



# High/room temperature mechanical properties of 3Y-TZP/CNTs composites

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## Abstract

Spark Plasma Sintering (SPS) method was practiced, in the current investigation, to obtain fully dense materials with an excellent dispersion of MWCNTs (Multi-walled Carbon Nanotubes) within the matrix. Scanning and transmission electron microscopy micrographs as well as Raman Spectroscopy revealed no conspicuous signs of damages on the nanotubes' structure, as a result of applying appropriate processing measures. Measurements of mechanical properties revealed the value of  $15.2 \pm 0.4 \text{ MPa m}^{1/2}$  for indentation fracture toughness, implying our success to align transformation toughening (while the zirconia has the grain size around  $1 \mu\text{m}$ ) with the effects of MWCNTs addition (5 wt%). High temperature mechanical properties of the composites have been investigated using Mechanical Spectroscopy (MS) up to 1600 K at frequency equal to 1 Hz. The MS results proved that CNTs could hinder grain-boundary sliding and subsequently lead to an enhanced creep resistance at high temperatures.

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## 1. Introduction

Discovery of carbon nanotubes (CNTs), during the recent decade, has aroused considerable interests in utilizing these materials as reinforcing or functionalizing elements for fabrication of CNTs added composites [1]. High Young's modulus ( $\sim 1500 \text{ GPa}$ ), high tensile strength ( $\sim 100 \text{ GPa}$ ), high aspect ratio, high electrical conductivity, and other promising properties are the examples of such fantastic performances of CNTs, that convinced researchers to incorporate CNTs into different composites [1–4]. In the case of Ceramic-based/CNTs composites, different processing routes have been carried out including hot isostatic press, hot press, and spark plasma sintering (SPS) [5,6]. Among all these methods, SPS has been found presumably successful to preserve CNTs from the probable damages through the fabrication process [7]. Such preservation in SPS method has been attained due to the

combined effect of lower sintering temperature and shorter sintering time in comparison to conventional techniques. For instance, we have reported a successful technique for dispersion of CNTs within 3Y-TZP particles followed by SPS at relatively low temperatures. Such an approach was found to result in near full-dense structures with well-distributed CNTs and consequently significant improvement in mechanical properties such as fracture toughness [7,8].

It is worth to mention that the great interests in the zirconia based ceramics through the recent decades stem from their notable hardness, wear resistance, chemical inertness, low thermal conductivity, and high melting temperature [9–11]. All the above fantastic properties have turned these materials into promising candidates for several engineering applications. They are also the focus of continuous efforts to develop an enhanced knowledge around the betterment of their mechanical behavior. While the literature is filled with several investigations on different properties of zirconia ceramics, one can assume that theatrical increase in demands on industrial applicability of these materials could be safely associated with the discovery of transformation toughening

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[12]. It was shown that the well-known stress-induced phase transformation mechanism could result in a remarkable increase in the fracture toughness of these ceramics. In particular, metastable tetragonal zirconia at room temperature can be recognized as the toughening agent, transformation of which to the stable monoclinic structure – in the vicinity of the crack front – has been always recognized to be responsible for the increased fracture toughness. On the other hand, the maintenance of the tetragonal structure at room temperature (the metastable phase) – as the key factor to attain toughened zirconia materials – depends on the stabilizer (type and amount) and tetragonal grain size [13,14]. In fact, a critical grain size is involved, below which the high temperature tetragonal phase can be retained in the microstructure. It has been also reported that decreasing the grain size from this critical value would lead to a monotonic decrement of fracture toughness up to a factor of 5 (from 15–16 MPa to 2–5 MPa). Such a failure might be attributed to over-stabilization of the tetragonal phase on the account of the small grain size. In such circumstances, the tetragonal phase may not be very much inclined to undergo any transformation process to the monoclinic phase upon the instigation of a crack [15]. The above results indicate that proximity of the specimens' grain size to the critical value should be appreciated as the essential parameter to derive the maximum value of toughness in zirconia ceramics. It has been reported that this critical grain size for 3Y-TZP (3 mol%  $Y_2O_3$  stabilized zirconia) is not less than 1  $\mu m$  at room temperature [16–21].

