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Tribological and electrical properties of ceramic matrix composites with carbon nanotubes

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Abstract

Tribological behaviur of carbon fibrous phases (nanofibers and nanotubes) containing composites with Si_3N_4, ZrO_2 and Al_2O_3 matrices was studied by pin-on-disk technique in conditions of dry sliding. Coefficients of friction and wear rates were measured, wear damage mechanisms were observed and identified. The resulting tribological behaviur was related to microstructure and mechanical properties of respective materials. Electrical conductivity was measured in wide range of frequencies by two-point method and effect of volume fraction and distribution of CNTs and CNFs on percolation threshold was evaluated. Both coefficient of friction and electrical resistivity decreased with increasing amount of carbon phases, in both cases the nanofibers were more efficient than the nanotubes. The wear resistance in most cases decreased but for Si_3N_4–CNT composite a certain optimum (~5 wt.% CNT) was found.

Keywords: B. Nanocomposites; C. Friction; C. Wear resistance; C. Electrical conductivity; Nanotubes

1. Introduction

In recent decades there has been a strong effort to develop better ceramics for structural applications. Among the most promising materials belong zirconia and silicon nitride based systems. Zirconia (ZrO_2) has potential to attain very high toughness due to transformation toughening [1–4]. It is considered as prime candidate for many cutting tool applications and as biomedical material. Silicon nitride (Si_3N_4) was developed in a search for fully dense, high strength and high toughness materials [5]. A prime driver for its development was to replace metals with ceramics in advanced turbine and reciprocating engines to give higher operating temperatures and efficiencies. Alumina (Al_2O_3), on the other hand, is one of the most cost effective and widely used materials in the family of engineering ceramics which can be found in a very wide range of applications.

However, the inherent brittleness is still the main limiting factor of these materials. This problem has been tried to overcome by creating appropriate composite materials [6]. In recent years, following discovery and suggestions of Iijima [7], novel composites with carbon nanotubes and fiber-like structures are being developed. The fibrous nature of the toughening elements could lead to higher fracture toughness. Moreover, unlike other non-metallic additives, also other functional (electrical) properties can be useful, thanks to carbon electrical conductivity.

In all mentioned applications the materials will have to face the challenge of friction and wear damage. Since wear is a complex problem, which cannot be easily predicted from basic mechanical properties [8] (hardness, fracture toughness) tribological studies of the developed materials are indispensable.

The aim of this work was to study tribological properties of the CNF/CNT containing CMCs based on ZrO_2, Al_2O_3 and Si_3N_4 and to determine the influence of content of the carbon phases on coefficient of friction and wear. Further purpose was to measure the electrical properties, namely the electrical conductivity of these materials with respect to the composition and microstructure, and to find the percolation thresholds for particular cases.

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2. Experimental details

2.1. Experimental materials

The procedure of preparation of the experimental materials and their microstructures were investigated and described in detail elsewhere [9–12].

To prepare the zirconia based materials [9,10] 3 mol.% Y-TZP powder (from Tosoh, Japan) was used. Carbon nanofibers were grade HTF150FF (Electrovac, Austria). According to producer these CNFs have a diameter of 80–150 nm, specific surface area in a range of 20–100 $m^2 g^{-1}$, Young's modulus of ~500 GPa, tensile strength of ~7 GPa and electrical resistivity of 10^{-3}–10^{-4} Ω cm.

The CNFs were dispersed by milling in Millipore water with dodecylbenzenesulfonic acid solution (DBSA) as a dispersing agent. The mixture was ultrasonically dispersed for 10 min using a probe. Separately Y-TZP powder was also ball milled in Millipore water. The CNFs solution was added to the ceramic slip, and the mixture was then given a further ultrasonic treatment. This slip was continuously stirred and spray dried in a small laboratory spray dryer (model 190, Büchi, Germany). Composite powders were prepared with 2.0 and 3.3 vol.% CNF content.

The resultant powder granulates were die pressed into 20 mm diameter discs for hot pressing. These samples were hot pressed in an argon atmosphere at a dwell temperature 1300 °C for 30 min at a pressure of 41 MPa. For comparison monolithic ZrO_2 was prepared under similar conditions, e.g. hot pressed at 1300 °C for 30 min at a pressure of 41 MPa. For SPS, discs of 20 and 30 mm diameter were prepared. The SPS samples were sintered at different dwell temperatures. The samples with 2.0 vol.% CNFs were sintered at 1400 °C and the samples with 3.3 vol.% CNFs were sintered at 1500 °C in both cases for with a 5 min dwell time and at 60 MPa.

