Original Article

Tribological behaviour of alumina-based nanocomposites reinforced with uncoated and Ni-coated cubic boron nitride

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\textbf{A R T I C L E  I N F O}

Article history:
Received 15 November 2018
Accepted 27 June 2019
Available online 23 August 2019

Keywords:
Alumina nanocomposites
Cubic boron nitride
Microstructural analysis
Wear
Tribophysics

\textbf{A B S T R A C T}

Currently, there is a rising demand to develop composite materials with outstanding tribological properties along with excellent thermal-mechanical properties for harsh tribological applications. In view of this demand, the tribological behaviour of spark plasma sintered (SPS) nano-alumina (~150 nm)-based composites reinforced with 10, 20 and 30 wt.% uncoated and nickel-coated cubic boron nitride (cBN) particles was evaluated using a ball-on-disc configuration against three different counterpart materials (WC, Si\textsubscript{3}N\textsubscript{4} and Al\textsubscript{2}O\textsubscript{3}) under dry sliding conditions. A variety of techniques were used to evaluate the phase composition and to study the dispersion, wear behaviour and wear mechanisms. The nano-alumina composites exhibited improved wear resistance and coefficient of friction (COF). These results were attributed to the unique combination and uniform dispersion of the reinforcing particles within the composites. Moreover, Ni-coated cBN particles displayed high wear resistance, primarily due to the excellent interfacial bonding provided by the nickel coating. The wear resistance of the sample reinforced with 30% cBN-Ni was improved by at least 85%. The wear mechanisms involved during sliding were abrasion, matrix grain pullout (dislodgement of matrix material), microcracking, chipping, and tribolayer formation.

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https://doi.org/10.1016/j.jmrt.2019.06.050
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1. Introduction

There is currently an increasing demand for inexpensive, wear-resistant, environmentally friendly and high-performance materials that can be utilized in industrial applications involving severe tribological conditions. One of the most abundant and widely used ceramic materials is alumina (Al₂O₃), which has numerous tribological applications, such as cutting tools, seal rings, wear pads, aircraft brakes and components of bearings, due to its excellent properties, including chemical and thermal stability, high hardness, good mechanical strength, excellent corrosion and wear resistance. However, a major drawback of alumina is its brittle nature and low fracture toughness [1,2], which may lead to unexpected catastrophic failure. To be highly functioning, abrasives and cutting materials should have characteristics such as high hardness (greater than that of the workpiece to withstand the wear action during cutting), high toughness (to bear vibrations or interruptions during the machining process) and the ability to conduct heat (resulting from friction at the cutting tool-workpiece interface) away from the workpiece [3].

Previously, researchers [2,4–14] reported that the mechanical and wear properties of monolithic Al₂O₃ was improved by reinforcing it with second-phase particles such as TiC, TiB₂, SiC, partially stabilized zirconia (PSZ), strontium, short nickel fibres, carbon nanotubes and graphene. The promising wear properties of cubic boron nitride (cBN), the hardest material after the diamond, as a reinforcing agent in the alumina matrix have received limited attention. cBN has extraordinary properties such as high resistance to chemical attack, thermally stable up to a temperature of 1200 °C, hardness, Young’s Modulus and thermal conductivity close to that of diamond and very little affinity to iron-based alloys [15,16]. However, some limitations include the fact that cBN, when subjected to conventional sintering processes, does not undergo proper densification because of its strong covalent nature and the proclivity of its structure to transform to the undesired softer hexagonal boron nitride (hBN) phase [17–19].