At high temperatures, on the other hand, different mechanisms are involved. Mechanical properties of zirconia polycrystals have been numerically reported [22–25] to endure a drastic decrease at high temperatures due to thermally activated grain boundary (GB) sliding, leading to plastic or even super-plastic deformation. Wakai and his colleagues [24], for instance, reported 120% elongation for a fine-grained (< 300 nm) 3Y-TZP compact at 1723 K. Since GB sliding is potential source of energy dissipation within the material, mechanical loss measurements are developed to study such a mechanism [26]. Using mechanical spectroscopy, therefore, Mazaheri [27,28] demonstrated a theatrical decrement of mechanical loss value at 1600 K for CNT reinforced 3Y-TZP composites. This indicated a restricted grain boundary sliding in the composite and consequently a lower creep rate compared to the monolithic zirconia.

What should be taken into account is the effect of transformation toughening. Such an effect has been clearly overshadowed in the previous researches, since the grain size of stabilized zirconia, in these reports, are much lower than the

critical size (around 100–300 nm). The objective of the present study, on the other hand, is to align the effects raised by transformation toughening and CNTs addition to increase the value of fracture toughness. As an additional scope of research, the present paper has focused on the high temperature mechanical properties of these materials. It also represents evidences to demonstrate a decreased GB sliding at high temperatures for CNT reinforced composites.

## 2. Material and methods

Commercially available high purity 3 mol%yttria stabilized zirconia (3Y-TZP) powder (Tosoh Co., Japan) and multi-walled carbon nanotubes (Arkema, France) were applied as starting materials. The multi-walled carbon nanotube material, used in this research, is the commercially available grade of Graphistrength C-100, with the length of around 10  $\mu m$  and a diameter of about 10–20 nm.

3Y-TZP powder with 5 wt% MWCNTs, were mixed using turbula mixer with zirconia balls for 24 h. For microstructural observations of mixed composites, carbon-coated specimens were put through the characterization tests using HR-SEM (HR-SEM, FEI-SFEG, Philips, Netherlands). In order to derive full-dense composites (> 98% of theoretical density, TD) and avoid damage of nanotubes, the specimens were consolidated using SPS (FCT GmbH, Germany) under vacuum at different sintering temperatures (Table 1). The composite powders were loaded into a graphite die (40 mm inner diameter) and a sheet of graphite paper was placed between the powders and die/punches to ease the specimen removal. The heating rate, soaking time and applied pressure during SPS were 50 K  $min^{-1}$ , 2 min and 50 MPa, respectively. High-resolution transmission electron microscopy (HR-TEM, CM-200, Philips, Netherlands) with a field emission gun operating at 200 kV on electron transparent area have been used to study the grain boundaries as well as the effect of carbon nanotubes on the microstructural evolution of the materials at high temperatures. TEM Specimens were prepared by the typical procedure of tripod polishing method, followed by ion-milling.

The X-ray diffraction (XRD) pattern of the fracture surface has been studied using Cu  $K\alpha$  radiation (Philips PW3710, Netherlands). The weight fraction of the monoclinic phase,  $X_m$ , was calculated using the following formula [29]:

$$X_m = \frac{I(11\bar{1})_m + I(111)_m}{I(11\bar{1})_m + I(111)_m + I(111)_t} \quad (1)$$

where  $I(11\bar{1})_m$ ,  $I(11\bar{1})_m$  and  $I(111)_t$  are the integrated intensity

Table 1  
Sintering temperatures and characteristics of sintered composites.