The Si_3N_4 based composites were prepared using starting powder mixtures of the investigated materials as follows [11]: 90 wt.% Si_3N_4, 4 wt.% Al_2O_3, and 6 wt.% Y_2O_3. In addition the batches of multi-walled carbon nanotubes were added. The powder mixtures were milled in distilled water in an HDDM attritor (Union Process) at 4000 rpm for 5 h. Zirconia agitator delta discs and zirconia grinding media with 1 mm diameter were used. Each batch contained zirconia as contamination from media and discs. Samples were compacted by dry pressing at 220 MPa. The materials were sintered at 1700 °C, 20 MPa, 3 h in high purity nitrogen by a two-step sinter-HIP method using BN embedding powder. The final compositions contained 0, 1, 3, 5 and 10 wt.% of multiwall CNTs.

The experimental materials based on alumina were prepared by spark plasma sintering at the Queen Mary, University of London, UK [12]. Multi-wall CNTs (NC-7000 by Nanocyl Inc., Belgium: average outer diameter 9.5 nm; lengths of up to 1.5 μm; and density 1.7 g cm^{-3}) were dispersed in dimethylformadie, DMF using high power sonication for 2 h and then hand-mixed with alumina nanopowder (Sigma–Aldrich, UK: gamma phase; particle size <50 nm; surface area 35–43 $m^2 g^{-1}$; melting point 2040 °C; and density 3.97 g cm^{-3}) for 2 min.

Table 1
Experimental materials, their compositions and processing routes.

Material	Matrix	Toughening phase	Fraction (wt.%)	Process route
ZrO_2	ZrO_2	CNF	0	HP
HP ZrO_2–1%CNF		(Electrovac)	1	HP
HP ZrO_2–2%CNF			2	HP
SPS ZrO_2–1%CNF			1	SPS
SPS ZrO_2–1%CNF			2	SPS
Si_3N_4	90% Si_3N_4	CNT	0	HIP
Si_3N_4–1%CNT	+	(KFKI)	1	HIP
Si_3N_4–3%CNT	4% Al_2O_3		3	HIP
Si_3N_4–5%CNT	+		5	HIP
Si_3N_4–10%CNT	6% Y_2O_3		10	HIP
Al_2O_3	Al_2O_3	CNT	0	SPS
Al_2O_3–2%CNT		(Nanocyl)	2	SPS
Al_2O_3–3.5%CNT			3.5	SPS
Al_2O_3–5%CNT			5	SPS
Al_2O_3–10%CNT			10	SPS
Al_2O_3–2%CB		Carbon black	2	SPS
Al_2O_3–5%CB			5	SPS

The liquid mixture was rotation ball milled for 8 h. It was then dried at 75 °C for 12 h on a heating plate in air, and then for 3 days in a vacuum oven at 100 °C for 3 days. Alumina and nanocomposite pellets (diameter 20 mm and thickness 2 mm) were prepared by SPS in a HPD 25/1 (FCT Systeme, Germany) furnace at 1800 °C. A pressure of 100 MPa was applied, heating rate was 300 °C min^{-1}, the sintering period was 3 min. As a reference, also carbon black containing Al_2O_3 based nanocomposites were prepared in the same way.

The overview of all experimental materials is summarized in Table 1.

2.2. Experimental methods

Wear behaviur of the experimental materials was studied in dry sliding for self-mated pairs. The surfaces were carefully prepared by polishing down to surface roughness below 1 μm. The wear testing was carried out on the tribometer HTT by CSM Instruments in air at the room temperature using the pin-on-disk technique, where the tribological partner for each material was a highly polished ball with 6 mm diameter made out of ceramics corresponding to the ceramic matrix of the tested material. The applied loads varied between 1 and 5 N, the sliding speeds from 2.5 to 15 cm/s and the sliding distance was 100 m.

The tangential forces during the test were measured and friction coefficients calculated. The worn surfaces were subsequently observed and the wear regimes, damage type and micromechanisms were identified. The material losses due to wear were measured by a contact profilometer and then specific wear rates (W) were calculated in terms of the volume loss (V) per distance (L) and applied load (F_p) according to the standard ISO 20808 [13]:

$$W = \frac{V}{L \cdot F_p} \quad [\mathrm{mm^3/(m\ N)}]$$

The microstructure and fracture surfaces of the investigated materials were studied using scanning electron microscopy.

The electrical conductivity measurements were carried out at ambient temperature using a two-point probe setup on a high precision impedance analyzer Agilent 4294A at frequencies from 40 Hz to 40 kHz.

3. Results and discussion

3.1. Microstructures

Microstructure studies of the experimental materials were described in detail in our previous works [9–12,14–18].

The study of nanofibers used in these materials found that they have microstructure which differs from typical multiwall carbon nanotubes, used in other two material systems [9,16]. The nanofibers had average diameter average about 120 nm, and length from several micrometers to several tens of micrometers. Two types of fibers were identified; typical hollow cylindrical ones and so-called bamboo shaped fibers. The hollow cylindrical fibers are usually defect free and consist of a distinct sandwich of graphite layers parallel to the fiber axes, so they are basically MWCNTs with high number of carbon layers. The bamboo shaped fibers (which were less than 5% by volume) often contain defects at the nano-level, and their walls are built from domains with different orientation of graphite layers.

