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Ceramics International 40 (2014) 1289–1295

CERAMICS INTERNATIONAL

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Mechanical properties and electrical conductivity of alumina/MWCNT and alumina/zirconia/MWCNT composites

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Received 22 April 2013; received in revised form 5 June 2013; accepted 1 July 2013
Available online 6 July 2013

Abstract

The alumina/multi-wall carbon nanotubes (MWCNT) and Al_2O_3/ZrO_2/MWCNT composites with up to 2 vol% of MWCNT were prepared and characterized. The homogeneity of the composite powders was ensured by application of freeze drying and granulation as a method for drying of stabilized aqueous suspensions. The soft spherical granules were hot pressed at 1500 °C for 2 h under uniaxial mechanical pressure of 30 MPa to prepare dense composites. The mechanical properties, namely Vickers hardness (*HV1*) and fracture toughness (K_{Ic}), together with DC electrical conductivity (σ) were measured, and compared with the pure alumina reference prepared under similar conditions. Incorporation of 1 vol% of the MWCNT into the alumina, and alumina/zirconia matrices (composites AC and AZC, respectively) lead to the increase of fracture toughness by 8% and 35%, in comparison to the monolithic alumina reference. The electrical conductivity increased from 10^{-12} S/m in the pure alumina to 2.7×10^{-1} S/m in the composites containing 2 vol% of the MWCNT.

Keywords: C. Mechanical properties; Alumina; MWCNT; Percolation effect

1. Introduction

Since the discovery of carbon nanotubes in 1991 [1] attention has been paid to their incorporation into various materials, such as ceramics, alloys or polymers, with the aim to modify their physical properties. The functional properties such as electrical and thermal conductivity are of special interest. When considering the application of a composite, the influence of carbon nanotubes on mechanical properties is also of importance. Due to the extraordinary properties of the MWCNT, like extremely high Young's modulus (~1 TPa) [2], tensile strength (~60 GPa) [3], electrical conductivity ($>10^5$ S/m), and thermal conductivity (>3000 W/mK) [4,5] their addition can modify the properties of the composites to a large extent. Due to high aspect ratio of the MWCNT, the percolation threshold and related increase of the electrical conductivity in the materials with insulating ceramic matrix can be easily achieved. Lee et al. [6] reported that the percolation threshold can be reached by the addition of only 0.18 wt% of the MWCNT dispersed in the Al_2O_3 matrix. The highest electrical conductivity of a ceramic composite (3345 S/m) was achieved by the addition of 15 vol% of single-walled carbon nanotubes (SWCNT) into ceramic matrix [7]. However, the question of reinforcement of ceramics by the addition of MWCNT is still a matter of controversy. The improvement of mechanical properties comes preferably at lower contents of the MWCNT (≤4 vol%) [8], whereas at higher concentrations the reported values of mechanical properties, and the fracture toughness in particular, are more scattered [9,10]. Whether the mechanical properties of the composite are improved or not is closely related to the homogeneity of distribution of the CNT in the ceramic matrix, and the ability to prepare a fully dense and defect-free material. Due to high aspect ratio and the tendency of CNT to agglomerate, their deagglomeration remains a challenge. Various processing routes targeting deagglomeration of the MWCNT have been proposed and employed, such as conventional powder mixing [11], colloidal processing [12] or *in situ* growth of the CNT by chemical vapour

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http://dx.doi.org/10.1016/j.ceramint.2013.07.008

decomposition [13]. For densification of composite, CNT-containing green bodies, hot pressing (HP) [14] has been commonly applied, along with other pressure-assisted techniques, such as spark plasma sintering (SPS) [15], hot isostatic pressing (HIP) [16] or pressureless sintering (PS) [17].

