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# On the Performance of Carbon Nanotubes on Sintered Alumina-Zirconia Ceramics

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## Abstract

The alumina ( $\text{Al}_2\text{O}_3$ ) and zirconia ( $\text{ZrO}_2$ ) ceramic monoliths and their combination are used in both technical and biomedical applications due to their combination of excellent chemical, physical, and mechanical properties. Pressureless sintering (PLS), reaction bonding (RB), hot pressing (HP), hot isostatic pressing (HIP), and spark plasma sintering (SPS) are the sintering methods more commonly used. The high brittleness, the low fracture toughness, and low thermal stability that possess these ceramics are its Achilles heel for numerous engineering applications. The incorporation of a second phase such as carbon nanotubes (CNTs) into the ceramic matrix has been attempted to overcome these drawbacks but the obtained results are still controversial considering that the homogeneous dispersion of CNTs and the interfacial bonding between two different ceramic materials remains as a difficult task leading to little or even no improvement in mechanical properties. Besides, the role of CNTs in the sintering of ceramic materials is not clear in the scientific literature taking into account parameters such as materials used and particularly inconsistencies in dispersion and mixing of the CNTs. We discuss how the CNTs can affect the sintering behavior and microstructural evolution of alumina and zirconia ceramics and the combination of them.

**Keywords:** alumina, zirconia, carbon nanotubes, sintering, fracture toughness

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## 1. Introduction

Carbon nanotubes (commonly abridged as CNTs) are structures of nanometric dimension built up entirely by atoms of carbon and they have a high Young's modulus with good flexibility and

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good thermal and chemical stability being visualized as a graphene sheet that has been rolled into a tube with hemispherical caps at one or both ends [1, 2]. It is well known that the  $sp^2$ - $sp^2$  covalent carbon-carbon bonding is one of the strongest existing in nature, which in turn leads to exceptional material properties as a consequence of their symmetric structure. In the scientific literature, many researchers have reported mechanical properties of CNTs that exceed those of any previously existing materials [3] attracting an intense interest from the scientific community, as well as from industry. Nanotubes along with graphene are currently the subject of several papers per day. Undoubtedly, the discovery of CNTs [4] has aroused greatest interest as a potential reinforcing agent for different composite materials [5, 6] in order to impart stiffness, strength, and toughness considering their outstanding intrinsic physical properties and low density.

Taking into account that ceramic oxides such as aluminum oxide ( $Al_2O_3$ ), zirconium oxide ( $ZrO_2$ ), and a combination of them (alumina toughened zirconia (ATZ) and zirconia toughened alumina (ZTA)) are used in both structural and biomedical applications, it is of paramount importance to impart in these composites outstanding mechanical properties. In spite of combining the high strength and toughness of the tetragonal zirconia with the excellent hardness of the alumina ceramic, the low fracture toughness of brittleness of the alumina-based ceramics is still the main issue [7]. To overcome this weakness, the reinforcement in oxide ceramics can be carried out by short and long fibers. However, the CNTs due to their outstanding properties are fascinating materials as a reinforcement agent [5] and commonly two major structural forms of CNTs are known to exist as follows: single walled carbon nanotube (SWCNT) bundles and multi-walled carbon nanotubes (MWCNTs) [8]. Some experimental measurements have indicated that SWCNTs have Young's moduli ranging from 1 to 5 TPa, meanwhile MWCNTs have an average value of 1.8 TPa and a very similar relative density value [9, 10].

Nonetheless, the reduced size and dimensionality of CNTs lead to form complex networks of aggregates and bundles within ceramic composites [11]. In this context, this aggregation state plays an important role in defining the mechanical, electrical as well as thermal properties of the ceramic composites.

## 2. CNTs into the ceramic matrix during sintering

Although the role of CNTs in the sintering and microstructural evolution of ceramic composites is not completely clarified in the literature, these can be processed using the regular processing route and then densified mainly by pressureless sintering (PLS), hot pressing (HP) sintering, and spark plasma sintering (SPS) [12, 13]. Regardless the sintering method used, the scientific literature has reported significant improvement of mechanical properties but the results obtained with CNTs reinforced alumina- and zirconia-based ceramics remain controversial and can be observed that the wide scattered and highly debatable could be arise from different testing techniques used [3]. Unfortunately, today there is very limited experimental data on whether the final distribution of CNTs within microstructure of composites is mainly achieved during the powder phase processing or the sintering process [14]. Two main challenges in the processing of CNTs as a reinforcement agent in ceramics remain the heel of Achilles: a homogeneous dispersion of CNTs in matrix materials and the interfacial bonding