The microstructure of the zirconia-based materials consisted of very small, submicron/nanometer sized tetragonal grains with randomly placed nanometer sized pores, Fig. 1a. The nanofibers containing composites have matrix finer than the monolithic ZrO_2 with well distributed carbon nanofibers with diameter several tens of nanometers and very high aspect ratios, Fig. 1b. Also in contrast to fully dense monolithic zirconia, the composites contained some residual porosity associated with the nanofibers clustering. The smaller matrix grain size in the composite suggests that the CNFs hinder the grain growth in the composite during the sintering. The slightly smaller grains of matrix in the composites sintered by SPS are explained by a shorter sintering time compared to the HP regime. In monolithic zirconia the grain boundaries were clean without any intergranular phases. In the composites, besides the CNFs TEM analysis found in intergranular spaces also amorphous carbon and free graphite sheets coming from CNFs disintegrated during processing [9].

The CNTs in the Si_3N_4 composites were located mainly in the intergranular places and they were well attached to the silicon nitride grains [11]. Their rather strong tendency to adhere and link together was observed even after long sonication time, Fig. 2. In all composites the matrix consisted of β-Si_3N_4 grains.

In the case of SPS-ed alumina based materials the studies [12,17] showed that the microstructure of the monolithic Al_2O_3 (Fig. 3a) consisted of relatively large, micron sized grains with randomly distributed submicrometric sized pores, located at the grain boundaries. The composites had much finer matrix with relatively well distributed carbon nanotubes (or carbon black particles) with diameter of several nanometers and with very high aspect ratio. The smaller matrix grain size in the composite suggests that both the carbon black and the CNTs hinder the grain growth during the sintering.

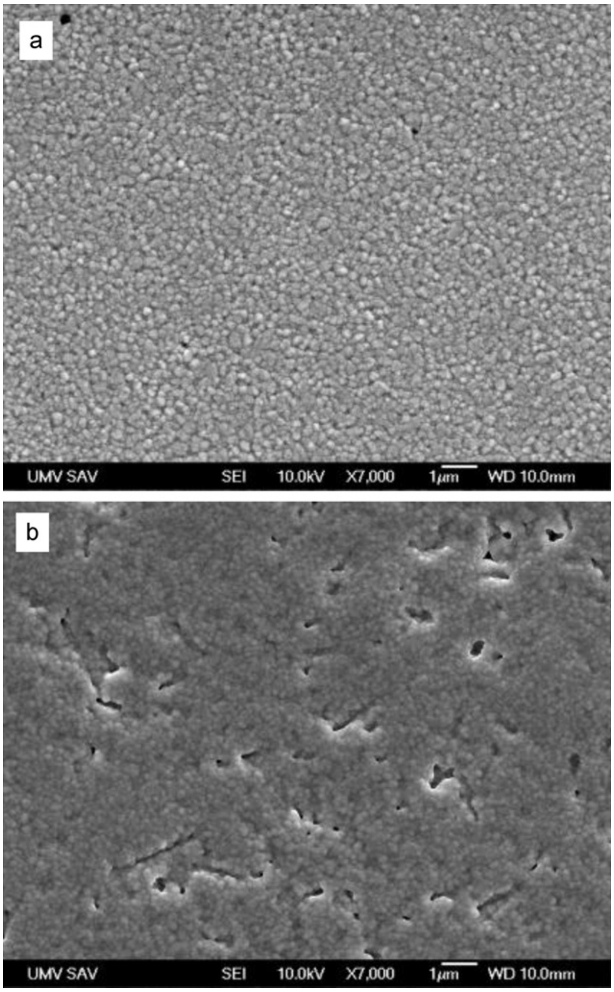

Fig. 1. SEM micrographs of the zirconia based experimental materials: microstructure of the HP monolithic zirconia (a) and HP ZrO_2–CNFs composite at low magnification, thermally etched with the locations of the burned out CNFs during the thermal etching (b).

The CNTs in the Al_2O_3–CNT composite were located mainly in the intergranular places and were well attached to the alumina grains, Fig. 3b.