Polycrystalline alumina as a matrix for the composites with added carbon nanotubes has several advantages, including good mechanical properties (high hardness, wear resistance, elastic modulus), and high chemical stability. The main disadvantages of majority of alumina-based ceramics, which prevent their wider application, are low fracture toughness and electrical conductivity. Preparation of zirconia toughened alumina (ZTA) containing typically around 5 vol% of tetragonal zirconia inclusions is often reported as the way of improving the fracture toughness of alumina-based ceramics [18–20]. The microstructure refinement (suppression of the growth of alumina matrix grains by pinning with zirconia inclusions), transformation toughening and micro-cracking, are reported as the main mechanisms that favourably influence the mechanical properties, and fracture toughness in particular. The combination of alumina matrix with zirconia and carbon nanotubes as minor additives could thus result in a composite with good mechanical properties and increased electrical conductivity, with a wide range of applications, including medicine, nanorobotics or cooling and heating elements.

This paper deals with the preparation and characterization of the Al_2O_3/MWCNT and Al_2O_3/ZrO_2/MWCNT composites. The composites are prepared by colloidal processing and freeze drying of the composite powders, followed by hot pressing at 1500 °C. The materials are characterized from the point of view of their microstructure and physical properties, and compared to the pure alumina reference prepared under similar conditions. The influence of relatively low additions (up to 2 vol%) of the MWCNT on fracture toughness, hardness, and electrical conductivity is investigated.

2. Experimental

2.1. Materials

High purity α-alumina powder Taimicron TM-DAR (Taimei Chemicals Co., Ltd., Tokyo), with the mean particle size ~150 nm and the specific surface area 13.7 $m^2\,g^{-1}$ was used as a starting material for preparation of the alumina matrix. The MWCNT (Chengdu Organic Chemicals Co., Ltd., China) with the length ~25 μm and the diameters in the range 7–15 nm, synthesized by chemical vapour deposition were incorporated into the matrix. The yttria partially stabilized tetragonal zirconia powder TZ-3Y-E (Tosoh Corporation, Tokyo, Japan) with the specific surface area 15.1 $m^2\,g^{-1}$ was also used. Polyelectrolyte dispersant Darvan C-N (ammonium polymethylmetacrylate, R.T. Vanderbilt Company, Inc., Norwalk, USA,) was used to stabilize the aqueous suspensions of the alumina and zirconia powders. An anionic surfactant sodium dodecylsulphate (later on denoted as SDS, Sigma-Aldrich 42 Co., Germany) was applied to improve the dispersion and to ensure homogeneous distribution of the nanotubes in the solution [8]. All other employed chemicals were of the analytical grade (Sigma-Aldrich 42 Co., Germany).

2.2. Processing

The pristine MWCNT were functionalized by heating them in 65% HNO_3 at 80 °C for 8 h to eliminate their hydrophobic nature [21,22].

The preparation of the suspension is in detail described in our previous work [23]. The dispersion of the MWCNT in SDS/water solution was ensured by a 2 min sonication with the use of a horn sonicator SONOPLUS UW 2200, (BANDELIN electronic GmbH & Co.KG), at the 200 W input power, frequency 20 kHz, and the amplitude 30%. The sonication was employed before mixing the MWCNT with other components. The fraction of the MWCNT was related to the amount of alumina, and varied between 0.5 and 2 vol%.

The Al_2O_3 and ZrO_2 suspensions were respectively stabilized by the addition of 2.2 wt% and 2.4 wt% of the commercial polyelectrolyte Darvan C-N, related to the amount of solid in the suspension. Before adding the polyelectrolyte the pH of the suspensions was adjusted to ~10 by the addition of the NH_4OH water solution. The composite suspensions were prepared by mixing the stabilised suspensions of individual components at the ratio, which ensured the total 35 vol% solid loading in the mixture, and its ball milling on rollers for 24 h.

