 mol% TiN at 1450°C in N_2 at 30 MPa.[15] The relevant properties of the ceramics are given in Table 1.

3 RESULTS AND DISCUSSIONS

3.1 Wear factor of Si_3N_4 and composites

The wear volume (V_w) has been plotted against sliding distance in Fig. 1. The hot pressed Si_3N_4 (HPSN) shows an almost linear increase in V_w,

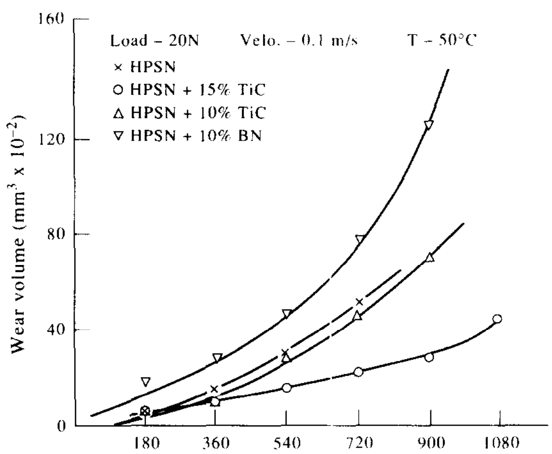

Fig. 1. Wear volume (V_w) plotted against sliding distance (S_D) for HPSN and composites. Load = 20 N, velocity = 0.1 m/s, temperature = 50°C, in air.

Fig. 2. Photograph showing microfracture in HPSN–10 vol% TiC composite.

with a slight upward curvature after a slow rise up to 180 m of sliding. The slope of the curve is 9.2×10^{-4} mm^3/m and the average wear factor (K) is 6.9×10^{-5} mm^3/m/N. K is defined as:

$$K=\frac{V_w}{S_D.P}$$

where S_D is the sliding distance and P is the load. The V_w of the composites containing 10 vol% TiC

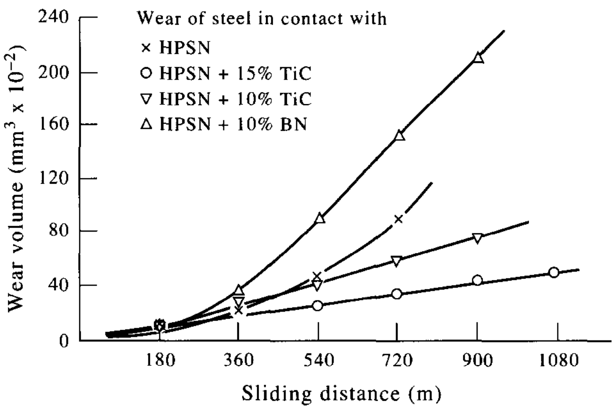

Fig. 3. V_w of steel in contact with HPSN and composites.

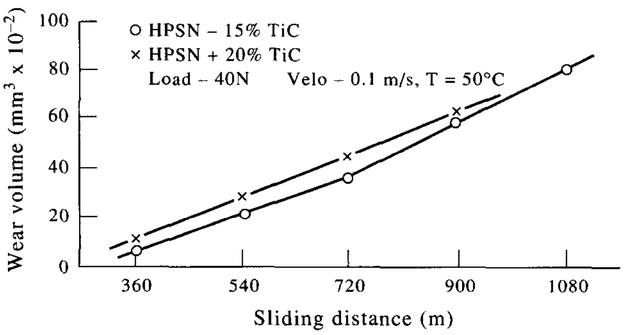

Fig. 4. V_w and S_D for HPSN–15 vol% and 20 vol% TiC at 40 N load and 0.1 m/s velocity.