3.2. Tribological properties

Fig. 4 shows examples of development of the friction coefficient over the test running distance. In all cases was friction after short initial stage (in order of meters) rather stable and reproducible. For monolithic ZrO_2 (Fig. 4a) the values of COF were slightly lower than similar results found in the literature. The sliding wear behaviur of ZrO_2 using the ball on disk method and alumina as a friction partner was also investigated in [19]. The applied loads ranged from 19.8 N to 98 N. There, the coefficient of friction varied between 0.6 and 0.7 depending on the sliding speed, and appeared to be independent on the applied load. Higher COF can be explained

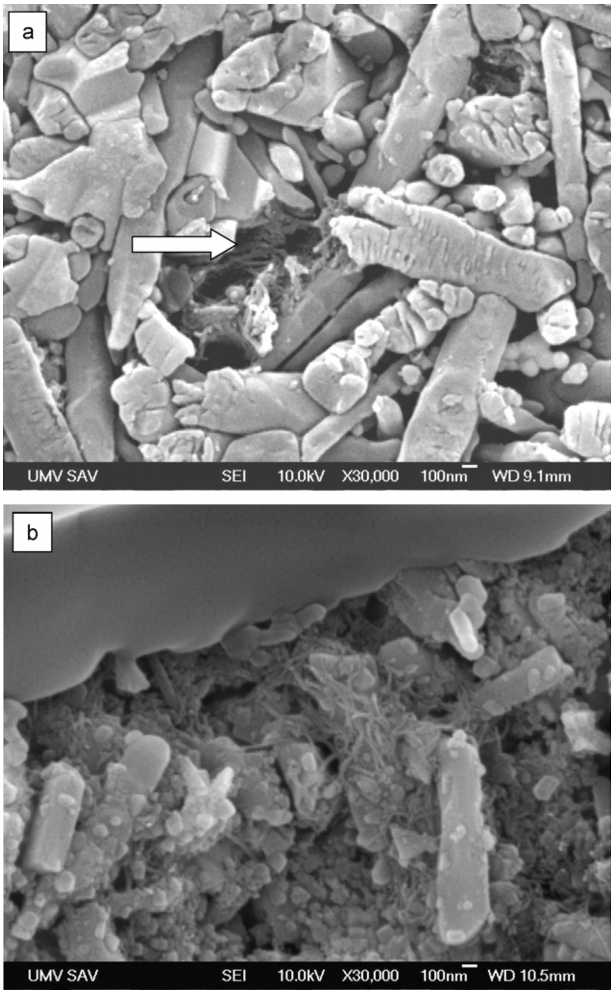

Fig. 2. Microstructures of silicon nitride based materials: small islands of nanotubes (arrow) in Si_3N_4–1% CNT (a) and dispersed nanotubes in Si_3N_4–5% CNTs (b).

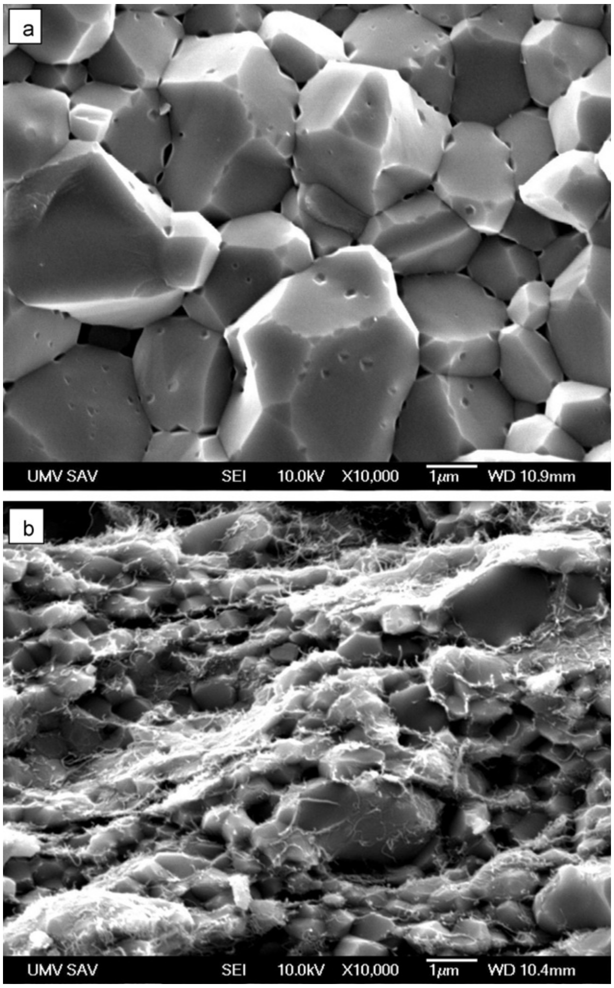

Fig. 3. Fracture surfaces of monolithic Al_2O_3 (a) and Al_2O_3–3.5 wt.% CNT nanocomposite (b).

by the damage mechanisms – the alumina grains detached from the alumina surface and caused abrasion of zirconia. For lower applied load, however, the main wear mechanism in the zirconia is typically self-polishing by low intensity abrasion.