The homogenous suspension was then sprayed into liquid nitrogen ($T\sim-196$ °C) and freeze dried ($T\leq-50$ °C in vacuum) for 24 h to prevent agglomeration during drying and to preserve homogeneous dispersion of ZrO_2 and the MWCNT in the dried powder. The granules were spherical, with the diameter of ~100 μm. For preparation of the pure, pressureless sintered alumina references the granules were filled into a steel die, and axially pressed at 50 MPa to prepare green pellets 50 mm in diameter and 6 mm thick. The pellets were then sintered for 1 h at 1300 °C, 20 °C/min heating rate, in an electric resistance furnace (NETZSCH GmbH Selb Nemecko) with $MoSi_2$ heating elements. The composite granules were filled into a rectangular graphite die (60×60 mm^2), and hot pressed in Ar atmosphere, without previous consolidation, for 2 h at 1500 °C, 20 °C/min heating rate. Uniaxial mechanical pressure of 30 MPa was applied at 1200 °C during heating, and released at the same temperature during cooling.

The compositions of the suspensions and the composites are in the following text denoted as follows:

- **AA** – the monolithic alumina reference,
- ***x*AC** – Al_2O_3/MWCNT composite, where *x* indicates the volume fraction of the MWCNT, and
- ***x*AZC** – Al_2O_3/ZrO_2/MWCNT composite, where *x* indicates the volume fraction of the MWCNT; the fraction of ZrO_2 was kept constant at 5 vol% with respect to alumina.

2.3. Characterization

The relative densities of sintered materials were determined by the Archimedes method in distilled water. The microstructure was examined by scanning electron microscopy (SEM) (Zeiss EVO 40 HV, Germany, and JEOL 7600f, JEOL, Japan) on fracture surfaces of sintered samples. The linear intercept method (software LINCE, TU Darmstadt, Germany) was employed for determination of the mean size of alumina and zirconia grains. At least 200 intercepts was considered for each material, and the correction factor 1.56 was applied [24]. Mechanical properties, namely the hardness (*HV1*) and fracture toughness (K_{1c}) were determined by Vickers indentation with the use of the hardness tester LECO LV 100 (FutureTech Corp, USA). The fracture toughness was calculated from the minimum of 10 indentations carried out at the load of 98.1 N, using Shetty's equation [25]. The Vickers hardness was evaluated adopting an indentation load $P=9.81$ N. In both cases a dwell-time of 10 s was applied at the maximum load. The DC electrical resistivity was measured on flat rectangular samples with dimensions 15×15 mm^2, polished from both sides, by four point probe technique using the RLCG bridge TESLA BM 595 [26], and recalculated to the electrical conductivity using Ohm's law. The value of the electrical conductivity is the mean value of at least 5 independent measurements at various places of the specimen. The scatter of measured values did not exceed ±5%.

3. Results and discussion

3.1. Densification

Table 1 summarizes the basic characteristics of prepared materials, namely the mean size of alumina and zirconia grains and relative densities of the hot pressed AC and AZC composites, and of the monolithic alumina reference AA. The starting powder Al_2O_3 is known for its good sinterability at relatively low temperatures. The temperature of 1300 °C and 1 h isothermal dwell at the maximum temperature is usually sufficient for achievement of the density close to the theoretical (99.8 ± 0.2% of the theoretical density) [27]. The incorporation of the MWCNT or both the MWCNT and ZrO_2 particles severely impaired densification so that 2 h of isothermal dwell at 1500 °C, and application of the mechanical pressure was required to obtain a dense material. The relative densities at the level of 99% of the theoretical density were achieved in all composite materials by the hot pressing. The influence of residual porosity and the effects related to insufficient densification on mechanical and functional properties could be thus neglected. All observed changes were therefore attributed to compositional and microstructural changes related to the addition of ZrO_2 and the MWCNT, and their dispersion in the alumina matrix. In the composite 0.5AC the growth of the alumina matrix grains was not suppressed. However, some microstructure refinement was observed with the increasing content of the MWCNT. In the composites AZC the addition of zirconia led to slight increase of the relative density in comparison to the composites AC containing the same volume fraction of the MWCNT. Moreover, significant microstructure refinement was observed in the ZrO_2-containing composites, which was primarily attributed to the pinning effect of the ZrO_2 inclusions. The SEM micrographs are shown in the Fig. 1a–h: the trends in the microstructure development are clearly visible.