between the two different materials (CNTs and ceramic matrix) [5, 11]. In spite of this, many interesting works have been carried out to improve the mechanical properties of ceramic composites. Indeed, Zhan et al. [15] prepared 100% dense  $\text{Al}_2\text{O}_3 + 10 \text{ vol}\%$  SWCNTs at  $1150^\circ\text{C}$  during 3 min with SPS as densification method obtaining a fracture toughness of  $9.7 \text{ MPa m}^{1/2}$  being nearly three times that of nanocrystalline alumina ( $3.5 \text{ MPa m}^{1/2}$ ); however, the results have not been reproduced up to now. In fact, these results were refuted by Wang et al. [16] who reported that CNT-alumina composites are highly contact damage-resistant and also showed that a more reliable single edge V-notched beam test could reveal no enhanced toughening, refuting therefore the claims of high toughness by Zhan et al. [16] in reference to the fracture toughness technique used. In other ceramic system prepared by HP, a mixture of MWCNTs and nano-SiC powders were reported by Ma et al. [17] being the dispersion of the MWCNTs very poor. However, an increase in both the bending strength and fracture toughness was obtained with a carbon nanotube content of around 10 vol%.

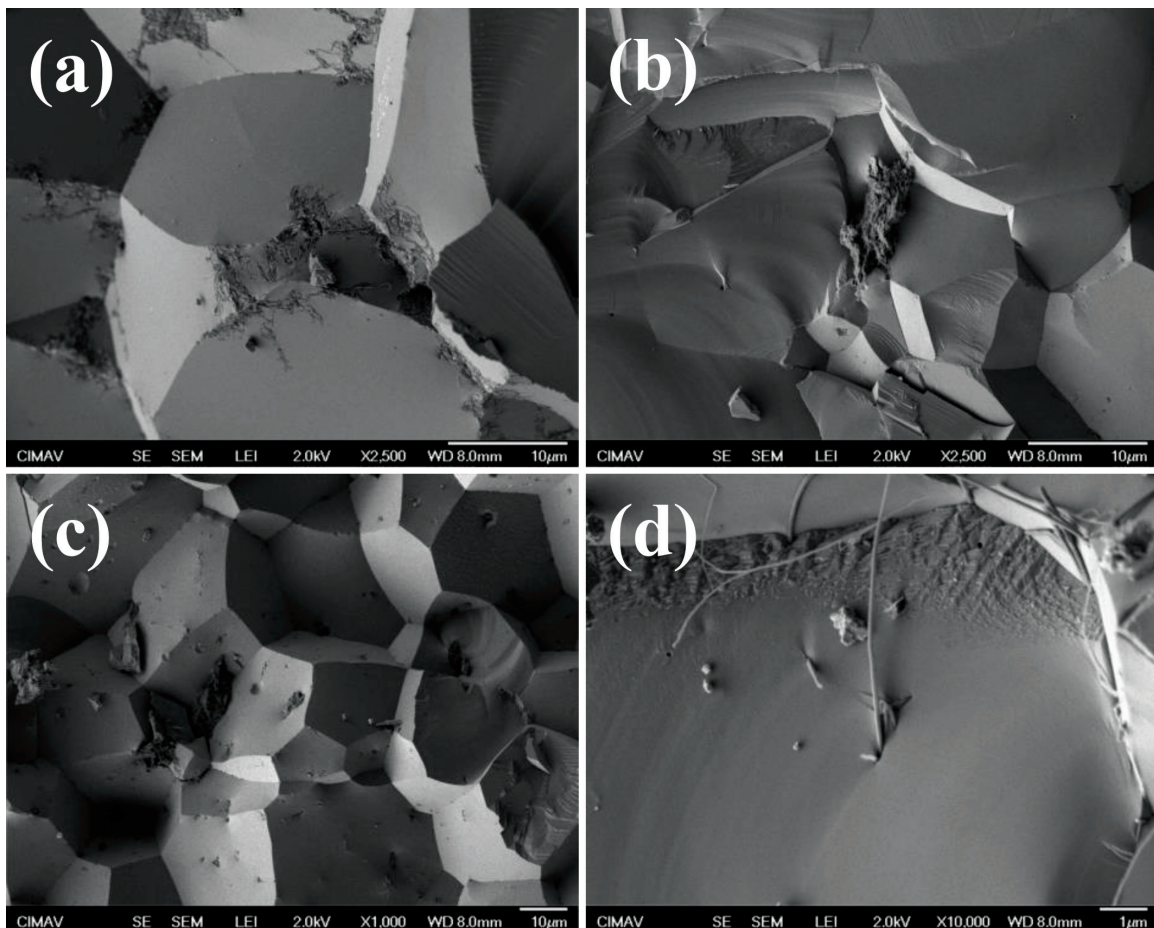
The above mentioned are few examples where both SWCNTs and MWCNTs have been used as reinforcement agents and the results have been some controversial and contradictory. To reinforce ceramic matrices, there are different kinds of CNTs available and there has been much documented research reporting the incorporation of SWCNTs and MWCNTs into the ceramic matrices in order to convert them into tough, strong, electric, and thermal conductive materials [18, 19]. Indeed, approximately 88% of the reported cases used the readily available and economically feasible MWCNTs as a reinforcement agent in comparison to SWCNTs. Regardless of this, it is noteworthy that the problems to achieve homogeneous distribution of CNTs as well as the related problems to the reproducible preparation of ceramic composites with improved mechanical properties can be considered as key obstacles. This difficulty to disperse CNTs into the ceramic matrices has led to explore and to develop efficient and economical processing methods that enable homogeneous dispersion of different types of CNTs in ceramic hosts. Those dispersion methods are referred to colloidal, sol-gel, and electrophoretic deposition processing techniques that are of paramount importance as methods to directionally emplace the CNTs while reducing the energy demands for the manufacture of the final product. Nonetheless, nowadays the main attention is paid to ultrasonic, plasma techniques, and other physical techniques in combination to the use of surfactants, functionalizing, and debundling agents of distinct nature including elemental substances, metal and organic salts, mineral and organic acids, oxides, inorganic and organic peroxides, organic sulfonates, polymers, dyes, natural products, biomolecules, and coordination compounds in order to produce ceramic nanocomposites with excellent mechanical properties [20].