Self-mated wear of monolithic silicon nitride was studied in various situations and can be summarized both in terms of the friction coefficient (Figs. 4b and 5, values 0.7–0.8) and wear rates (Fig. 6, values 1–2 × 10^{-5} $mm^3/(N\ m)$).

Fig. 5 summarizes the average values of friction for all materials at 5 N load, however our studies [15,17,18] yielded similar results at all used load levels. The Si_3N_4 based materials exhibited the highest friction, particularly for low CNT contents. Here, the COF started to decrease only when the CNT content reached ~5 wt.%. In Si_3N_4–10%CNT the COF values dropped to ~0.2 which was comparable to the values for ZrO_2–CNF. Similar values were found for self mated friction of alumina. Here, only monolith and 10% CNT containing composite are given, but the tendency is very similar to that of silicon nitride materials and more than 60% reduction of friction coefficient was found.

Fig. 6 shows the wear rates of various materials at different loads. Generally, the presence of CNT/CNF tends to decrease the wear resistance due to less than optimal microstructure. However, for the silicon nitride based composites there was found a sort of an optimum for fractions of around 5% CNT, where the wear resistance improved to the levels typical for the much harder monolithic Si_3N_4, probably thanks to the significantly reduced friction. Then, for Si_3N_4–10%CNT the wear reached values similar to those for Si_3N_4–1%CNT. In the case of alumina, however, such beneficial effect was not observed. The wear rate increased considerably, as if continuing a monotonous trend.

Fig. 7 illustrates typical damage observed in the wear tracks. The wear testing of the monolithic materials of both types of materials left faint, quite smooth wear tracks with polished look, as it is shown in Fig. 7a and c for sliding at 5 N load. This is consistent with the low wear rates of materials with high quality microstructure.

Fig. 7b and d illustrates the wear tracks of the composite materials and present the micromechanisms of the wear surface damage. Microscopic observations of the worn surface of the ZrO_2–CNF composite showed pulled out CNFs (Fig. 7b [14]) and suggested the presence of a smeared layer of graphite and crushed CNFs, the so-called transferred film, which

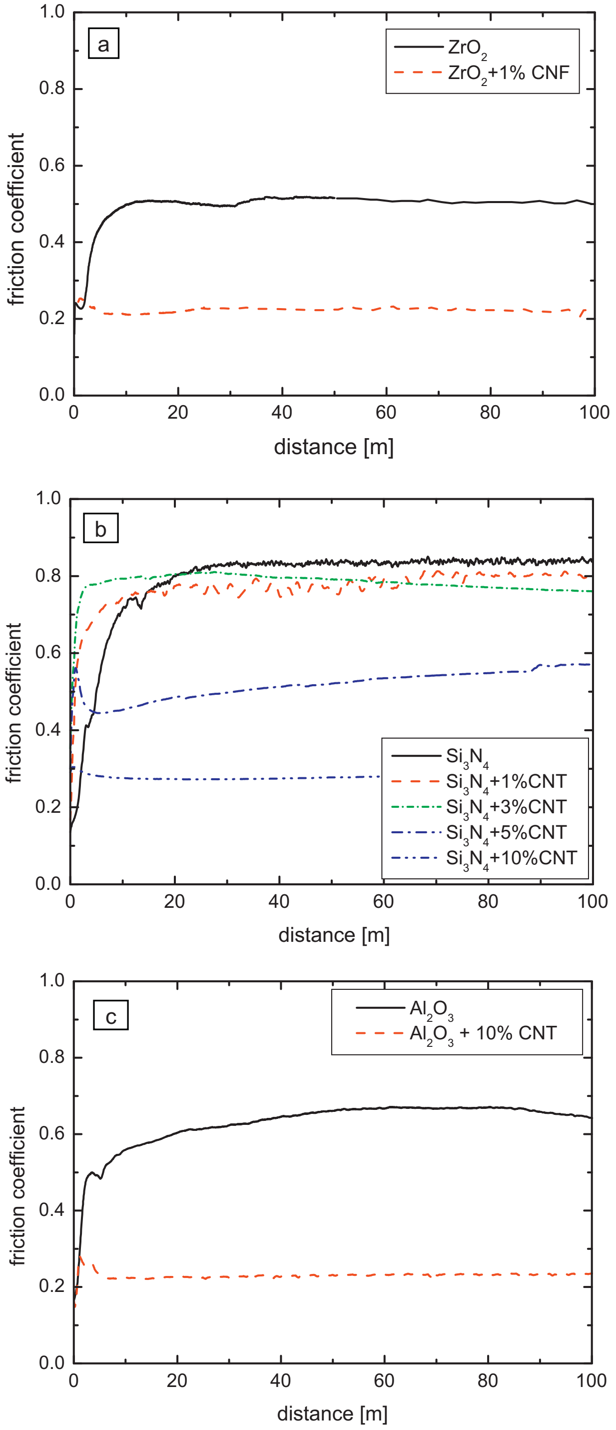

Fig. 4. Friction coefficient during the testing: (a) ZrO_2 based materials tested at 1 N load, (b) Si_3N_4 based materials at 1.5 N load, and (c) Al_2O_3 based materials, 5 N load.