3.2. Mechanical properties

Homogeneous dispersion and distribution of carbon nanotubes is prerequisite for improvement of mechanical properties of the CNT-reinforced composites. This requirement is often in contradiction with the requirement of high volume fraction of the reinforcing phase: high content of the MWCNT has often deleterious effect related to the presence of agglomerates of entangled nanotubes. The influence of lower volume fractions of the MWCNT on mechanical properties is therefore often more pronounced. The toughening mechanisms, such as pull-out of nanotubes, crack deflection, and crack bridging [28–30] come forward only in the absence of agglomerates. Another parameter influencing the fracture toughness – the mean size of the alumina matrix grains – should be also considered. Coarser grained microstructure in ceramics is often identified with higher fracture toughness, due to more winding crack path, and the contribution of the toughening mechanisms, such as crack deflection and pull out of the alumina matrix grains. However, our previous investigations [23] as well as the results published by Casellas et al. [31] indicate limited influence of the size of the alumina matrix grains on the fracture toughness. This can be attributed to isometric shape of the matrix grains, which do not represent an effective obstacle to crack propagation. It can be thus concluded that any observed change of the fracture toughness can be attributed to the influence of the addition of the reinforcing phases (i.e. MWCNT and/or *t*-ZrO_2) alone, and not to the size of alumina matrix grains. In the composites AC

Table 1
The mean size of Al_2O_3 and ZrO_2 grains and relative densities of prepared materials after sintering.

Samples	Grain size Al_2O_3 [nm]	Grain size ZrO_2 [nm]	Relative density [%]
AA	940 ± 80		99.8 ± 0.2
0.5AC	1700 ± 100		98.9 ± 0.3
1AC	1600 ± 100		99.5 ± 0.3
1.5AC	1900 ± 200		98.4 ± 0.2
2AC	1200 ± 100		98.6 ± 0.4
0.5AZC	900 ± 100	240 ± 40	99.4 ± 0.1
1AZC	800 ± 50	290 ± 10	99.0 ± 0.3
1.5AZC	760 ± 80	290 ± 30	99.1 ± 0.2
2AZC	850 ± 90	300 ± 10	99.0 ± 0.1

