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Anne Kasperski , Dalya Alkattan , Viviane Turq , Claude Estournes , Christophe Laurent , [Alicia Weibel](#) *

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Article

Friction and Wear Behaviour of Double-Walled Carbon Nanotube-Yttria-Stabilized ZrO₂ Nanonanocomposites Prepared by Spark Plasma Sintering

A. Kasperski, D. Alkattan, V. Turq, C. Estournès, Ch. Laurent and A. Weibel *

CIRIMAT, Université Toulouse 3 Paul Sabatier, CNRS-INP-UT3, Université de Toulouse, 118 route de Narbonne, 31062 Toulouse cedex 9, France

* Correspondence: alica.weibel@univ-tlse3.fr; Tel.: +33 0561556175

This paper is dedicated to the memory of our colleague and friend Pr. Alain Peigney

Abstract: Double-walled carbon nanotube-yttria-stabilized ZrO₂ nanocomposites are prepared by a mixing route followed by Spark Plasma Sintering. Incorporation of carbon at contents higher than 2 wt.% results in significantly lower friction coefficients, both against alumina and steel balls. Double-walled carbon nanotubes improve wear resistance and reduce friction without severe damage, contrary to multi-walled carbon nanotubes.

Keywords: Spark plasma sintering; Carbon nanotubes; Zirconia; Friction; Wear

Introduction

The interest for all-solid self-lubricating materials, which eliminate the necessity of liquid lubricants, is well recognized. Carbon-containing nanocomposites, especially carbon nanotubes (CNTs), are particularly noteworthy for their tribological applications [1,2]. Reports on the tribological properties of CNT-ceramic nanocomposites or coatings, in particular for Al₂O₃ matrix [3–14], are abundant. Key points for achieving higher microhardness, lower friction coefficients and lower wear include a high carbon content, the homogeneity of the CNT dispersion, a good interfacial bonding and a high relative density. In spite of many potential applications, notably in the field of biomaterials, the friction behavior of CNT-zirconia nanocomposites is scarcely addressed [15–17] compared to the mechanical properties [18]. These studies are focused on yttria-stabilized zirconia in which either single-wall CNTs (SWCNTs) [15] or multi-walled CNTs (MWCNTs) [16,17] have been dispersed. An alternative way to the use of SWCNTs or MWCNTs for the preparation of self-lubricating CNT-ceramic nanocomposites is that of double-walled CNTs (DWCNTs), which are a unique class of CNTs [19,20], possibly more interesting for tribological applications. It has been notably shown that the lubricating mechanisms depend on the number of walls and diameter of the CNTs: MWCNTs are cut and exfoliated, which leads to the formation of a lubricating film in the contact containing carbon debris whereas, DWCNTs have a better resistance to contact pressures and are elastically deformed [21]. Electrical and mechanical properties of spark plasma sintered DWCNT-ZrO₂ nanocomposites have already been published [22], the aim of the present work is to investigate their tribological properties which, to the best of our knowledge, have not been reported.

Materials and Methods

Powder Preparation

A commercial nanometric (grain size slightly lower than 100 nm) 3 mol.% yttria-stabilized zirconia powder (TZ-3Y, Tosoh, Japan) was used for the study. The proportions of tetragonal and monoclinic ZrO₂ phases determined by X-ray diffraction (XRD) are 77 and 23 vol.%, respectively. DWCNTs were synthesized by a CCVD route [23]. The catalytic material designated as