Sample code	Sintering temperature (°C)	Relative density	Grain size ( $\mu m$ )	Monoclinic phase (wt%)	Fracture toughness ( $MPa m^{1/2}$ )
L-Z	1250	$99.3 \pm 0.08$	0.14	$32.7 \pm 0.5\%$	$5.8 \pm 0.28$
L-C	1350	$98.4 \pm 0.06$	0.1	$22.8 \pm 0.5\%$	$10.9 \pm 0.4$
H-Z	1500	$99.0 \pm 0.05$	1.7	$64.5 \pm 0.5\%$	$10.8 \pm 0.7$
H-C	1500	$98.1 \pm 0.10$	1.1	$59 \pm 0.5\%$	$15.2 \pm 0.4$

from the monoclinic (111), monoclinic ( $11\bar{1}$ ) and the tetragonal (111) peaks, respectively.

Indentation tests were carried out with a diamond Vickers indenter under 20 kg load and dwell time of 20 s on the polished surfaces. The fracture toughness was also calculated by Antis equation in which the crack lengths were measured immediately after indentation using a calibrated optical microscope. Each data point was reported from the averaged value of at least 10 measurements.

Mechanical spectroscopy measurements were accomplished in an inverted forced torsion pendulum, working at subresonant frequency. Specimens with the size of  $25 \times 1 \times 4 \text{ mm}^3$  were, primarily, excited as a result of torsion effect, and an optical cell could detect deformation of the samples. The measurements were performed under a high vacuum ( $10^{-3} \text{ Pa}$ ) as a function of temperature in the range of 300–1600 K, at constant frequency of 1 Hz. The mechanical loss,  $\tan(\phi)$ , was measured from phase lag between stress and strain signals. The amplitude of the applied cyclic stress during measurements was about 6 MPa.

### 3. Results and discussion

Fig. 1 shows HR-SEM of mixed zirconia nanopowders with 5 wt% ( $\sim 12.5 \text{ vol\%}$ ) MWCNTs. No evidence of CNTs damage and agglomeration can be observed. As can be seen in the figure, nanotubes of carbon seem to be neatly separated from bundles to the single ropes and wrap around borders of the matrix agglomerates. Such an impressive dispersion of carbon nanotubes through the particles of zirconia nanopowders seems to be hardly achievable at specifically 12.5 vol%, as according to the literature, many of the previous researchers have reported unsuccessful experiences of dispersing carbon nanotubes in much lower fractions [30–33]. This dazzling effect could be associated to the application of turbula mixer as the state of the art method for dry dispersion of carbon nanotubes.

Table 1 represents the relative density, grain size and monoclinic weight fraction of zirconia in addition to fracture toughness of the samples sintered at different temperatures. As

can be observed in the table, monolithic zirconia specimens have been sintered at 1250 and 1500 °C labeled as L-Z and H-Z, respectively. In order to highlight the effect of carbon nanotubes in the properties of final material, the specimens containing 5 wt% carbon nanotubes coded by L-C and H-C have been sintered at 1350 and 1500 °C, respectively. Considering the data available in the table, all the specimens have attained the relative densities closed to the full dense value, thanks to the SPS. What should be taken into account is that the higher sintering temperatures have resulted in the specimens with larger grain size and such an effect has been suggested to account for the generation of higher fractions of monoclinic phase in the fracture surface of these specimens compared to their low temperature sintered counterparts. The occurrence of such an incident, apart from the effect of CNTs, is expected to cause transformation toughening mechanism in the zirconia microstructure and consequently a remarkable increment of fracture toughness as a result of tetragonal to monoclinic transformation.

On the other hand, the incorporation of 3Y-TZP samples with nanotubes of carbon seems to have presumably given rise to relatively different properties compared to those of monolithic specimens. As can be observed in the table, the addition of CNTs has increased the sintering temperature required for the full densification of the composite. Such an effect is not planned to be focused in the current paper, as the retarding effect of CNTs for densification of zirconia powder has been previously discussed in another publication of the authors [7]. In particular, what should be noted is the obvious grain size refinement in the L-C and H-C samples compared to the monolithic ones.