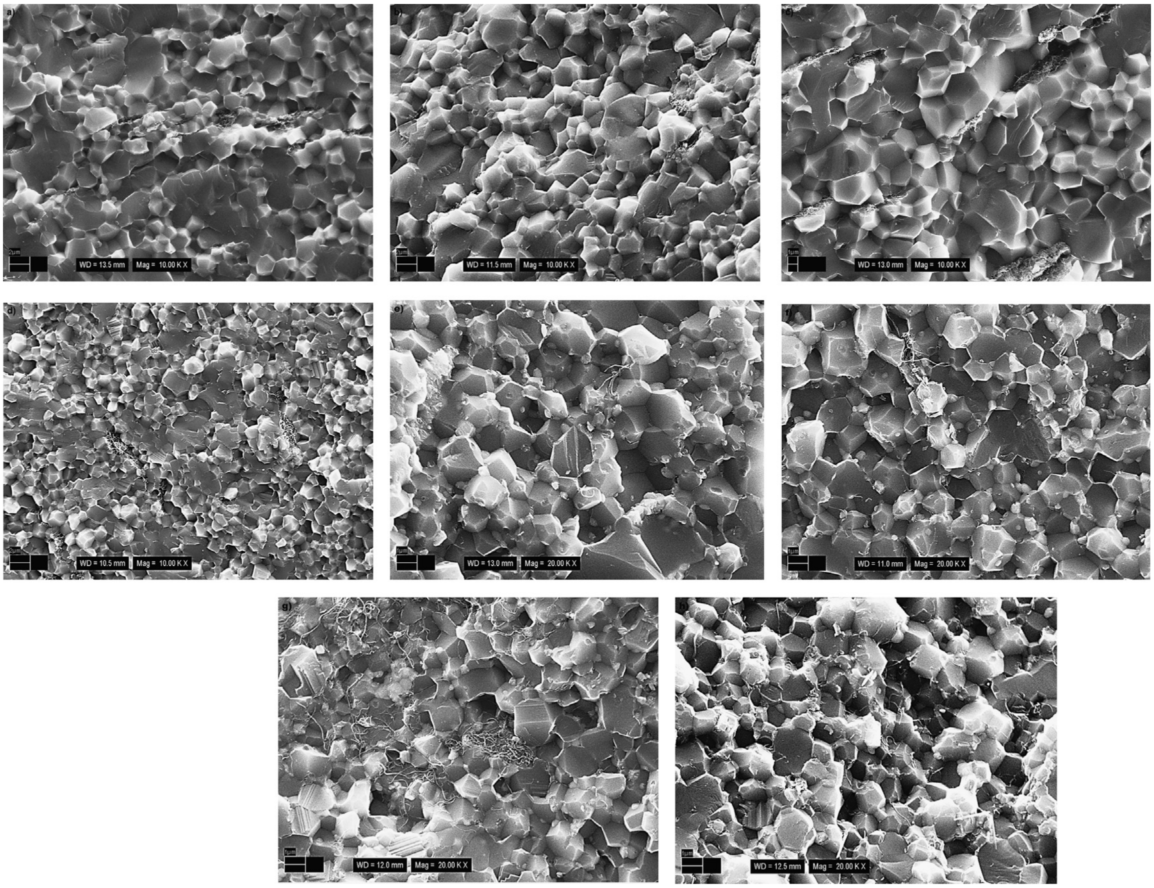

Fig. 1. Microstructures of the composites AC and AZC: (a) 0.5AC; (b) 1AC; (c)1.5AC; (d) 2AC; (e) 0.5AZC; (f) 1AZC; (g) 1.5AZC, and (h) 2AZC.

the increase of the indentation fracture toughness of only 8% with respect to the monolithic alumina reference was achieved by the addition of 1 vol% of the MWCNT (Fig. 2). The result is in a good agreement with the observation of Balani et al. [32], who reported the increase of the fracture toughness by 11.6% in the composite with 0.5 wt% of carbon nanotubes. The indentation fracture toughness of the composites with different volume fractions of the MWCNT prepared in this work was comparable to that of the monolithic alumina reference. Slight decrease was observed in the composite 2AC (Fig. 2). This result was attributed to frequent occurrence of agglomerates and poor dispersion of the nanotubes in the alumina matrix, which were frequently observed in the microstructure of sintered composites by SEM (Fig. 1).

The expected increase of hardness reported by Inam et al. [33] (25% increase of hardness in the composite Al_2O_3+1 vol% CNT in comparison to the pure alumina reference) was not achieved. In the contrary, the addition of the MWCNT to the alumina matrix led to significant decrease of the Vickers hardness with respect to the monolithic alumina, irrespective of the volume fraction of the MWCNT added. The result was attributed to the coarser grained microstructure of the composites prepared in this work and weak interfacial bonding between the MWCNT and the grains of the alumina matrix [29]. Additionally, the agglomerates of the MW CNT dispersed throughout the composites' microstructure, acted as pores, further decreasing the hardness. However, in the composition range examined, and considering the Vickers hardness and the indentation fracture toughness, the addition of 1 vol% of the MWCNT, which corresponds to 0.3 wt%, could be considered as the optimum content of the nanotubes in the composite in terms of its mechanical properties.

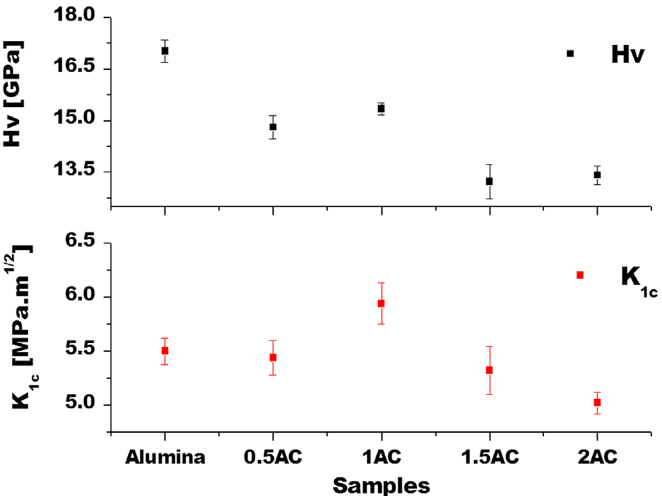

Fig. 2. Vickers hardness and indentation fracture toughness of the samples AA and AC.