Although currently there are several processes to manufacture CNTs containing alumina- and zirconia-based ceramic nanocomposites, some of them with a range of controversial results, it is evident that MWCNTs are preferably used as the reinforcement compared with SWCNTs [18, 21]. With the purpose to achieve nanostructures with outstanding mechanical properties, undoubtedly the nature of the different available CNTs and their processing conditions are of paramount importance and must therefore be considered. Commercially a great variety of CNTs are available with different conditions of preparation and subsequent treatment, which in turn and considering the sintering route adopted for the densification of the ceramic composite, could be the main reason for the controversy of results on their mechanical properties.

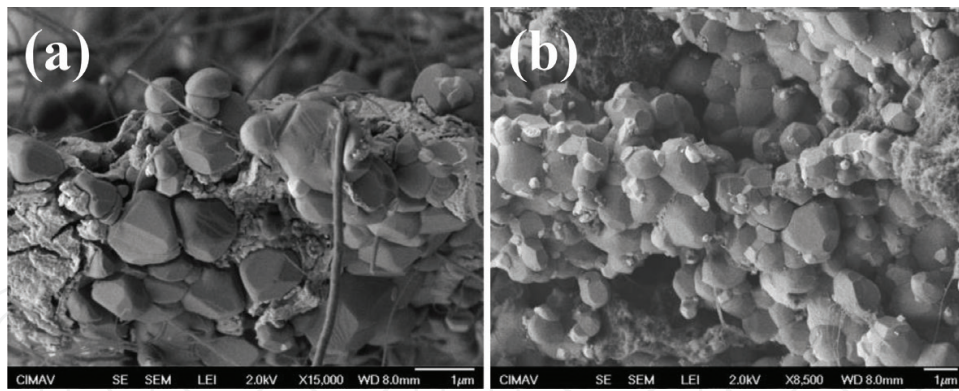


Under this assumption, it could be asserted that the different types of CNTs and the amount of these, added to the ceramic matrix, can lead to strong variations of the mechanical properties and affinity to the ceramic matrix as a consequence of their tubular structure, the number of the roller graphene sheets, diameter, length and their crystallinity linked to the number and nature of surface defects, and surface chemistry [23, 34]. All these aspects in turn will produce microstructural variations and fracture behavior of ceramic nanocomposites manufactured under different densification methods even with the same percentage of CNTs content within the ceramic matrix and using the same sintering technique. **Figure 1** shows fracture surfaces corresponding to an alumina ceramic with additions of 0.1 wt% of four different kinds of MWCNTs and sintered by SPS at 1500°C 3 min.