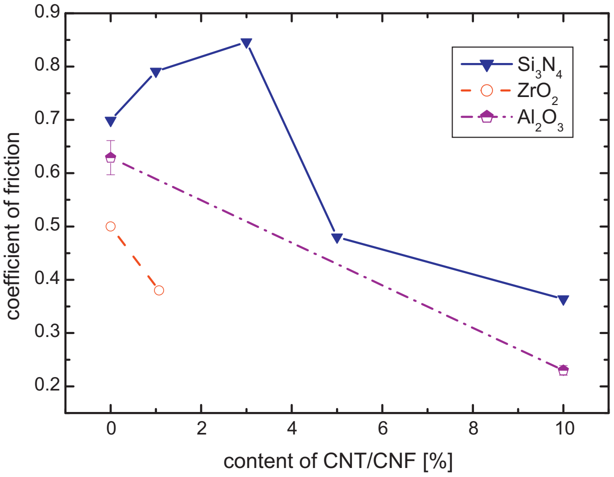

Fig. 5. Coefficient of friction as function of amount of carbon phases in the CMC materials, 5 N load.

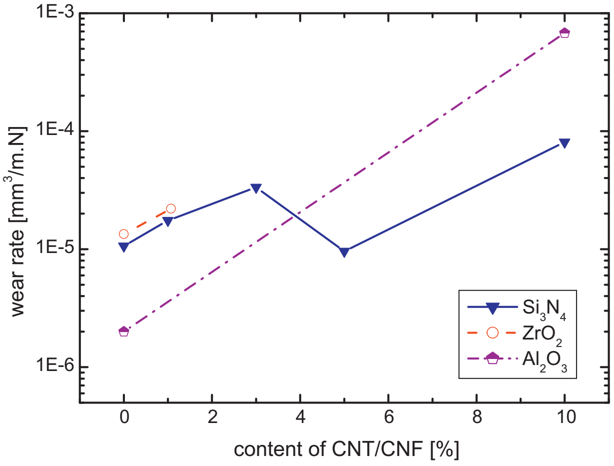

Fig. 6. Dependence of wear resistance of the experimental materials on their carbon content tested at 5 N load.

significantly lowers the friction coefficient, even though relatively low amount of carbon (1.07 wt.%) was incorporated into the microstructure. As Fig. 7d as well as the values of COF and wear rates suggest, the CNTs in silicon nitride based composites were much less effective in creating the transferred film. It may be due to their greater mechanical stability and better distribution throughout the microstructure. This could mean that their pull-out and also their destruction in the contact zone are more difficult.

In all cases by incorporating appropriate amount of carbon filaments the coefficient of friction between 0.2 and 0.3 was achieved. A similar lubricating effect by carbon phases was reported for MWCNTs-containing Al_2O_3 composites by [20,21] and for SWCNT solids by [22]. In [20] the authors reported a 40% decrease in friction coefficient in 12 wt.% MWCNT–Al_2O_3 composite down to the value of 0.3. Yamamoto with co-workers, for their composites reached the minimum of the friction coefficient (with the value of 0.3) at 4 mass% of MWCNT content. According to An et al. the rolling motion of CNTs at the interface between the specimen and the ball can probably lower the friction coefficient as well. This effect in the case of ZrO_2–CNF composite was not proven. Our results for silicon nitride based composites are in a very good agreement with the results reported for SWCNT solids sliding against Si_3N_4 where steady-state friction coefficients of 0.22–0.24 were found.