As a result of such a refinement, and consequently limiting the chance of transformation toughening, lower amount of monoclinic phase is quite understandable at fracture surface of L-C and H-C.

In order to attain a visual evidence for the above hypothesis, one can observe the HR-TEM figures (Fig. 2a and b) of 3Y-TZP/5 wt% CNT composite microstructures after SPS at 1350 and 1500 °C, respectively. Fairly homogenous dispersion of well-survived CNTs in the zirconia matrix has allowed them to be perfectly distributed through the whole microstructure and be located on the grain boundaries, as can be observed in the figures. The stretched and wavy structure of carbon nanotubes, on the other hand, lead them to wrap around the zirconia grains and set serious restrictions against the grain growth at final stages of sintering when the collapsed porosities of the ceramic can be hardly disappeared unless with the occurrence of grain growth to some extent. That can be the other reason for the higher sintering temperatures required to densify CNTs contained composites.

Fracture toughness values of the specimens are the other important factors that should be carefully taken into consideration. By a comparison between the monolithic samples (L-Z and H-Z), there remains no doubt that the higher sintering temperature of H-Z sample has resulted in coarser grain size value and consequently higher probability of transformation toughening (the higher amount of monoclinic phase would

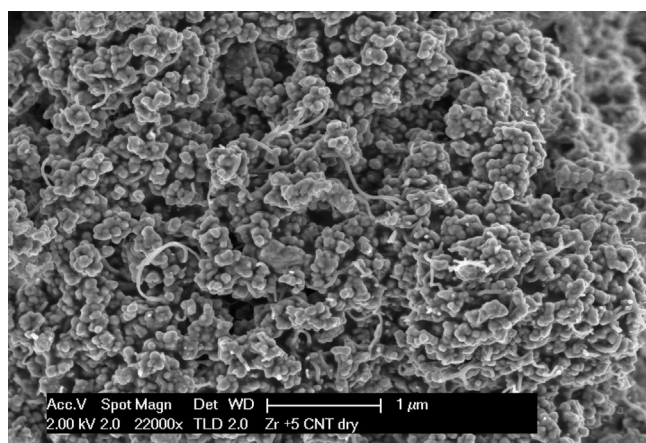


Fig. 1. HR-SEM of mixed zirconia nanopowders with 5 wt% ( $\sim 12.5 \text{ vol\%}$ ) CNTs.