Knowing that the CNTs addition significantly retards grain growth during sintering [16, 19, 22, 25, 26] due to the pinning of matrix grains by the presence of CNTs, it is curious to observe from **Figure 1** the growth grain in alumina for low CNT contents and dispersed into the ceramic matrix under same conditions. These results reflect the unexpected effect that CNTs



**Figure 1.** Scanning electron microscopy (SEM) micrographs of fracture surfaces of Al<sub>2</sub>O<sub>3</sub> with additions of 0.1 wt% of four different kinds of MWCNTs. (a) MWCNT 1, outer diameter 50–80 nm, length 10–30 μm; (b) MWCNT 2, outer diameter 10–20 nm, length 10–30 μm; (c) MWCNT 3, outer diameter < 8 nm, length 10–30 μm; and (d) MWCNT CIMAV, outer diameter 10–70 nm, length 120–160 μm sintered by SPS at 1500°C 3 min. Figures (b) and (c) from the paper: SWCNTs versus MWCNTs as reinforcement agents in zirconia- and alumina-based nanocomposites: which one to use, Bocanegra-Bernal et al. [24]. © 2016 Scrivener Publishing LLC. With permission.



**Figure 2.** SEM of fracture surfaces of ZTA with additions of (a) 10 vol% MWCNT, and 10 vol% SWCNT (b), sintered at 1520°C during 1 h, showing agglomeration of SWCNT in (b).

could have on the microstructural evolution, affecting mainly the mechanical properties what contrasts with the results reported in the literature [11, 27–29]. On the other hand, it is expected that low levels of CNTs can be easier to disperse into the comparison with high levels of them. Likewise, the presence and distribution of the CNTs within the ceramic matrix induces a combination of fracture mode in each composite where the CNTs as the second phase could be responsible for altering the fracture modes [18]. Conversely, high levels of both MWCNTs and SWCNTs induce to an undesirable agglomeration state which is detrimental to achieve high densification by the formation of clusters owing to van der Waals forces [30] and the concentration of reinforcement at certain point leading to worsening of overall mechanical properties [11] such as can be illustrated in **Figure 2** for zirconia toughened alumina (ZTA) with additions of 10 vol% of CNTs.

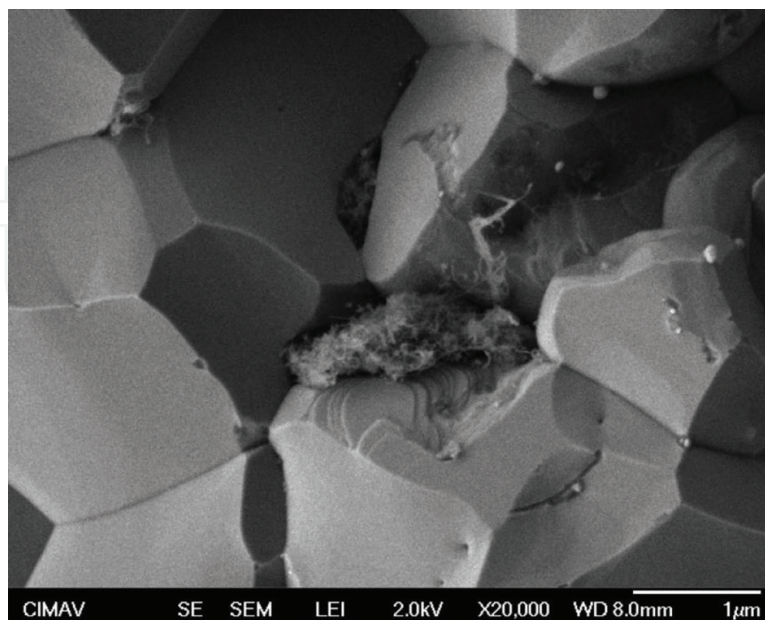
For the same nanotubes content in **Figure 2**, generally the MWCNTs can be homogeneously dispersed (**Figure 1a**) meanwhile denser agglomerates of SWCNTs were formed (**Figure 2b**). Similar observations have been reported by Zhang et al. [31] in CNTs- $\text{Al}_2\text{O}_3$  with variable CNT content and Zhang et al. [32] in alumina ceramics with additions of 1, 3, and 5 vol% MWCNTs. In view of experimental evidences, such as **Figure 1** [33] and **Figure 2** [34], and other more reported in the scientific literature (for example Tables 1 and 2 in Ref. [18]) showing a broad spectrum of final densities and mechanical properties using different methods of purification and dispersion of CNTs into the several ceramic matrix systems, a too high CNT content must be avoided with the purpose to achieve a good dispersion by means of conventional and economic methods, since the use of sophisticated techniques can make the process expensive and its industrial scaling more difficult.