$\text{Mg}_{0.99}(\text{Co}_{0.75}\text{Mo}_{0.25})_{0.01}\text{O}$ was submitted to a catalytic chemical vapor deposition (CCVD) treatment (H_2 - CH_4 , 18 mol.% CH_4 , heating and cooling rates $5^\circ\text{C}\cdot\text{min}^{-1}$, maximum temperature 1000°C , no dwell), producing a CNT-Co/Mo₂C-MgO nanocomposite powder. The powder was immersed in a 37% HCl aqueous solution in order to dissolve MgO along with the majority of cobalt and molybdenum species, without damaging the CNTs [24]. The resulting suspension was filtered, washed with deionized water until neutrality, and kept wet (without any drying step) to facilitate further dispersion. The CNTs in the sample are mostly DWCNTs (80%), SWCNTs (15%) and CNTs with three walls (5%). The outer diameter is in the range 1-3 nm and the inner diameter in the range 0.5-2.5 nm [24]. The wet as-prepared DWCNTs were acid-functionalized using a mixture of nitric, sulphuric and hydrochloric acidic solutions at room temperature [25]. The mixture was neutralized with ammonia and filtered while keeping the CNTs wet. Five different DWCNT-ZrO₂ nanocomposite powders were prepared using the following route. The appropriate amount of acid-treated CNTs was dispersed in deionized water with a sonotrode (Vibra Cell 75042, 20 kHz, 500 W) for 15 min. The so-obtained CNT suspension was poured into a suspension of ZrO₂ in water (pH = 12), which was prepared previously (15 min tip sonication and 1 h mechanical stirring). The mixture was then tip-sonicated for 30 min. The vessel containing the DWCNT-ZrO₂ suspension was immersed in liquid N₂ until freezing and freeze-dried (Christ alpha 2-4 LD, Bioblock Scientific) at -84°C for 48 h in a primary vacuum (12 Pa). The carbon content (Cn) in the so-obtained DWCNT-ZrO₂ composite powders was measured by flash combustion (Perkin Elmer 2400 Series II) and is equal to 0.5, 1.2, 1.7, 4.5 and 6.3 wt.%.

Spark Plasma Sintering

The ZrO₂ and DWCNT-ZrO₂ powders were densified by spark plasma sintering (SPS, Dr Sinter 2080, SPS Syntex Inc., Japan). A graphite die with a 20 mm inner diameter was loaded in the following order from bottom to top: a graphite punch, a sheet of graphitic paper, an alumina powder bed approximately 1.2 mm thick (in order to block the current and ensure a uniform heating in specimens with varying electrical conductivities), another sheet of graphitic paper, the powder sample, and then the same materials in reverse order. Graphitic paper was also placed along the sides of the die for easy removal. SPS was conducted in an argon atmosphere using a conventional pulse pattern of 12-2 (12 current pulses followed by 2 periods of no current). The heating rate was $250^\circ\text{C}/\text{min}$ from room temperature to 600°C , with a 3 min hold at 600°C to stabilize the temperature reading. A heating rate of $100^\circ\text{C}/\text{min}$ was then applied from 600°C to the target dwell temperature, either 1200 or 1350°C depending on the carbon content (Table 1), with a 10 min dwell period. A uniaxial load (equivalent to 100 MPa on the pellet) was gradually applied during the hold at 600°C and maintained throughout the remaining heating and dwell period, then released in the final minute of the dwell. The cooling rate was set at $60^\circ\text{C}/\text{min}$. The sintered specimens were formed into pellets 20 mm in diameter and approximately 2 mm thick, which were then polished to a $1\ \mu\text{m}$ finish using diamond slurries. These sintered specimens will be referred to as ZrO₂, C0.5, C1, C2, C4.5 and C6 hereafter.

Characterization

Raman spectroscopy (Jobin-Yvon LabRAM HR800, 632.82 nm laser excitation) was used to characterize the raw DWCNTs and the nanocomposite powders, averaging at least three spectra for each specimen. X-ray diffraction (XRD, Bruker D4 Endeavor, Cu K α radiation) was performed on sintered specimens. The density of the pellets was measured using the Archimedes' method after removing the graphitic surface contamination by polishing with 600 grade SiC paper. Relative densities were calculated based on $6.05\ \text{g}/\text{cm}^3$ for tetragonal zirconia and $1.80\ \text{g}/\text{cm}^3$ for DWCNTs, with a relative uncertainty estimated at 1%. The fracture surfaces of the pellets, coated with a 1 nm thick platinum layer, were examined using field-emission-gun scanning electron microscopy (FESEM, JEOL JSM 6700F). For each sample, the linear intercept method [26] was used to measure a hundred ZrO₂ grains. Indentation tests (3 N applied for 10 seconds in air at room temperature) were conducted on the polished surfaces of the specimens using a Vickers indenter (Shimadzu HVM 2000). The calculated microhardness values (HV) are the average of ten measurements. Friction tests were conducted using a pin-on-disc reciprocating flat geometry (CSM Tribometer) in ambient air (30-60%

relative humidity, room temperature). 6 mm diameter alumina and 100C6 steel balls were used against the flat surfaces of ZrO₂ and DWCNT-ZrO₂ samples. The sliding speed was set at 5 cm.s⁻¹. Tests were performed with normal loads of 1, 5, and 10 N, depending on the ball used. Higher loads were not tested to avoid damaging the pellets and altering the contact geometry. The frictional force was recorded throughout the test using a load cell. Each friction test was repeated three times, yielding consistent results. Initial sample roughness was measured by white light interferometry (Zygo NewView 100). Wear tracks were analyzed by 3D optical profilometry (SENSOFAR S neox) on the samples and optical microscopy (Keyence VHX-1000E) on the balls.