The tribological and mechanical properties of composites have been well established to be strongly dependent on their final microstructures formed during processing, which can vary based on the manufacturing process used [20,21]. Spark plasma sintering (SPS), due to its excellent control of the final microstructure, has been reported to be an efficient consolidation process to produce a coherent body with increased density and improved mechanical properties [22–27]. Moreover, SPS provides the possibility to control undesirable phase transformations (e.g., cBN to hBN) [28–30] due to its very rapid heating/cooling rates together with low sintering temperatures. Hotta and Goto [30] evaluated the effects of various concentrations of cBN (from 0 to 30 vol.%) and sintering temperatures (between 1200 and 1600 °C) on the hardness and fracture toughness of cBN/Al₂O₃ composites. The composites reinforced with 20 vol% cBN and sintered at 1300 °C showed excellent mechanical properties. Klimeczik et al. [3] compared the physical, mechanical and tribological properties of cBN/Al₂O₃ composites prepared using SPS and high-pressure high-temperature (HPHT) processes. They found that cBN/Al₂O₃ composites prepared using SPS showed better wear resistance than those produced using HPHT. Shonhiwa et al. [31] improved the mechanical properties of alumina matrix by reinforcing it with cBN (10–30 vol.%). Some of the most recent developments in enhancing the physical and mechanical properties of cBN/Al₂O₃ composites have included the use of sintering binders such as Ni nanoparticles [32] or the use of SiO₂-coated cBN [33] or Ni and Ni-coated cBN [26,34] in the Al₂O₃ matrix. These binders and coated cBN forms have been separately shown to assist in strengthening the interfacial bonding and suppressing the cBN-to-hBN phase transformation at particular processing parameters. However, to our knowledge, the synergistic effect of the Ni-coated cBN reinforcement on the tribological performance of cBN/Al₂O₃ composites has not been investigated for highly demanding tribological applications, such as abrasive and cutting/grinding tools.

In a previous study [34], we evaluated the effect of Al₂O₃ particle size (micro versus nano) on the physical, structural and thermomechanical behaviours of Al₂O₃-cBN and Al₂O₃-cBN/Ni composites prepared using SPS. We found significant improvement in the above-mentioned properties in the case of the nanosized alumina matrix, where no cBN-to-hBN phase transformation occurred, compared with micro-sized alumina matrix, where this transformation was clearly observed. Moreover, the presence of nickel along the grain boundaries not only suppressed the transformation of cBN to hBN but also improved the interfacial bonding strength and hence improved the mechanical properties.

In the present work, we investigated the tribological properties and performance of nanosized alumina matrices reinforced with various amounts of uncoated and Ni-coated cBN reinforcements against different counterface ball materials to evaluate their viability for industrial applications involving harsh tribological situations. We evaluated the effect of reinforcement percentage on a specific set of tribological parameters (load, speed, and sliding distance) to determine the optimized composition for improving tribological performance under this particular condition.

2. Experimental details

2.1. Materials

Alpha alumina powder with a particle size of 150 nm was procured from Chempur (Germany) and used as the matrix. Cubic boron nitride (cBN) particles with and without Ni coating (60 wt.%) and with an average particle size of 42 µm were procured from Element Six (USA) and used as reinforcements. Notably, in our previous study [34], we evaluated the effect of Al₂O₃ particle size (micro versus nano) on the physical, structural and thermomechanical behaviours of Al₂O₃-Ni, Al₂O₃-cBN and Al₂O₃-cBN/Ni composites [26,34]. With the aim of studying the tribological performance of these developed composites (in the current study), we used similar compositions to determine the relationship among mechanical, thermomechanical and tribological properties. Furthermore, cBN reinforcement 42 µm in size was selected based on prior work.
Table 1 – Classification of the composites and their respective sample IDs. The average dimension of the cBN particles was 42 μm [34].

<table>
<thead>
<tr>
<th>Sample IDs</th>
<th>Alumina</th>
<th>cBN-uncoated (Ni)</th>
<th>cBN-coated (Ni) (60 wt.%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>S1</td>
<td>S2</td>
<td>S3</td>
<td>S4</td>
</tr>
<tr>
<td>Composition (wt. %)</td>
<td>Pure Al₂O₃</td>
<td>10% cBN</td>
<td>20% cBN</td>
</tr>
<tr>
<td>Matrix (grams)</td>
<td>9</td>
<td>8.1</td>
<td>7.2</td>
</tr>
<tr>
<td>Reinforcement (grams)</td>
<td>0</td>
<td>0.9</td>
<td>1.8</td>
</tr>
</tbody>
</table>

* Pure Al₂O₃ (150 nm) is referred to as nanosized alumina.