The volume changes related to the phase transformation of the metastable tetragonal zirconia to monoclinic are known to result in transformation toughening of a material with added transformable particles of partially stabilized tetragonal ZrO_2 [34]. The transformation toughening is believed to play an important role also in the composites AZC studied in this work. The indentation fracture toughness increased from 5.5 MPa $m^{1/2}$ in the monolithic alumina reference to 6.9 ± 0.5 MPa $m^{1/2}$ in all AZC composites with the exception of the composite 1AZC (1 vol% of the MWCNT) where the indentation fracture toughness of 7.5 ± 0.5 MPa $m^{1/2}$ was achieved (Fig. 3). This represented a 35% increase of the indentation fracture toughness in comparison to the pure alumina reference. Homogeneous dispersion of the MWCNT and ZrO_2 inclusions contributed to the improvement. The toughening mechanisms in operation are illustrated in Fig. 4. The pull out and crack bridging by a MWCNT is shown in Fig. 4b, and possible transformation of a ZrO_2 inclusion in contact with propagating crack is shown in Fig. 4c. The presence of radial microcracks propagating from the surface of the ZrO_2 inclusion into the alumina matrix grains (marked with arrows in Fig. 4c) provides the evidence that a transformation of tetragonal zirconia to monoclinic, accompanied by volume expansion, took place during the crack propagation, and the transformation toughening contributed to the observed increase of the fracture toughness. The Vickers hardness decreased from 17.5 GPa in the monolithic alumina reference (sample AA) to ~14.5 GPa for all prepared AZC composites. Incorporation of ZrO_2 into the alumina matrix was thus found to impair the hardness of the prepared composites to larger extent than the addition of the MWCNT in the composite AC. This result was in accord with the published data, which reported increased fracture toughness of zirconia toughened alumina (ZTA) [19,35], but also detrimental effect of zirconia addition on its hardness [36,37]. The hardness of the used zirconia was 12.5 GPa (the data provided by the producer, and determined according to JIS R1610 at the load of 98.07 N). The reduction of hardness can be thus attributed both to the addition of the softer phase and to spontaneous micro crack formation around transformed monoclinic zirconia particles.

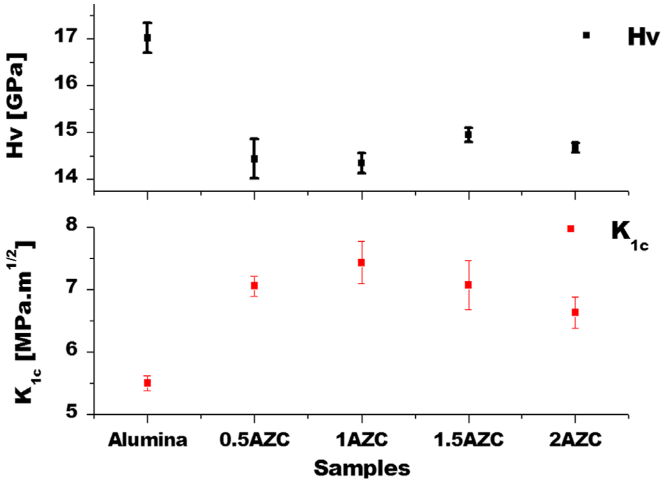

Fig. 3. Vickers hardness and indentation fracture toughness of the samples AA and AZC.