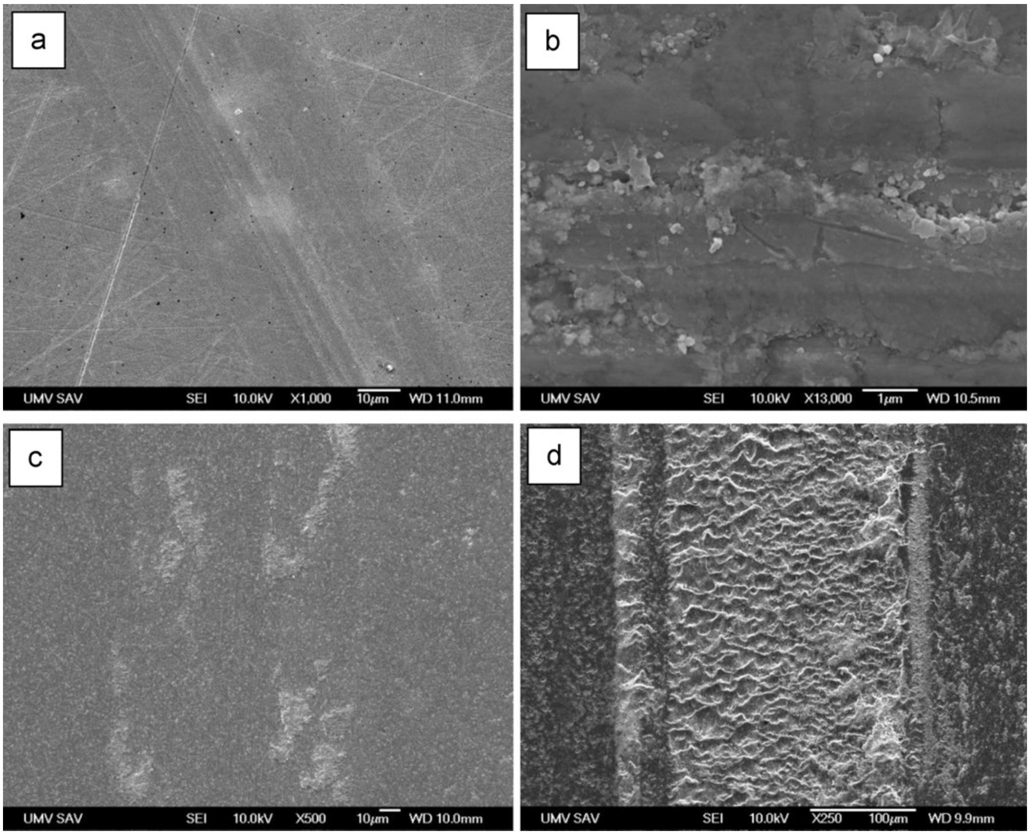

Fig. 7. Wear damage at normal load 5 N: (a) smooth wear tracks in monolithic ZrO_2, (b) detail of the wear track in ZrO_2–CNF composite – pull-out of CNFs and smeared transferred film, (c) monolithic Si_3N_4 – smooth track with polished look, and (d) Si_3N_4–3%CNT – surface abrasion.

Alumina–CNT composites with CNT contents from 0 to 12 wt.% were fabricated by two different processing techniques by Lim et al. [23] in order to compare the effect of fabrication techniques on the tribological properties of CNT added alumina composites. Superior tribological properties were achieved by introduction of tape casting, followed by lamination and hot pressing. Wear loss decreased significantly and friction coefficients were maintained to be about the same by the addition of the CNTs up to 12 wt.%. Agglomeration of CNTs, frequently observed with hot-pressed specimens, was significantly reduced and relatively uniform distribution of CNTs was obtained. The effective dispersion of CNTs contributed to the densification of composites and to superior mechanical and tribological properties of CNT added alumina composites.

3.3. Electrical properties

The conductivity of an individual CNT can be metallic or semiconducting, depending on the chirality of the CNT with axial component extremely high, 2×10^7 S/m [24] comparable to that of silver, cooper or gold. Larger CNFs have properties similar to graphite. As it was shown in the section on microstructure, the CNTs and CNFs can be distributed along matrix grain boundaries and/or between matrix grains. When interconnected they can form continuous networks. Such networks in CNT/CNF nanocomposites can be electrically conductive thanks to various mechanisms that involve fluctuation-assisted tunnelling or variable range hopping between the individual nanofibers.

Inam et al. [12], collected and summarized a number of studies of electrical properties of various CMCs prepared by hot-pressing and SPS. It seems that about 5–10 wt.% of carbon filament phases is necessary to ensure the percolation of electrical charges, and conductivities in orders of tens or hundreds S/m were found. Generally speaking, the carbon black, which was used for reference reasons, is less efficient,

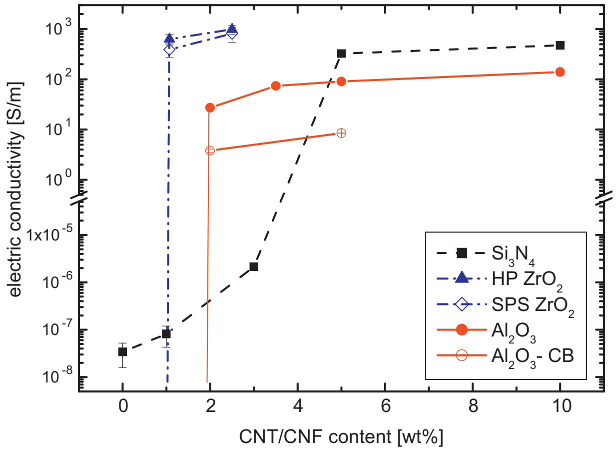

Fig. 8. Electrical conductivity vs. carbon phases content: illustration of percolation threshold and increase of conductivity for different CMC–CNT/CNF composites.