Table 2 – Properties of counterface balls (provided by Anton Paar) used in the current study.

<table>
<thead>
<tr>
<th>Ball Material</th>
<th>Diameter (mm)</th>
<th>Roughness (Rₐ) (µm)</th>
<th>Hardness (HV)</th>
<th>Thermal conductivity (W/mK)</th>
<th>Compression strength (MPa)</th>
<th>Elastic modulus (GPa)</th>
<th>Fracture toughness MPa·m⁰.⁵²</th>
</tr>
</thead>
<tbody>
<tr>
<td>Tungsten carbide</td>
<td>6.3</td>
<td>0.25</td>
<td>1600</td>
<td>100</td>
<td>5700</td>
<td>640</td>
<td>9.9</td>
</tr>
<tr>
<td>Silicon nitride</td>
<td>6.3</td>
<td>0.02</td>
<td>1600</td>
<td>40</td>
<td>3000</td>
<td>320</td>
<td>7</td>
</tr>
<tr>
<td>Alumina</td>
<td>6.3</td>
<td>0.02</td>
<td>1650</td>
<td>29</td>
<td>2400</td>
<td>380</td>
<td>4</td>
</tr>
</tbody>
</table>

2.2. Sample preparation

Table 1 shows the compositions (wt.% of the starting powders used to prepare the samples along with their respective sample identifications. For the Ni-free samples (S2–S4) the mass of reinforcement (10–30 wt.%) was calculated with respect to mass of composite material (mass of alumina + mass of the cBN). In case of samples where Ni additive was used (S5–S7), to have a similarity in the analysis, mass of reinforcement (10–30 wt.%) was calculated using the same criteria (i.e. mass of Ni was not taken into the account). The sample preparation procedure described in the following text is the same as that reported in our previous work [34]. An ultrasonic probe sonicator (Model VC 750, Sonics, USA) was used to uniformly mix each sample in a separate volume of ethanol. Afterwards, these mixtures were dried in a furnace at 80 °C for 24 h to remove the ethanol. An SPS apparatus (FCT system, model HP D5, Germany) was used to consolidate the powder mixtures. The following parameters were used during sintering for all of the samples: sintering temperature = 1400 °C, holding time = 10 min, pressure = 50 MPa, and heating rate = 100 °C/min. The sintered samples were ground using 60-grit SiC paper to remove the graphite protection sheet used during the sintering process, and further fine grinding was carried out on diamond grinding wheels (with grit sizes varying from 225 µm down to 10 µm) installed on an AutoMet 300 Buehler grinding machine. The samples were then polished with a diamond suspension down to a surface finish (Rₐ) of approximately 1 µm.

2.3. Characterization techniques

An X-ray diffractometer (Rigaku MiniFlex, Japan) with Cu (Kα) radiation as the source of X-rays with a wavelength (λ) of 0.15416 nm was used to identify the phases present in the sintered samples. A field emission scanning electron microscope (FESEM) (Lyra3, Tescan, Czech Republic) was used to analyse the microstructures of the sintered samples. A tribometer (UMT-3, Bruker, USA) was used to conduct wear tests under dry conditions at room temperature and relative humidity of 30 ± 5%. Sliding wear tests were conducted on the samples to study their tribological behaviour at a normal load of 40 N, wear track radius of 3 mm and linear sliding speed of 0.1 m/s for a sliding distance of 500 m (2.6,400 cycles) using a ball-on-disc configuration (under unidirectional rotation) against three different counterface materials (WC, Si₃N₄ and Al₂O₃). Three different balls were used as counterfaces with specific properties, as shown in Table 2. Before each test, the counterface ball was cleaned with acetone. After the wear tests, the wear tracks on the samples were analysed with the help of FESEM to study the wear morphology and types of wear mechanisms involved in each sample. To further investigate the wear mechanism, the wear tracks were also analysed using a 3D optical profilometer (GTK-A, Bruker, USA), which was specifically used to measure the wear track depth (Z) by