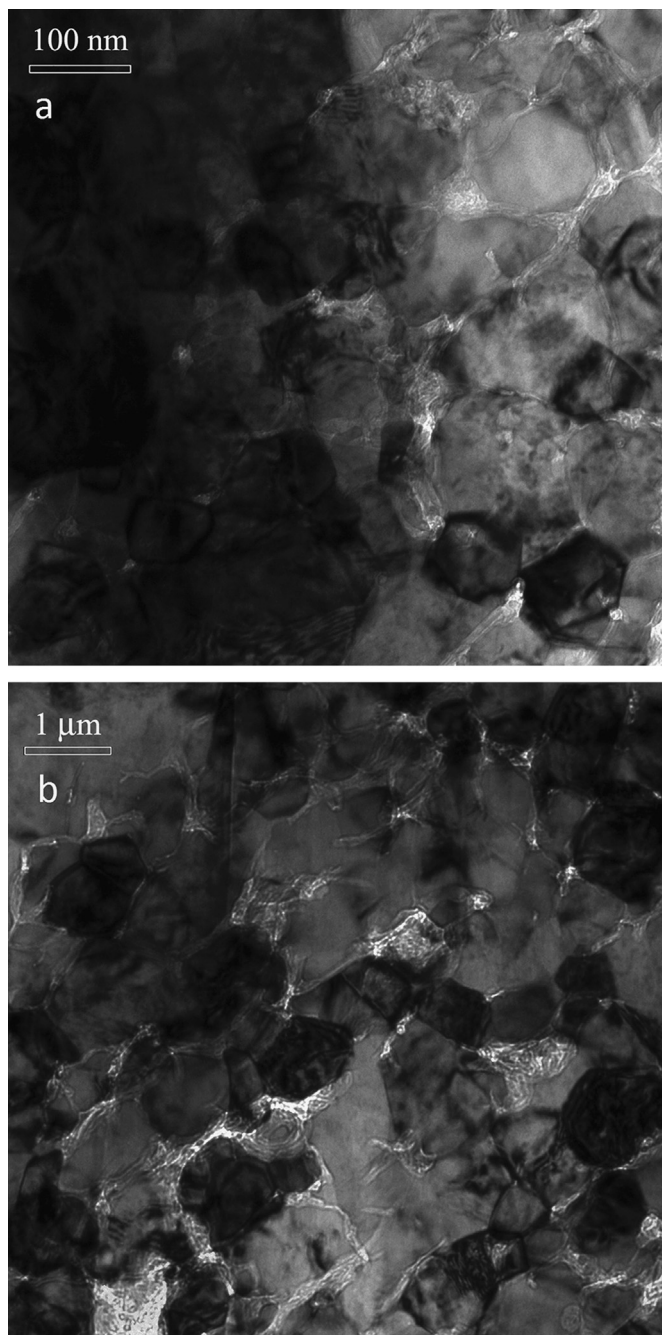


Fig. 2. HR-TEM figures of 3Y-TZP/5 wt% CNT composite microstructures after SPS at (a) 1350 and (b) 1500 °C.

prove this claim). On the other hand, the closed value of L-C fracture toughness compared to that of H-Z, at the first glance, may utterly question the idea of adding CNTs into the zirconia matrix. However, the essential effect of carbon nanotubes can be demonstrated when the H-C specimen has been highlighted to hit the remarkably high fracture toughness value of  $15.2 \pm 0.4$ . It is noteworthy that such incredible fracture toughness in H-C has been attained while the refining effect of CNTs is expected to limit the transformation toughening of zirconia matrix compared to H-Z (of-course to some extent). A major part of such an enhancement should be safely related to the presence of CNTs and the toughening mechanisms that

are activated as a result of the second reinforcing phase. It is needless to say that the aligned effects of transformation toughening and presence of CNTs in the H-C sample have led to an improved fracture toughness compared to the specimens in which either of these effects proposes the dominant role (L-C and H-Z).

In addition to the above advantages, the coincidence of aforementioned improvements in room and high temperature mechanical properties of CNTs/zirconia composites may propose another evidence to justify the addition of CNTs in spite of all difficulties within the dispersion and sintering stages of these materials. To investigate the high temperature mechanical properties of the composites, Mechanical Spectroscopy could be regarded as a convenient option due its non-destructive nature. This method has been usefully applicable for investigating significant features such as local distortion of lattices (point defect relaxation), dislocation motion, grain-boundary (GB) migration as well as the effect of precipitates and structural defects (for example point defects) on the motion of dislocations and GB sliding [34].

Fig. 3 illustrates variations of mechanical loss,  $\tan(\phi)$ , as a function of temperature for all classes of tested composites. As previously observed in monolithic zirconia-based composites, the spectra are characterized by an exponential increase in the mechanical loss at temperatures higher than a certain value. This temperature (starting point of exponential increase) is indirectly related to the onset of creep at low stress regime where the GB sliding accommodated by grain-boundary diffusion is considered as responsible mechanism of creep. Therefore, the delayed starting point for the abrupt change in the slope of H-Z diagram compared to L-Z should not be a surprising observation while the higher sintering temperature has generated a coarse grained microstructure for H-Z specimens. It has also resulted in decreasing the volume fraction of grain boundaries and consequently preventing the grain boundary sliding at high temperatures. In addition, for