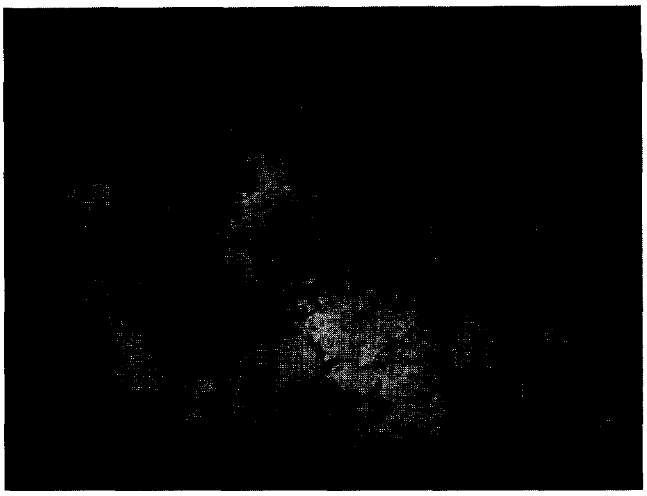

Fig. 5. SEM photograph of BN composite showing microfracture.

is lower than HPSN, and the V_w vs S_D curve shows a distinct upward curvature and does not follow Archard's law,[16] where a linear relationship with distance is expected. According to this law:

$$V_w=\frac{K}{3}\left(\frac{P.S_D}{H}\right)$$

where H is the hardness of the material. The significant upward curvature after about 360 m of sliding may be due to the appearance of cracking at contact points due to fatigue. Figure 2 shows a photograph of a Si_3N_4–TiC (10 vol%) composite where micro-chipping is evident. The holes in the microstructure may be due to the removal of TiC grains during rubbing.

The composite containing 15 vol% TiC, on the other hand, shows a slow linear rise in V_w up to 720 m sliding, showing a slight upward curvature thereafter where accelerated wear may commence. The 15 vol% TiC composition shows lower wear than the base HPSN and HPSN + 10% TiC composite, the value of K being 2.0×10^{-5} mm^3/m/N. Blanchard-Ardid and Page[5,6] also found this

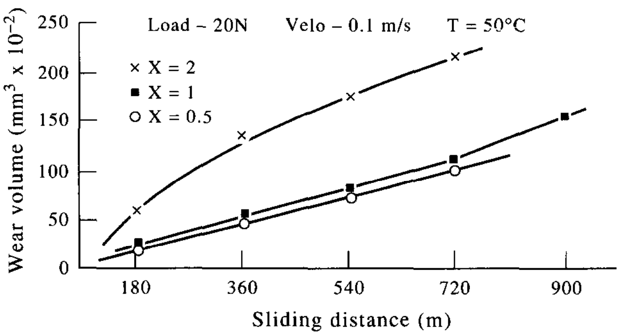

Fig. 6. V_w of SiAlON (x = 0.5, 1.0 and 2.0) plotted against S_D. Load = 20 N, velocity = 0.1 m/s, temperature = 50°C, in air.

particular composite to be around three times more wear resistant than the base Si_3N_4. They found the wear resistance to improve further with the addition of SiC whiskers. As the SiC percentage goes up to 20 vol% the wear rate increases, although it is still lower than the base HPSN. The HPSN 10 vol% BN composite has a higher wear than the base HPSN. The Knoop hardness of this composite at 1000 g load is 1390 kg/mm^2, as compared to 1750 kg/mm^2 for the base HPSN and 1821 kg/mm^2 for the 15% TiC composite The hardness therefore seems to control the wear of the composites.

The wear of the steel balls in contact with the HPSN has a more pronounced upward curvature (Fig. 3) than that of its mating ceramic — the total V_w being nearly double that of the ceramic for 720 m of sliding and the K value being 5.5×10^{-5} mm^3/m/N. The ball in contact with the 15 vol% TiC composite has a linear increase in wear, apparently obeying Archard's law. The average