Fig. 1 – XRD patterns of sintered composites of nanosized alumina matrix reinforced with 10–30 wt. % of coated (Ni) and uncoated cBN particles. Refer to Table 1 for the composition of each of the samples S1-S7. The following PDF card numbers are used for matching: Al₂O₃ = 00-043-1484, cBN = 01-073-0887, and Ni = 01-077-9726.
recording 2D and 3D wear profiles. Thermogravimetric analysis (TGA) (Mettler Toledo STAR® System, USA) was performed on a tungsten carbide/cobalt insert at a temperature of 600 °C and heating rate of 10 °C/min to evaluate the reaction product(s) and relate the TGA product with the debris from the wear track after the wear test. Moreover, the Raman spectra of the reinforcing cBN particles inside the wear tracks were acquired at room temperature using a Raman microscope (DXR2™, Thermo Scientific®, USA) with a laser power of 2.5 mW and excitation wavelength of 532 nm to study any possible phase transformation of cBN resulting from the stresses generated during the sliding wear tests. An optical microscope (MT7000, Meiji, Japan) was used to examine the transfer film and scar mark on the counterface ball before and after cleaning the ball with acetone at the end of each test. The wear loss of the samples was calculated by applying a weight loss method in which a balance (AUW220D, Shimadzu, Japan) was used to measure the mass loss of the cleaned sample (with acetone) with a precision of 0.01 mg before and after the wear tests. The volume loss (V) was calculated by measuring the weight loss and density (as measured in our previous study [34]) of the tested sample. The wear rate (W) was calculated using Eq. (1) [35].

\[
W = \frac{V}{F \cdot S}
\]  

In Eq. (1), F represents the applied load and S is the sliding distance. By keeping in mind, the importance of repeatabil-
ity and reproducibility [36], three wear tests were conducted on each sample, and the average values of the wear rate and coefficient of friction (COF) are reported.

3. Results and discussion

3.1. Structure, morphology and densification of the composites

The X-ray diffraction (XRD) patterns of the composites developed using a nanosized alumina matrix reinforced with cBN (with and without nickel coating) are shown in Fig. 1. These patterns show no evidence of any cBN-to-hBN phase transformation. We proposed in our earlier work that the large specific surface areas of the nanosized alumina particle matrix have impeded the transfer of thermomechanical stress to the cBN particles and thus decreased the likelihood of a cBN-to-hBN phase transformation [34]. Similar observations have been reported in some of the published literature [30,37]. Furthermore, no signs of any chemical reaction between Al₂O₃ and cBN reinforcements could be inferred from the XRD results, and the absence of such reactions is beneficial for the development of well-bonded and densified alumina-based cBN composites. All the nano-alumina composite samples were determined using Archimedes’ method to be well densified, with densification of over 98% as calculated using the rule of mixture.

Fig. 2 shows FESEM micrographs of (a) pure alumina and alumina-based nanocomposites reinforced with (b–d) uncoated cBN and (e–g) cBN coated with Ni. These micrographs show that the cBN within the alumina matrices is well dispersed and that all the sample composites were well consolidated. A comparison of these micrographs (Fig. 2) indicated
that the Ni coating (in the case of coated cBN) covered the edges of the cBN particles and thus strengthened these composites by enhancing the bonding of the cBN (Ni) particles with the alumina matrix. Zhang et al. [33] employed a similar concept of using SiO$_2$-coated cBN particles to improve the bonding between the particles and the alumina matrix for various tool applications.

3.2. Tribological and mechanical properties

3.2.1. Tribological performance of pure alumina and alumina/(Ni) cBN composites

Initially, sliding wear tests were conducted on the pure alumina and composite samples to study their tribological behaviour at a normal load of 40 N, wear track radius of 3 mm and linear sliding speed of 0.1 m/s for a sliding distance of 500 m (26,400 cycles) using a tungsten carbide (WC) counterface ball. Fig. 3 shows (a) a typical coefficient of friction (COF) graph of pure alumina, (b) counterface ball image after cleaning with acetone, (c) 3D optical profile image and (d) 2D optical profile image after the wear test. The scar mark length on the counterface ball was 0.96 mm, and the wear track depth (Z) was 11 µm, as shown in Fig. 3(b) and (d), respectively.