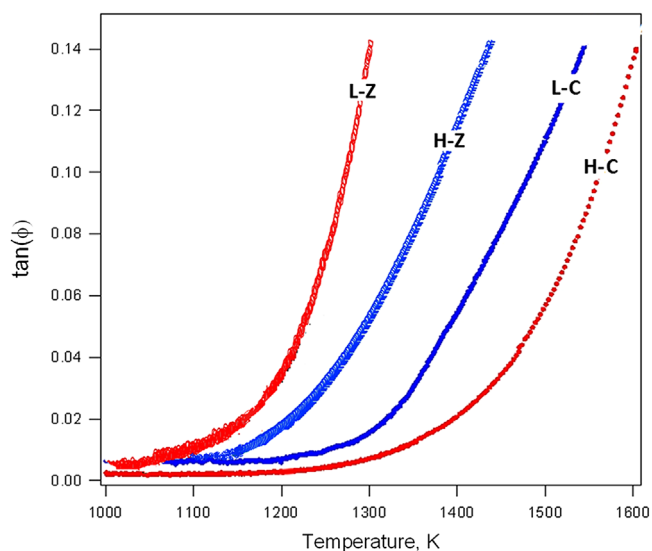


Fig. 3. Mechanical loss ( $\tan(\phi)$ ) as a function of temperature with measurement frequency of 1 Hz.

monolithic zirconia with fine grained structure (L-Z), this temperature is adjusted at approximately 1200 K. However, as Mazaheri [34] has shown, addition of nano-objects (e.g. CNTs), could increase this temperature to 1300 K. These reports are in a perfect accordance with the results of our experiments, in which the abrupt decrement of  $\tan(\varphi)$  in L-C diagram is conspicuously shifted to the higher temperatures compared to the monolithic zirconia samples (L-Z and H-Z). To distinguish the effect of carbon nanotubes from the grain growth, one should consider the clear shift in the onset point (abrupt increase of  $\tan(\varphi)$ ) of L-C diagram to the higher temperatures, compared to H-Z. Such a behavior implies the dominant role of carbon nanotubes in generating a creep resistant composite compared to a coarse grained monolithic zirconia based material. While the higher sintering temperature of H-Z specimens has resulted in an obvious grain coarsening and consequently a relative creep resistance due to the suppression of grain boundary sliding, presence of carbon nanotubes within L-C composites and preventing the movement of grain boundaries on the account of CNTs' dragging effect should be still regarded as the dominant factor. However, the onset point of creep, interestingly, shifts to even higher temperatures, i.e. 1400 K for H-C composites. Similar to the mechanical properties at low temperatures, the aligned effect of grain coarsening and adding CNTs at the final class of composites (H-C), should be still expected to propose the most successful results of creep resistance at high temperatures. This can be appreciated as another evidence to prove our success in combining the above effects for a better applicability of zirconia based composites.

#### 4. Conclusions

Among the conclusions of the present research, one can outline the following statements:

- 1) Contrary to the majority of research projects performed on ceramic composites incorporated with CNTs, the present research was successful to obtain an adequate dispersion of CNTs within zirconia matrix in high volume fractions up to 12.5 vol%, in a dry environment. Dispersion of carbon nanotubes has been severally reported as an absolute failure even in a wet environment and for much lower volume fractions.
- 2) High/Room temperature mechanical properties of Zirconia based materials were investigated considering two approaches: (1) increasing sintering temperature for monolithic zirconia ceramics and (2) addition of carbon nanotubes as the secondary reinforcing element to the zirconia matrix.
- 3) According to the results, increasing the sintering temperature could result in a coarse grained microstructure which led to enhanced mechanical properties at both room and high temperatures.
- 4) The addition of carbon nanotubes was found to propose a dragging effect on the grain boundaries which resulted in a remarkable creep resistance and prevention of grain boundary sliding as the dominant factor to render creep at high temperatures.
- 5) As the most essential achievement, the present research was successful to attain the highest values of mechanical properties at room and high temperatures by the alignment and combining the effects of carbon nanotubes and grain coarsening using a higher sintering temperature.

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