Results and discussion

For all composite powders, the intensity ratio of the D band to the G band (I_D/I_G) in the high-frequency range of the Raman spectra (not shown) is comparable to that found for raw DWCNTs (0.11 ± 0.06 vs 0.13) [22], indicating that the functionalization and mixing processes did not damage the DWCNTs.

Analysis of the XRD patterns (not shown) revealed only the presence of tetragonal zirconia in all the sintered specimens. The relative density (Table 1) is in the range 98-100% for ZrO₂, C0.5, C1, C2, and C4.5 and is lower for samples with a higher carbon content, reaching 96% for C6, despite the higher sintering temperature (1350 vs. 1200 °C). This is in agreement with earlier studies showing that CNTs above a proportion inhibit densification [27]. The higher I_D/I_G ratio for the sintered nanocomposites (Table 1) compared to the nanocomposite powder could indicate that some CNTs were damaged during the SPS treatment. Ukai et al. [28] reported the formation of zirconium carbide during hot-isostatic pressing of MWCNT/YSZ nanocomposites at 1450°C, responsible of CNTs damage. However, this compound was not detected in the XRD patterns of the sintered nanocomposites, likely because SPS was conducted at lower temperatures and for shorter durations in the present study. The potential for CNT damage during SPS may be more closely associated with the material's mixed ionic-electronic conductivity at elevated temperatures [29]. Owing to their significant mobility, O²⁻ may react with the outer wall of the DWCNTs, resulting in localized damage. FESEM observations of the fracture surface (Figure 1) reveals that the fracture mode is intergranular, for all samples. The average grain size of the ZrO₂ sample is equal to 100 ± 10 nm (Figure 1a), only slightly higher than in the starting powder. The same size of 100 nm is observed for the zirconia matrix in all nanocomposites. For C6, the high amount of DWCNTs hampered grain growth despite the higher sintering temperature (1350 vs 1200°C), thus accounting for the lower relative density, as noted above. For all nanocomposites, the DWCNTs are well distributed at the matrix grain boundaries, without forming agglomerates (Figure 1b, c). However, increasing amounts of DWCNTs lead to the formation of larger diameter bundles (Figure 1b, c). FESEM images also evidence some residues of DWCNT for C4.5 (Figure 1c) resulting from the damage of a part of CNTs, all the more that a covalent functionalization was used [30]. FESEM observations of the C4.5 composite support the high I_D/I_G ratio (1.07).

The Vickers microhardness of the nanocomposites (13.8 - 9.5 GPa, Table 1) is lower than that of the ZrO₂ specimen (14.5 GPa, Table 1) and decreases regularly upon the increase in carbon content. The same behavior has been reported in fully densified SWCNT-ZrO₂ [31] and MWCNT-ZrO₂ [32] nanocomposites and associated to a weak interfacial bonding between the CNTs and zirconia. The values obtained in this study are higher than those reported for SWCNT-ZrO₂ [15,31,33] and MWCNT-ZrO₂ [32,34] nanocomposites, for which the composite powders were also prepared by a mixing route. The higher values could reflect a better dispersion of DWCNTs in the matrix and/or a slightly lower matrix grain size.

The arithmetic average roughness (Ra) calculated from white-light interferential rugosimetry images (not shown) is equal to 0.01 μm for ZrO₂ and is in the range 0.05-0.07 μm for C2, C4.5 and C6. These higher values can be ascribed to the tearing of grains in the nanocomposites, caused by the weakening of grain boundaries due to the presence of the DWCNTs. Typical curves showing the friction coefficient (μ) against the alumina ball versus distance, for a 5 N load, are shown in Figure 2a. For ZrO₂, C0.5 and C2, μ increases sharply during the running-in period and then stabilizes on