The wear rate and average COF of pure alumina were compared with those of the developed composites for the wear tests performed at a normal load of 40 N, linear sliding speed of 0.1 m/s and a sliding distance of 500 m (Fig. 4). The wear resistance of all the composite samples was better than that of the pure alumina sample and increased with the increase in the percentage of cBN. Furthermore, the composites reinforced with Ni-coated cBN particles showed lower wear rates than did those reinforced with uncoated cBN particles. Conceivably, better bonding of cBN (Ni) with the alumina matrix improved the wear properties. Note that the hardness increased as the cBN percentage was increased (Fig. 5); detailed investigations of the hardness levels of the composites had been carried out in our previous study [26, 34].

Fig. 5 – Hardness and thermal conductivity values of the various developed composites. See Table 1 for the compositions of each of the samples S1–S7 [34].

Fig. 6 shows typical COF graphs and 3D optical profiles of the developed composites, as well as images of the counterface balls. All graphs, profiles, and images were obtained after the wear tests were performed under similar conditions against the WC counterface. The 3D optical profiles revealed an inverse relationship between wear volume loss and cBN percentage. The 30% cBN-Ni (S7) sample showed an 85% lower wear rate than that of the pure alumina sample (Fig. 4). This low wear rate was attributed to the excellent mechanical properties of S7, as evidenced by its relatively smooth and unworn wear tracks (see 3D optical profile image in Fig. 6). In contrast, counterface balls used against all the composites (Fig. 6) exhibited more wear than did pure alumina, which was expected due to the greater overall hardness of the composites. The counterface ball of S7 showed the most extensive scar, with a length of 2.28 mm, and showed severe wear and flattening, all attributed to the high hardness of sample S7.

On average, the COF values of the composites, as shown in Fig. 4, were lower than that of pure alumina. However, the COF values of the composites reinforced with Ni-coated cBN were higher than those of the composites reinforced with uncoated cBN. Interestingly, although the most extensive scar mark was observed for the counterface ball of S7 due to the superior properties of this sample, the average COF of this sample, at 0.3, was lower than that of pure alumina, which showed an average COF of 0.56. In a recent study by Yaman and Mandal [38], zirconia-toughened alumina exhibited an average COF of 0.3 against a WC counterface ball, while after the addition of cBN (17.5 vol.%) into the matrix, the COF increased to 0.53. This behaviour is ascribed to the third-body wear mechanism due to cBN particles being pulled out during sliding at a normal load of only 15 N, which is absent in the present study as a result of better interfacial bond strength. Moreover, in
our study, the low COF values of the composites (compared to that of pure alumina) were attributed to the formation of an adhesive layer of tungsten oxide (WO$_3$) on the wear tracks transferred from the counterface (WC) ball material. This layer is shown in the FESEM/BSE colour images in Fig. 7 along with the corresponding elemental maps obtained through energy dispersive spectroscopy (EDS). The transfer layer provides a lubricious property that helped to reduce the wear rate of the composites, as observed in previous studies [39,40]. This tribochemically formed tribolayer contains reaction products in the form of oxides such as WO$_3$ and CoO, resulting from the oxidation of WC and cobalt [38–40].

As reported in the literature, localized frictional heat generated during the sliding wear test can increase the flash temperature at the asperities, thus resulting in the oxidation reaction [38–40]. To confirm the type of reaction product formed on the wear track, TGA analysis (Fig. 8) of the WC/Co insert (WC/Co counterface/ball piece) was performed by subjecting the insert to an initial heating ramp of 5 °C/min from room temperature to a temperature of 600 °C followed by an isothermal heat treatment at 600 °C for 4 h. The TGA curve (Fig. 8a) shows that an increase in weight starts to occur very close to 500 °C and the temperature continues to increase to 600 °C due to the oxidation reaction (Eq. 2), thus implying the