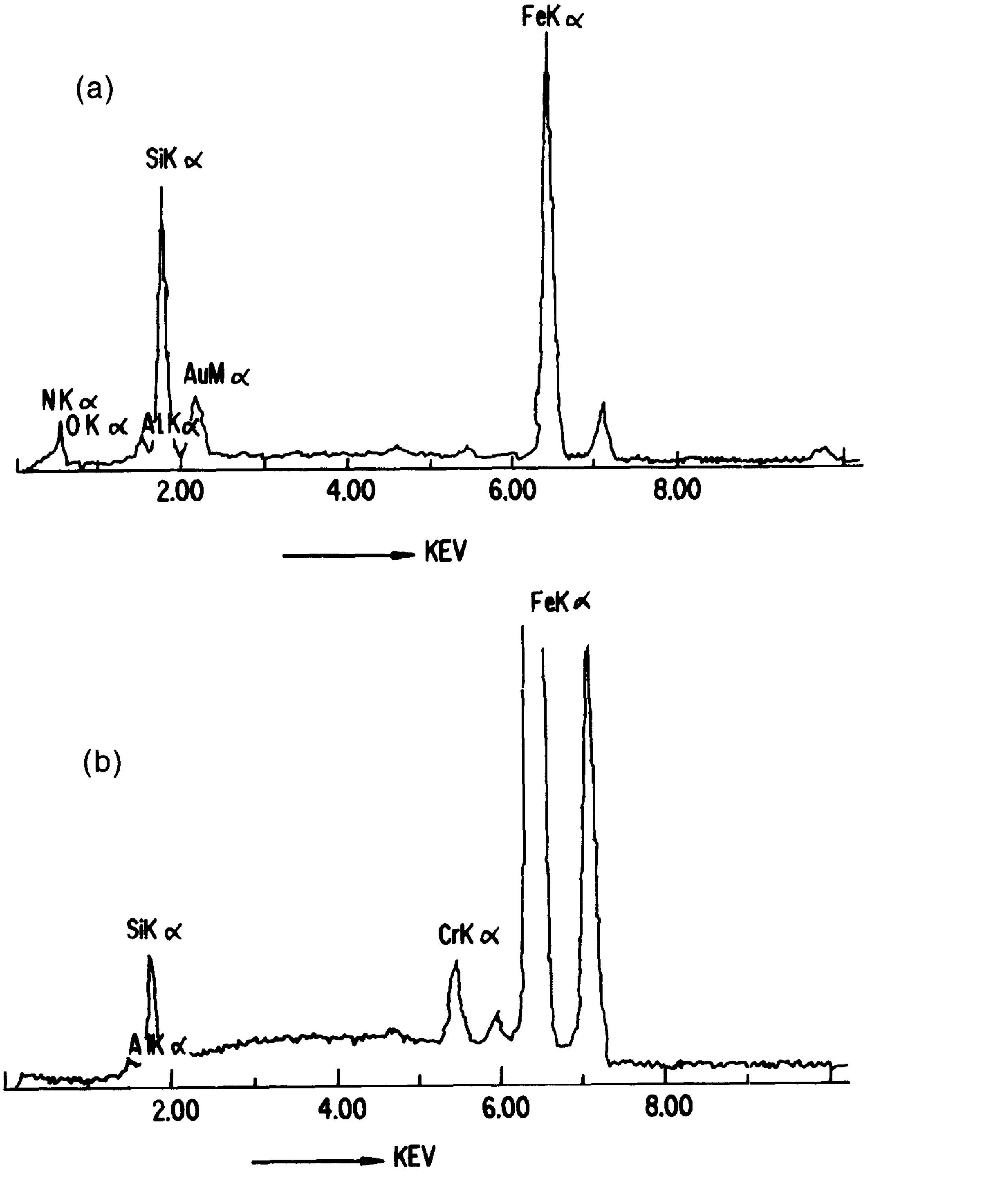

Fig. 7. EDAX of SiAlON and steel ball showing mutual transfer of materials: (a) ceramic, (b) steel ball. $S_D = 360$ m, velocity = 0.1 m/s, load = 20 N at 50°C in air.