### 3. Requirements for optimum performance of CNTs as reinforcement agents in ceramics

Taking into account that intrinsic factors of CNTs such as diameter, length, nature of surface defects, orientation, mechanical strength, and affinity with the ceramic matrix have a strong influence on the microstructure and grain growth of the ceramic composites, the effective and optimum

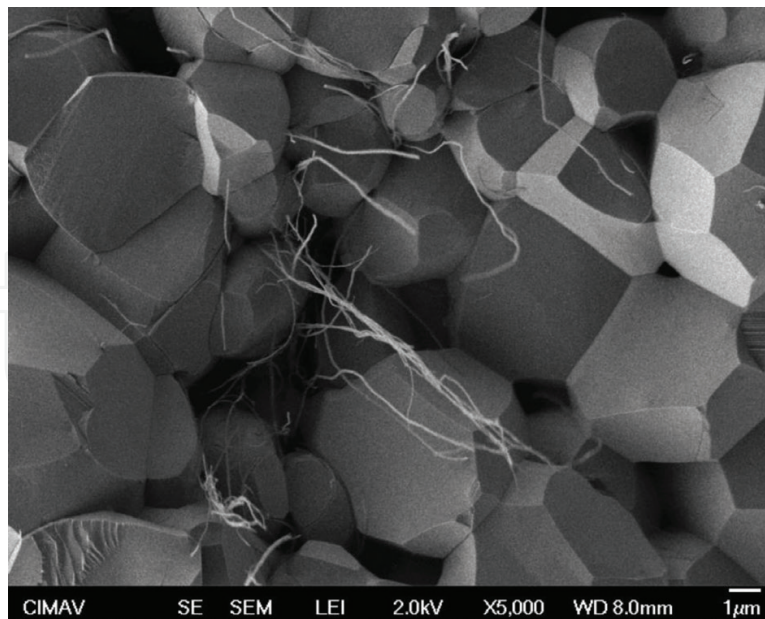
utilization of CNTs in composite applications depends strongly on the ability to disperse CNTs homogeneously throughout the matrix to obtain a good interfacial bonding which is required to achieve an efficient load transfer across the CNT-matrix interface as a primary condition for improving the mechanical properties of ceramic composites [35]. As a consequence of high van der Waals force, surface area and high aspect ratio of CNTs (most notorious in SWCNTs), inevitably self-aggregation occurs and therefore, the improvement of dispersion has become a challenge to maximize the properties of CNTs [36, 37]. **Figure 3** shows the fracture surface of alumina with additions of 0.5 wt% of SWCNTs where bundles of CNTs located intergranularly are evident impeding the densification of the ceramic composite during the sintering at high temperatures. Nevertheless, the characterization of the dispersion of CNTs within the microstructure in the sintered composites is often based on the visual observation of micrographs obtained from scanning electron microscopy (SEM). It is of paramount importance to quantify the quality of distribution of CNTs in the microstructure of the sintered samples to understand the broad properties that the CNTs can offer as reinforcement agents in the ceramic nanocomposites [14, 30].

Regardless of dispersion method used, it is indisputable that the quantity, location, and distribution of CNTs in the ceramic matrix play an important role in the sintering of the composites producing compounds with a varied range of mechanical properties stressing that the dispersion of CNTs is not an easily controllable and reproducible process and, therefore, that the final properties can depend in first instance on the route followed for their dispersion into the ceramic matrix, as well as the sintering route chosen. **Figure 4** illustrates the fracture surface of an alumina ceramic doped with 0.5 wt% of MWCNTs that are located parallel to the fracture surface indicating a poor bonding to the ceramic matrix not contributing to the improvement of the fracture toughness by the absence of toughening mechanisms such as crack branching, pull out, and crack deflection.



**Figure 3.** Scanning electron microscopy (SEM) micrograph of fracture surfaces of  $\text{Al}_2\text{O}_3$  with additions of 0.5 wt% of SWCNTs sintered at  $1520^\circ\text{C}$  under atmospheric pressure with graphite powder as powder bed.