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**Fig. 6** – Typical COF graphs of the developed composites (left) — along with 3D optical profiles (centre) and images of counterface balls after cleaning with acetone (right). The wear tests were performed at a normal load of 40 N and a linear sliding speed of 0.1 m/s for a sliding distance of 500 m against a WC counterface ball.
Fig. 7 – FESEM images of the wear tracks (after cleaning with acetone) of (a–c) pure alumina, 10% cBN and 30% cBN-Ni along with (d–f) corresponding BSE coloured images, (g–i) magnified images inside the wear tracks along with (j–l) corresponding BSE coloured images and (m–o) EDS elemental maps showing the distribution of W as a representative of the WO₃ tribofilm after the wear tests were performed at a normal load of 40 N and a linear sliding speed of 0.1 m/s for a sliding distance of 500 m against a WC counterface ball.

Note: The arrows show the transfer film (tribofilm), and the circular marks show the reinforcing cBN particles in the matrix.
transformation of WC into stable WO₃, which remained stable throughout the isothermal heat treatment. Furthermore, XRD analysis of the TGA product (yellowish orange), shown in Fig. 8b, confirms the formation of WO₃ as a major reaction product.

\[
WC + 2.5O_2 (Ar) \overset{\Delta 600^\circ C}{\rightarrow} WO_3 + CO_2
\]  

(2)

Analyses of the micrographs, images and maps presented in Fig. 7 show that the adherent transfer films (yellowish-orange colour as shown clearly in the BSE colour images of Fig. 7) remained intact on the wear tracks after the wear tests, suggesting that there was plastic deformation of the tribochemically formed layers along with some plastic deformation of the contact surfaces after a certain period of runtime, which ultimately helped in reducing the mechanical stresses at the contacts and simultaneously decreasing the COFs and wear rates of the samples [32]. However, in the case of pure alumina, this transfer film was very thin and relatively discontinuous, as indicated by its EDS map (Fig. 7m), and its counterface ball showed a very small scar mark and little flattening because the material from the pure alumina sample was more easily dislodged than was the material from the counterface ball (Fig. 3b) due to the low resistance to wear of the alumina. The reduced COFs and wear rates of the composites may also be due to self-lubrication provided by cBN particles yielding
relatively easy slides between the mating surfaces of the counterfaces and cBN particles, as observed in previous studies [41,42].

Our study showed that increasing the amount of cBN in the composite led to an increase in the resistance of the composite to wear, and these results were consistent with previously reported enhancements in the wear properties of ceramic-based matrices by the inclusion of cBN, specifically by the reinforcement of a micro-sized alumina matrix with 30 vol.% cBN [3] and of a WC matrix with 25 vol.% cBN [40]. In contrast, Yaman and Mandal [38] found a decrease in wear resistance of alumina-based composites upon the inclusion of 25 vol.% cBN particles. They attributed this decrease to the micro-sized alumina matrix, which favoured the pulling out of second-phase particles (cBN) and hence caused third-body wear. This difference was presumably attributed to the low densification of the micro-sized alumina matrix, the comparatively weak interactions of its grain boundaries and the bonding at the alumina/cBN interfaces, contrary to the excellent densification of the nanosized alumina matrix observed in this study. The wear results clearly confirmed that the improved wear properties resulted from the enhanced consolidation of the nanosized alumina matrix, as well as, from the inclusion of nickel-coated cBN particles as reinforcements.

As shown in Fig. 5, the thermal conductivity of the composite increased with the increasing amount of cBN (with and without Ni coating) in the matrix in experiments carried out at room temperature. This result was attributed to the transport phenomena of heat flow across an interface between two different materials, e.g., at the interface between the matrix and inclusions in the matrix. The thermal conductivity of a material depends on various factors, such as the presence of voids, the interfacial resistance, and the particle size of both the reinforcement and matrix, the latter being one of the important factors that can affect the temperature build-up at the tribocontacts during sliding wear under dry conditions [43,44]. High thermal conductivity plays a beneficial role in preventing a build-up in local temperature that would otherwise occur due to frictional heat generated during dry sliding or other cutting applications and is also helpful in reducing thermal shocks created during cycles of heating and cooling. However, poor densification at interfaces leads to low thermal conductivity. It does so by reducing the carrier (heat/phonon) mean free path, which occurs due to the propagation of phonon–electron...
interactions across such poorly densified interfaces, usually resulting in the diffused scattering of phonons and loss of energy in the matrix [26,34].