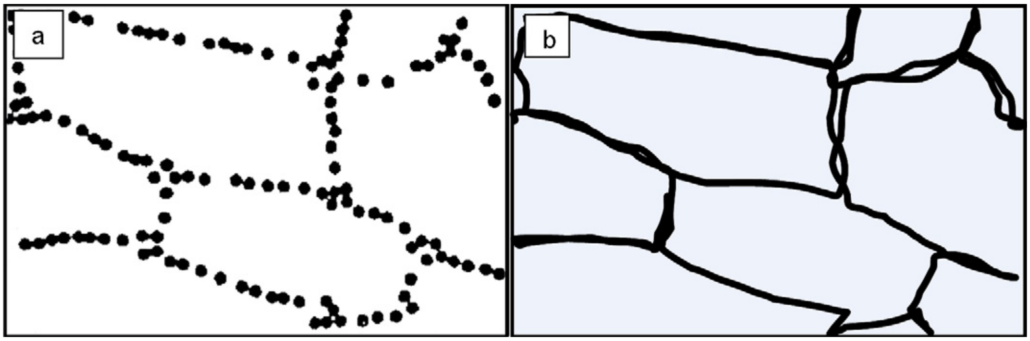

Fig. 9. Scheme of the microstructure of CMC with carbon black (a) and CNTs (b).

while larger CNFs or higher amounts of SWCNT + MWCNT form more conductive networks (588 S/m for HP alumina–5%CNF, and 853 S/m for magnesium aluminate–12.2%CNT, respectively).

Similar results were found in our measurements and the results are summarized in Fig. 8. Here, for the materials with CNTs, the percolation threshold was between 3 and 5 wt.% for Si_3N_4 and between 2 and 5 wt.% for alumina. The former material showed increase in conductivity of about 10 orders of magnitude. For the latter composite the electrical conductivity increased from practically zero to the maximum value of 140 S/m for 10 wt.% of CNTs. Analogous materials prepared in the same way but with admixture of carbon black yielded electrical conductivity about an order of magnitude lower – e.g. for 5 wt.% of additive it was 90.5 ± 4.8 S/m for CNTs and 8.5 ± 0.1 S/m for carbon black. This means that the size and shape of the mixed carbon black additives resulted in a limited graphite particle connection in the matrix, which was much less efficient in securing the percolation as it is illustrated schematically in Fig. 9.

Carbon nanofibers were significantly more efficient, as shown in the case of ZrO_2–CNF composites (Fig. 8), where amounts as low as 1–3 wt.% of carbon nanofibers were sufficient to increase the electrical conductivity from $\sim 10^{-12}$ S/m to 983 ± 200 S/m (hot pressed) or 813 ± 273 S/m (SPS-ed). Furthermore, this result suggests that for better conductivity the coarser matrix formed by hot-pressing is more advantageous than the finer one prepared by spark plasma sintering.

In summary it can be concluded that thanks to their fibrous nature and intergranular distribution, very little amount of carbon nanofibers (only about 1–5 wt.%, depending on the basic material microstructure) is sufficient for the percolation effect to take place. In this way an increase in electrical conductivity by more than 13 orders of magnitude can be achieved.

4. Conclusions

Three carbon nanofibers and nanotubes containing types of ceramic matrix composites were studied-based on ZrO_2, Si_3N_4, and Al_2O_3. They contained various amounts of CNT/CNF, from 0 up to 10 wt.%.

Microstructure studies showed that the addition of CNFs/CNTs makes densification more difficult. As regards the density of the final composite SPS seems to be more effective than hot pressing. The grain growth was suppressed by the CNFs/CNTs, which resulted in a very fine nanocrystalline ceramic matrix. The problem remains with clusters of CNFs/CNTs, which together with related residual porosity were very persistent in the composites as a result of the difficulty of their dispersing.

The tribological studies showed clear benefits of the presence of carbon fibers. By a small amount of CNFs (as little as 1 wt.%) the coefficient of friction of ZrO_2 can be lowered from 0.45 to 0.2 by formation of carbon film which acts as a solid lubricant. The CNTs were slightly less effective and higher amount was necessary to achieve similar results for both Si_3N_4 and Al_2O_3. The wear resistance generally decreased, which can be correlated to lower hardness and fracture toughness, but in several instances a sort of optimum was found (e.g. Si_3N_4–5 wt.% CNT) where the wear rate was similar to that of monolithic material but with much lower coefficient of friction.

The electrical conductivity can be increased very significantly and low amounts of CNFs or CNTs are sufficient to change an insulator to a functionalized conductive ceramics. Here again the larger CNFs appear to be more effective as the percolation occurred at ~1% CNF in ZrO_2, whereas between 3% and 5% CNT in Si_3N_4 and 2–5% CNT in Al_2O_3 were necessary.

Acknowledgments

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