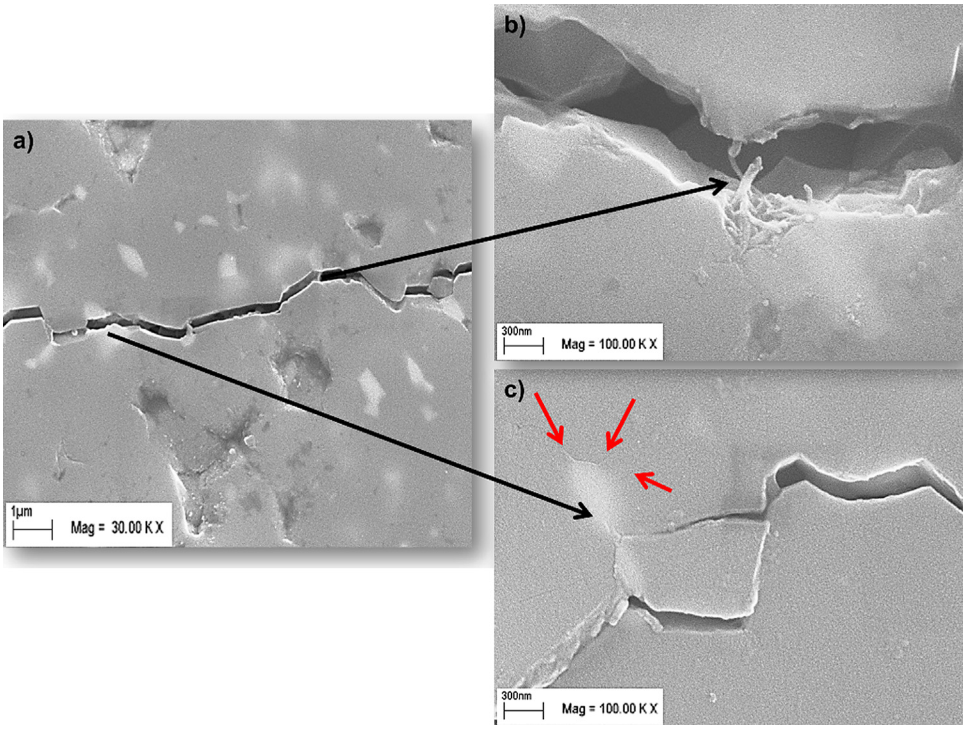

Fig. 4. The examples of toughening mechanisms observed on SEM micrgraphs of the 1AZC composite: (a) crack propagation; (b) nanotube pull out and crack bridging by a MWCNT, and (c) possible transformation of a ZrO_2 particle.

3.3. Electrical conductivity

Due to the high aspect ratio of the MWCNT, which in our case was around 1700, the percolation threshold could be easily achieved in the MWCNT-reinforced composites. However, the achievement of the percolation threshold was related to the microstructure of the composite, as was demonstrated by the results shown in Fig. 5. The addition of identical volume fraction of the MWCNT (0.5 vol%) resulted in the increase of the electrical conductivity from 10^{-12} S/m in the pure alumina reference to 2.2×10^{-3} S/m and 2.8×10^{-5} S/m in the composites 0.5AC or 0.5AZC, respectively, which represented the difference of two orders of the magnitude for qualitatively similar materials. In the composite AC, the coarser-grained alumina matrix with the MWCNT aligned along the grain boundaries facilitated the formation of free conductive paths from mutually interconnected MWCNT. The increase of the electrical conductivity with increasing content of the MWCNT was therefore approximately linear (Fig. 5a). Non-linear dependence of the electrical conductivity observed for the composites AZC, and shown in the Fig. 5b, was attributed to finer grained microstructure of the material due to the presence of pinning of ZrO_2 inclusions. The finer-grained microstructure in the AZC composites led to poor alignment of the MWCNT with related increase of the percolation threshold. Only moderate improvement of the electric conductivity in the materials with lower content (up to 1 vol%) of the MWCNT was therefore observed. Marked increase of the electric conductivity was observed once the volume fraction of the MWCNT achieved 1.5 vol% (the composite 1.5AZC). The highest measured value 2.2×10^{-1} S/m in the composite 2AZC was comparable to that obtained for the material 2AC. It can be thus speculated that with the increasing total number of conductive paths the negative influence of small grain size on the electric conductivity was less pronounced. However, it is worth mentioning that in the composites containing 2 vol% of the MWCNT the difference in the mean size of alumina grains (848 and 1238 nm for 2AZC and 2AC, respectively) was smaller than in the case of the materials with 0.5 vol% of the MWCNT (1732 and 845 nm in the composite 0.5AC and 0.5AZC, respectively). The conductivity threshold could be thus at the low MWCNT content achieved more readily in coarse-grained composites.