The presence of nickel-coated particles was observed to hinder the cBN-to-hBN transformation during sintering, thereby further improving the thermal conductivity because of the presence of nickel-coated cBN particles in the nano-alumina matrix. Additionally, among all of the samples tested, the sample with 30% cBN-Ni (S7) displayed the highest thermal conductivity (measured in our previous study [34]), as shown in Fig. 5, as well as the highest wear resistance, which is attributed to its improved densification and thermomechanical properties. The presence of nickel along the grain boundaries improved the interfacial bonding strength, which resulted in more efficient load transfer, thereby improving the mechanical properties. The hardness of the composite increased as the amount of cBN increased, and the hardness further improved as the amount of nickel-coated cBN increased (Fig. 5). These results confirmed that the use of a nanosized matrix results in higher hardness compared to the micro-sized matrix, with 30 wt.% cBN(Ni) showing the highest hardness of 29 GPa.

Fig. 9 shows the FESEM micrographs of (a) pure alumina, (b) alumina with 10% cBN and (c) alumina with 30% cBN-Ni after they were subjected to wear tests. All samples showed major wear features. The pure alumina subjected to the wear test showed severely dislodged material. However, after the wear tests, the nanocomposites with a low percentage of cBN (samples S2 and S5) showed micro-sized cracks and chipping. These cracks and marks were less severe for the composites with a high percentage of cBN. The sample with 30% cBN-Ni (S7) showed only micro-sized chipping from the matrix due to the generation of stress during sliding, along with slight abrasion marks with no removal of second-phase particles (cBN), attributed to the additional bonding strength provided by the Ni coating on the cBN particles.

Raman spectra (Fig. 10a) and an optical image (Fig. 10b) were acquired for reinforcing cBN particles within the wear track zone to investigate if these reinforcing particles underwent any possible phase transformation inside the wear track that may have resulted from the stress and heat generated during the sliding wear tests. The peaks corresponding to the hBN phase were absent in these spectra, suggesting that no stress (heat)-induced cBN-to-hBN phase transformation occurred.

### 3.2.2. Effect of counterface ball material on tribological performance

WC balls were used for the initial screening tests as described earlier. However, subsequent tests were conducted to evaluate the effect of two other counterface ball materials, Si₃N₄ and Al₂O₃, on the tribological performance of the two best performing composites, namely, those made with 30% cBN (S4) and 30% cBN-Ni (S7). The tribological parameters were the same as those used earlier to compare the results for all counterface balls. Fig. 11 shows the (a) wear rates and (b) average COFs of the alumina composites reinforced with 30% cBN (S4) and 30% cBN-Ni (S7) after these composites were subjected to wear tests performed in similar conditions when using the WC and Si₃N₄ counterface balls. However, the sliding was kept at 250 m for the tests with the Al₂O₃ counterface balls due to the severe damage and flattening of these balls and damage to their holders at sliding distances greater than 250 m (Fig. 11a). This complete flattening of the Al₂O₃ counterface ball extended to the edge of the holder. This damage may have been due to the relatively low compression strength, low thermal conductivity and low fracture toughness of the Al₂O₃ counterface (alumina balls) when compared to that of the WC and Si₃N₄ counterface balls, as shown in Table 2. The analysis of the results indicates a significant increase in wear rates and COFs when Si₃N₄ and Al₂O₃ balls were used compared to when WC balls were used, as shown in Fig. 11(a) and (b). Moreover, the composite made with 30% cBN-Ni (S7) showed a low wear rate with a small increase in COF compared to that
Fig. 12 – FESEM images of wear tracks (without removing the wear debris) for (a–c) the alumina/30% cBN-Ni composite (S7) along with (d–f) corresponding BSE coloured images, (g–i) EDS spectra of the inside of the wear tracks, (j–l) magnified images of the corresponding wear debris and (m–o) cleaned counterface ball images. The wear tests were performed at a normal load of 40 N and a linear sliding speed of 0.1 m/s for a sliding distance of 500 m using three different counterface balls, namely, WC (left), Si₃N₄ (centre) and Al₂O₃ (right).
made with 30% cBN (S4) in all the tribo-pairs due to the better mechanical properties of the former, as described earlier.