**Figure 4.** Scanning electron microscopy (SEM) micrograph of fracture surfaces of Al<sub>2</sub>O<sub>3</sub> with additions of 0.5 wt% of MWCNTs sintered at 1520°C under atmospheric pressure with graphite powder as powder bed. Note the poor bonding of the CNTs to the ceramic matrix affecting the mechanical properties.

Recent work [38] reports for a same content of CNTs (0.1 wt%), a wide range of grain sizes and fracture toughness values, with the hardness remaining practically the same in alumina ceramic composites prepared by PLS, hot isostatic pressing (HIP), and sintering + hot isostatic pressing (sinter + HIP) routes. In all cases, the dispersion of CNTs and mixture preparation was performed under same conditions and the substantial difference observed in the final results could be explained by the different sintering kinetics of the three techniques applied in that work [38]. According to Orsolya [14], at temperatures higher than 1500°C and long sintering times, large amount of mass diffusion takes place facilitating a significant rearrangement of the nanotubes as well, while for SPS the sintering is completed in short time, where it is expected less and most likely short range rearrangements of the CNTs aggregates formed during the powder phase processing. However, it is well known that PLS and HP are the techniques that commonly require high temperatures, which could induce in some cases to the partial destruction of CNTs at these temperatures. Based on this, these sintering techniques have been replaced long ago by SPS, due to the damage of the CNTs by the higher temperatures and longer sintering times attained in those conventional methods.

Considering the pros and cons of the different sintering methods used in the manufacture of ceramic nanocomposites, undoubtedly that SPS so far only facilitates the fabrication of simple geometries such as discs, rings, and cylinders, but the manufacture of more complex geometries is still in the development stage implying that on an industrial scale, PLS remains as the main sintering method adopted when complex geometries must be manufactured, mentioning that this method has been continuously perfected to obtain the best properties in the final compounds without compromising the integrity of nanotubes at high temperatures. Thus, intimate interfacial bonding between CNTs and ceramic matrix by an optimum dispersion of CNTs to achieve toughening of ceramic nanocomposite by a specific sintering method are the main key points for preparing CNTs reinforced oxide ceramics [39].



## 4. Conclusions

Many attempts have been made to improve the mechanical properties of ceramics through incorporating CNTs taking advantage of its mechanical and physical properties combined with their low density and judging from the results of several researchers, where unfortunately most of them have been disappointing for toughening, since very little or no increase in toughening upon introduction of either single- or multi-walled carbon nanotubes into alumina-zirconia-based ceramics has been shown. During the last decade, the CNTs and their processing and dispersion methods have been intensively studied. However, the controversial results reported could be arise from different dispersion techniques, sintering processes, and finally testing techniques used for their characterization. On the other side, there is the controversy to choose between the different kinds of CNTs to reinforce ceramic matrices to improve the mechanical properties. Another debatable question is to define the proper amount of CNT content to obtain a ceramic composite with improved mechanical properties, considering that even using the same contents (vol% or wt%) as well as the same type of CNTs into the ceramic matrix, the expected values of fracture toughness, and/or hardness could differ from each other favorably or unfavorably, particularly for higher concentrations of CNTs. Therefore, the meticulous selection of a specific kind of CNT as a reinforcement agent for a determined ceramic must be carried out after experimental trials, since the prediction of results starting from raw material known such as ceramic powders and CNTs (even knowing its diameter, length, and agglomeration state) is not an option, considering as above mentioned that dispersion of CNTs is nowadays considered by a sector of scientific community as a process very difficult to control and to reproduce and with base of this, the final properties will be different for each specific ceramic composition.

Concluding, the development of CNT-based ceramic nanocomposites is a promissory subject but still with many difficulties and challenges and there is a lack of sufficient knowledge to systematically improve properties over traditional ceramic composites or their monoliths with notable enhancements. As can be pointed out by Curtin and Sheldon [40], *“the traditional interplay of careful processing and evaluation, coupled with mechanistic assessment of properties, remains a valid paradigm at the nanoscale and should be assiduously applied to future research in CNT-composite systems.”* In other words, the optimum dispersion of CNTs into liquids could be achieved by mechanical (physical) or chemical methods, taking into account the amount and the type of CNTs to be dispersed as well as the optimal concentrations of aqueous surfactant solution and sonication time to contribute to the efficiently dispersion of CNTs.

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## Conflict of interest

The authors declare that there is no conflict of interest.

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