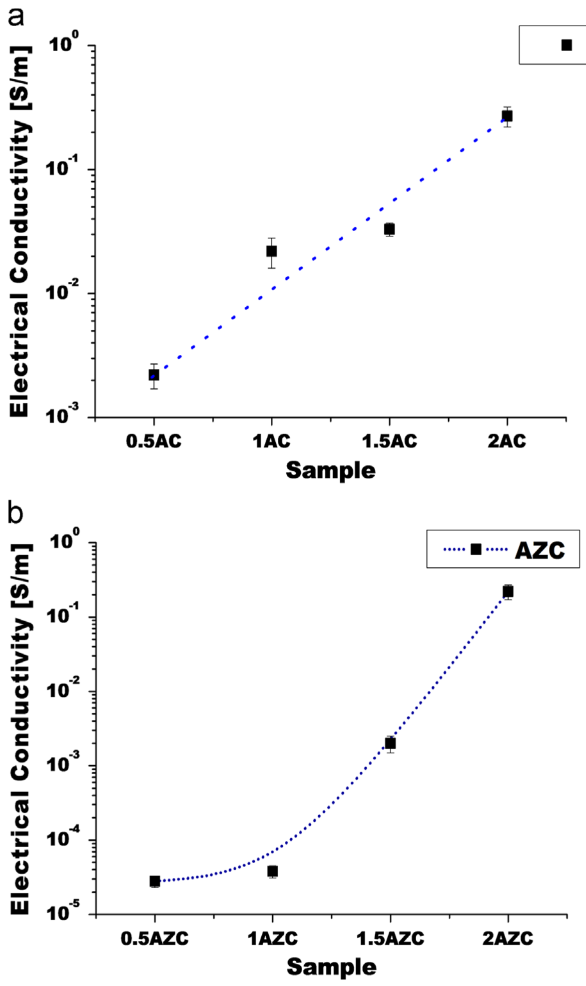

Fig. 5. Electrical conductivity of the prepared composites (a) AC and (b) AZC.

4. Conclusions

Homogeneous MWCNT-containing alumina and alumina/zirconia powders with 0.5–2 vol% of the MWCNT were prepared by freeze drying of homogeneous and stabilized aqueous suspensions. Dense Al_2O_3–MWCNT (AC) and Al_2O_3–ZrO_2–MWCNT (AZC) composites with 5 vol% of partially stabilized ZrO_2 were then obtained by hot pressing. The indentation fracture toughness of the composites increased by 8% and 35% for 1AC and 1AZC, respectively, in comparison to the pure alumina reference material. The optimum content of the MWCNT with respect to the fracture toughness was 1 vol% (or 0.3 wt%), for both studied compositions. The DC electric conductivity increased with increasing volume fraction of the MWCNT. However, the measured dependences differed for AC and AZC, and were related to different microstructures. The highest electric conductivity was achieved in the materials with the highest volume fraction of the MWCNT, and was comparable for both studied matrices (2.2×10^{-1} S/m and 2.7×10^{-1} S/m for the composites 2AZC and 2AC, respectively). These values were more than 10 orders of magnitude higher than the electric conductivity of the pure alumina reference.

Acknowledgements

The financial support of this work by the grant LPP 0297-09, and VEGA 1/0206/11 is gratefully acknowledged. This publication was created in the frame of the project “Centre of excellence for ceramics, glass, and silicate materials“ ITMS code 262 201 20056, based on the Operational Program Research and Development funded from the European Regional Development Fund. The authors wish to express their thanks to dr. Peter Švec from the Institute of Physics, Slovak Academy of Science, for measurement of the electrical properties.

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