Fig. 12 shows a comparison of the FESEM images of wear tracks (without removing the wear debris) of the 30% cBN-Ni-reinforced alumina (S7) composite (a–c) along with the corresponding BSE colour images (d–f), EDS spectra inside the wear tracks (g–l), magnified images of the corresponding wear debris (j–l) and cleaned counterface ball images (m–o) after the wear tests using each of the three counterface ball materials. The wear track width, amount of wear debris (Fig. 12c) and scar mark length on the counterface ball (Fig. 12o) were all highest when using the Al2O3 counterface ball, indicative of severe wear of both the sample and counterface. A similar type of wear behaviour was observed when using the Si3N4 counterface, but the extent of the wear was much lower than when using the Al2O3 counterface. Hence, for the three counterface(s) tested, the WC counterface produced the least wear and lowest COF for the 30% cBN-Ni-reinforced alumina composite. This result may have been due to the formation of an adherent transfer layer on the sample as a result of possible oxidation of WC, as discussed earlier (where wear tracks were cleaned with acetone). This adherent layer of WC is also indicated in Fig. 12(a) and (d), distinguished by a colour difference compared to a less adherent transfer layer. The yellowish orange colour shown in the BSE colour images of Fig. 12d clearly indicates a highly adherent transfer layer. Moreover, the WC counterface displayed cylindrical and roll-shaped wear debris (WO3) (Fig. 12j), which may have provided effective lubrication, as discussed earlier, whereas the Al2O3 and Si3N4 counterparts showed non-cylindrical chunks of debris. Furthermore, in the case where the WC counterface was used, the energy dispersive X-ray (EDX) spectrum (Fig. 12g) acquired from the surface of sample S7 revealed the smaller amount of elemental aluminium than that measured in the case where the Si3N4 counterface was used (Fig. 12h). These EDS results suggested that an adherent transfer layer formed in the case of WC was responsible for the low wear rate and COF.

4. Conclusions

- Due to the uniform dispersion of hard cBN particles, all the composites showed lower wear rates and COFs than did pure alumina. The resistance to wear was increased with the increase in the amount of cBN reinforcement.
- Samples reinforced with Ni-coated cBN particles exhibited higher wear resistance, with slightly higher COF, than did the composites reinforced with uncoated cBN, primarily due to the better interfacial bonding provided by the nickel coating. The sample reinforced with 30% cBN-Ni showed approximately 85% higher wear resistance against the WC counterface ball than did the Si3N4 and Al2O3 counterface ball materials.
- The wear rates and COFs of samples tested against the WC counterface ball material were lower than those tested against the Si3N4 and Al2O3 counterface ball materials under the same tribological conditions. This behaviour is attributed to the formation of a WO3 adhesive transfer layer on the wear tracks, providing lubricious properties.
- The excellent mechanical and tribological performance of alumina/30% cBN-Ni composite can be beneficial for applications suspected of wear under harsh environments, including cutting tools, seal rings, wear pads, aircraft brakes and components of bearings.

Conflicts of interest

The authors declare no conflicts of interest.

Acknowledgements

The authors would like to acknowledge the support provided by the Deanship of Scientific Research (DSR) at King Fahd University of Petroleum and Minerals (KFUPM), Dhahran, Saudi Arabia, for funding this work through KFUPM-Internal Project No. IN131055.

References


*Note: The wear tests were performed at 250 m sliding distance only in the case of the Al2O3 ball, as discussed and instructed earlier.*