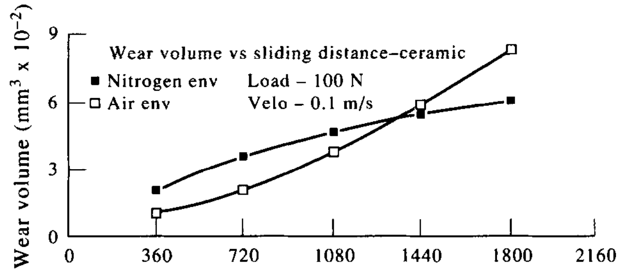

Fig. 8. V_w vs S_D for Al_2O_3–TiN composite in N_2 and air. Load = 100 N, velocity = 0.1 m/s.

wear factor is 4.6×10^{-5} mm³/m/N, which is only slightly higher than the ceramic. Similarly, the V_w of steel in contact with the 10 vol% BN composite shows a linear rise after 360 m of sliding. TiC is known to be converted into TiN by reaction with Si_3N_4 or in the presence of N_2 in the sintering atmosphere.[17] TiN has a non-seizing character with respect to steel and such improvement of wear behaviour and reduction of wear of both the ceramic and the steel ball may be due to formation of TiN in the composite, which exists along with TiC. This result, compared at 40 N load, is shown in Fig. 4. The wear rate, however, remains constant at 1.8×10^{-5} mm³/m/N, which is about 2.0×10^{-5} for the 20 N load experiment.

15 vol% TiC in HPSN appears to be the optimum composition for improved wear resistance.

The composite with 10 vol% BN shows, upon microscopic examination, ceramic fragments produced by microfracture (Fig. 5). The debris thus produced may bring about wear of the steel balls by normal abrasive wear and the wear equation follows.

3.2 Wear of SiAlON

The wear of SiAlON of the three compositions, viz. $x = 0.5$, 1.0 and 2.0, are shown in Fig. 6. The first two SiAlONs show wear rates which are very close to each other, although the $x = 2$ SiAlON shows

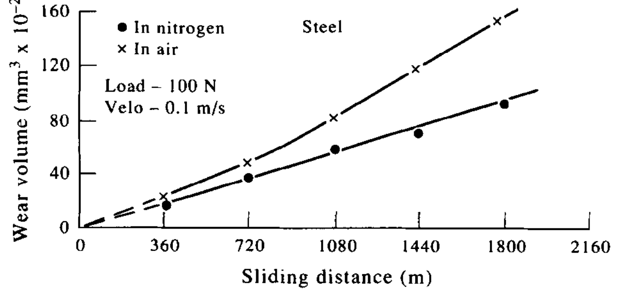

Fig. 9. V_w of steel in contact with Al_2O_3–TiN composite in N_2 and air. Load = 100 N, velocity = 0.1 m/s.

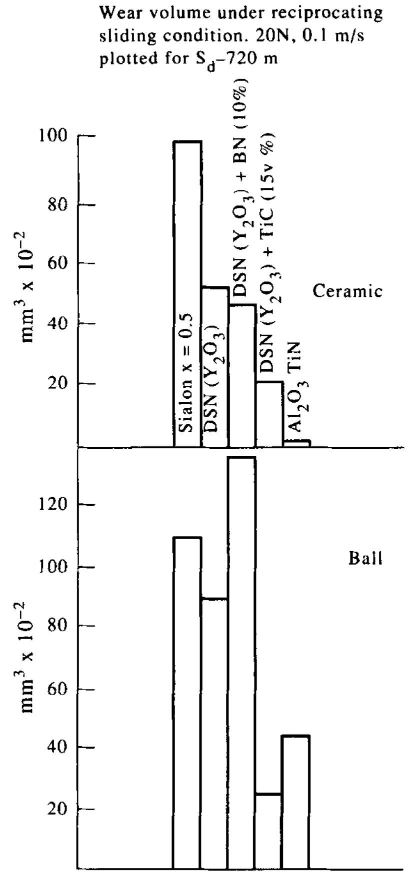

Fig. 10. Comparative V_w of various ceramics and steel.