the last 5 m. By contrast, for C4.5 and C6, μ increases smoothly, with much less noisy curves. The observed noise reflects that the contact lacks stability and also a certain amount of wear, which is more pronounced for ZrO₂ and the nanocomposites with low carbon contents. The alumina ball being harder (15 GPa) than the samples (9.5 - 14.5 GPa), wear can probably be attributed to that of the sample. The average friction coefficients calculated on the last 0.5 m, versus carbon content, are presented in Figure 2b. For the sake of comparison, earlier results on eight-walls carbon nanotube-yttria-stabilized ZrO₂ nanocomposites (8WCNT-ZrO₂) [16] are reported in Figure 2b. At 5 N and 10 N, μ decreases for carbon contents higher than 2 wt.% and reaches a value of 0.23, i.e., 2.4 times lower than for ZrO₂ ($\mu \approx 0.55$). Thus, small amounts of DWCNTs probably weaken the ZrO₂ grain boundaries but do not provide a lubricating effect, in agreement with results on SWCNT-ZrO₂ [15] and MWCNT-ZrO₂ nanocomposites [16,17,35]. The diminution of the average friction coefficient is faster for 8WCNT-ZrO₂ than for DWCNT-ZrO₂ nanocomposites, probably because DWCNT pull-out is less easier than 8WCNT pull-out, limiting their participation to the contact lubrication. Indeed, DWCNTs are longer (up to several tens of micrometers) than 8WCNTs (1.5 μm) and their significant sinuosity between the ZrO₂ grains forms a network more firmly anchored in the ceramic. The lowest value of μ (0.23) reached at both 5 and 10 N, is lower than the one reported (0.35) by Hvizdoš et al. [36] who conducted tests under similar experimental conditions (pin-on-disk test, alumina ball, 5 N, 25 m, room temperature and dry conditions) on ZrO₂ matrix nanocomposites containing 1.07 wt.% of carbon in the form of carbon nanofibers (NFC).

Typical curves showing the friction coefficient (μ) against the steel ball versus distance, for a 5 N load, are shown in Figure 3a. At 1 N against steel, μ starts decreasing at a carbon content above 1 wt.%, at least two times lower than at 5 N (Figure 3b). Beyond 2 wt.%, μ no longer depends on the load and carbon content. The lower value (about 0.2 for both C4.5 and C6) is almost 3 times lower than that for the steel/ZrO₂ pair. The steel ball being less hard (8.6 GPa) than the samples (9.5 - 14.5 GPa), wear can probably be attributed to that of the ball and only the track width on the steel ball versus carbon content is presented (Figure 4). At 1 N, the wear track width on the steel ball remains constant at about 100 μm regardless of carbon content. At 5 N, wear track widths vary: those on steel ball in contact with samples with less than 2 wt.% of carbon are about 400 μm (similar to ZrO₂), while with C4.5 and C6, they are less than 200 μm , showing significantly less wear. Moreover, this reduced wear is associated to lower and similar friction coefficients for C4.5 and C6 in comparison to other samples. The difference between the hardness of the ball (8 GPa) and the hardness of the nanocomposites (Table 1) decreases as the carbon content increases. This makes the contact between the two less aggressive, leading to a reduction in the wear of the ball with a 5 N load. The surface and wear tracks for C4.5 and C6 were analyzed using Raman spectroscopy. In each case, a signal corresponding to carbon appeared. The I_D/I_G ratios calculated from the spectra (not presented) are similar, revealing no significant damage to the DWCNTs, in agreement with their elastic deformation [21]. Contrary to MWCNTs [16], DWCNTs are not severely damaged, cut, or destroyed to form a third lubricating body in the contact, at least in these experimental tribological conditions. Also, increasing carbon content in the form of DWCNTs appears to limit steel/ceramic contacts and improve the ball's sliding.

Conclusion

For the first time, a significant decrease of the average friction coefficient against both an alumina ball and a steel ball (by a factor of 2.4 to 3) is reported for DWCNT-ZrO₂ nanocomposites in comparison to ZrO₂. For the steel/nanocomposites pair, the decrease could result from the elastic deformation of the surface DWCNTs and the reduction of the steel/ceramic contacts. The presence of the DWCNTs also reduces the wear of the steel ball at high load. These tribological properties are achieved because of the specific microstructure of the nanocomposites DWCNTs quality, DWCNTs homogeneous dispersion, low matrix grain size and sample high densification even for a relatively high carbon content (6 wt.%).

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