almost double the wear rate. The wear volume varies linearly with distance in the case of the first two, whereas in the case of $x = 2$ the initial rapid wear seems to slow down after 360 m of sliding. It is apparent that the wear decreases in the order $x = 2 > 1 > 0.5$. The wear factors of the three SiAlONs $x = 0.5$, 1.0 and 2.0 are 6.9×10^{-5} mm³/m/N, 8.3×10^{-5} mm³/m/N and 1.5×10^{-4} mm³/m/N, respectively. The wear factor therefore becomes double for $x = 0.5$, and almost four and a half times for the $x = 2$ SiAlON as compared to dense Si_3N_4. This is attributed to the increasing O content in the SiAlONs, giving rise to adhesive wear and mutual transfer of materials in the sliding pairs, as shown in the EDAX of the ball and ceramic after the wear test done after carefully removing the debris (Fig. 7). The V_w remains the same, irrespective of the variation of the load up to 100 N, and decreases with sliding velocity up to 15 m/s for 360 m of travel. High load and velocity may bring about softening of aspereties due to generation of heat and may be responsible for reduced wear. The same observations are true for HPSN as well.

3.3 Wear of Al_2O_3–TiN composite

The V_w vs S_D curve shows an upward trend for experiments done in dry air, whereas that done in

Table 2. Wear factor and Archard's coefficient of nitrogen ceramics

Ceramic	Wear factor (K) (mm^3/mN)	Archard's coefficient (k)
HPSN	3.40×10^{-5}	1.93×10^{-3}
HPSN+15 vol% TiC	2.10×10^{-5}	1.13×10^{-3}
SiAlON x=0.5	6.90×10^{-5}	3.12×10^{-3}
SiAlON x=1.0	8.30×10^{-5}	5.35×10^{-3}
Al_2O_3–TiN	4.72×10^{-7}	2.40×10^{-4}

N_2 shows a downward trend (Fig. 8). This is attributed to the oxidation of TiN in air, which starts at 800°C.[15] Oxidation produces a loosely adhering TiO_2, which gets easily removed during sliding. The average value of K is 4.72×10^{-7} $mm^3/m/N$ and 1.94×10^{-7} $mm^3/m/N$ for the two atmospheres, respectively. As compared to Si_3N_4 and SiAlON, this composite is almost 100 times more wear resistant. The V_w of the steel in contact with the ceramic shows an almost linear rise with sliding distance (Fig. 9), the value being 15–20 times higher than the ceramic. This is in contrast with HPSN and SiAlON, where the steel has slightly higher wear than the ceramic pair. The K value for the steel does not vary with S_D and remains constant at an average value of 4.5×10^{-6} $mm^3/m/N$ over a S_D of 1800 m.

A comparative analysis of V_w for the various nitrogen ceramics and composites and of mating steel balls are plotted in Fig. 10. The wear factor and the Archard's wear coefficient (k) are given in Table 2. k is the probability that a junction formed during sliding will be removed. The Si_3N_4 based ceramics fall within the same order of magnitude, while the Al_2O_3–TiN composite has a value two orders of magnitude lower. The best result is obtained with the HPSN–15 vol% TiC composite, where the ceramic and steel wear at the same rate. Although the V_w of Al_2O_3–TiN composite is low, it appears to be too abrasive towards the steel ball. Therefore, in the case of a steel–nitrogen ceramic sliding pair, HPSN sintered with Y_2O_3 additive and its composite with TiC appear to have a performance above the others.

4 CONCLUSION

(1) HPSN with 15 vol% TiC has the lowest wear factor as compared to the base HPSN, as well as SiAlONs.

(2) The wear factor of SiAlON is higher than the HPSN and it increases with an increase in O in the general formula of SiAlON $Si_{6-x}Al_xO_xN_{8-x}$, where x represents the amount of O substitution.

(3) The Al_2O_3–TiN composite shows a very low wear rate in N_2 and is almost one order of magnitude lower than the other nitrogen ceramics in air.

(4) The wear of the steel mating pair is lowest for HPSN and composites, being almost equal to that of the latter ceramic; but the Al_2O_3–TiN composite wears away the steel almost 20 times faster, showing its unsuitability as a steel mating pair.

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