

Zirconia-Based Ceramics Reinforced by Carbon Nanotubes

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The use of CNTs (carbon nanotubes) as a secondary phase in a zirconia matrix is motivated by their outstanding crack self-healing ability, the possibility to tailor the desired nano-structural properties, and their exceptional wear behavior. Therefore, a detailed investigation into CNT features has been provided. The debate of using the different Vickers indentation fracture toughness equations to estimate the resistance of crack propagation was critically reviewed according to crack characteristics.

Keywords: CNT-zirconia composites ; spark plasma sintering ; ball milling ; indentation fracture toughness

1. Introduction

Ceramic-matrix composites (CMCs) afford a new generation of technical applications with excellent efficiency due to their great potential and unique properties. CMCs are known to be robust, lightweight, and highly wear-resistant, which makes them a proper class of materials in automotive applications (e.g., oxygen sensors and brake systems), aerospace engineering (e.g., hot structures), and remarkable tribological applications ^{[1][2][3]}. Ceramics and their composites have shown great advances in most tribological applications and have replaced metals and conventional materials, such as grey cast iron, in the fabrication of brake systems. High wear resistance combined with a low friction coefficient are desirable for reducing the losses in the movement of rotating parts and increasing the lifetime of components, respectively. A wide range of factors, including the applied load, sliding speed, time of contact, temperature, lubrication, and surface characteristics, such as composition and roughness, influence the wear properties of CMCs.

CMCs also allow for improving the mechanical behavior of conventional polycrystalline ceramics, which typically suffer from high brittleness and low fracture toughness. In general, CMCs are produced from at least one micro-/nano-sized reinforcing/toughening phase embedded in a ceramic matrix ^[4]. The second phase can be constituted by ceramic micro- or nanoparticles (SiC and Si₃N₄), nanotubes (e.g., CNTs), nanoplatelets (graphene), or hybrids of these materials.

Niihara et al. were the first to introduce the concept of ceramic nanocomposites in 1991 ^{[5][6][7][8]}. Their work mainly dealt with SiC nanoparticles' incorporation as a second phase in Al₂O₃, Si₃N₄, and MgO ceramic matrices. After they reported significant improvements, a series of research works succeeded in incorporating different nanoparticles (e.g., SiC, Si₃N₄, TiN, TiC, TiO₂, and ZrO₂) in various ceramic matrices, such as Al₂O₃, Si₃N₄, MgO, SiAlON, etc. Reinforcing ceramic matrices with CNTs has gained much interest since the first report by Iijima ^[9]. Owing to their one-dimensional nanostructure, CNTs provide exceptional mechanical, electrical (10⁷ S/m), and thermal properties (1800–6000 W/mK) compared with nanoparticles ^[10]. Hence, CNT addition can increase not only ceramic matrix toughness, but is also responsible for the improvement of its functional properties, namely its electrical and thermal properties.

In order to produce ceramic/CNT composites with improved mechanical and functional properties, appropriate interfacial bonding between the ceramic matrix and CNTs and the uniform dispersion of CNTs along the grain boundaries are required. Owing to their high aspect ratio, CNTs tend to agglomerate, leading to stress concentration and, therefore, the overall properties of the composites being decreased. The preparation process and the choice of an appropriate sintering technique play an important role in defining the final mechanical, as well as tribological and functional, properties of composites ^{[6][7]}.

ZrO₂-based nanocomposites are among the most widely investigated and technologically powerful ceramic materials involved in several fields. The important properties of ZrO₂, such as its high ionic conductivity, low density, chemical inertness, good wear resistance, high mechanical strength, and stability at high temperatures, have enabled its wide use in a range of challenging structural, tribological, and multifunctional applications ^{[7][11]}. Typical applications are designed to withstand harsh environments and to increase the worldwide clean energy demand, either by direct electrochemical

power generation, for example, solid-oxide fuel cell (SOFC) devices, or in an indirect way by serving as a thermal barrier coating in engines, thereby ensuring low-cost electrical energy for the customers due to the higher engine efficiency [12]. Other important applications are in the field of load-bearing joint prostheses and dental implants [13], where the mechanical properties play a key role [14].

Yttria-stabilized ZrO_2 possesses the advantage of phase transformation from tetragonal to monoclinic under applied stress. This mechanism is usually referred to as transformation toughening in zirconia and occurs as the volume expansion associated with the tetragonal-to-monoclinic transformation produces a stress intensity decrease at the crack tip, thus yielding a dissipation of the energy of the propagating crack. Cracks become self-stopping as, in order to allow crack propagation, both the energy barrier required for phase transformation and that associated with the volume expansion of the transformed material have to be overcome [2][3]. However, monophasic and even stabilized ZrO_2 materials still exhibit brittle behavior, which constitutes a major issue [15]. To overcome this drawback, several attempts have been made to further improve the fracture behavior of ZrO_2 , for example, by introducing a second-phase material, while preserving all the other advantages of the matrix. In this regard, multi-walled carbon nanotube (MWCNT)-reinforced ZrO_2 composites are attracting growing interest owing to their crack self-healing ability and the possibility to tailor the desired nano-structural properties. Therefore, to completely benefit from their outstanding properties, a deep understanding of the material's behavior across the different length scales is required. Although great effort and progress have been made over the past ten years in the investigation of zirconia-based composites, only a few new material systems and novel processing techniques have been proposed. The high-energy ball milling process has been assessed for successfully synthesizing a wide range of nano-structured ceramic powders, including ZrO_2 . The easy operating principle uses the solid-state reaction at a temperature close to room temperature and affords low cost with an effective preparation process [16]. On the other hand, spark plasma sintering (SPS) has been emphasized by a large number of researchers to show minimal damage to CNTs and high efficiency in sintering nanostructured ceramic-based materials [9].

2. Zirconia Matrix

Zirconium dioxide (ZrO_2), also named zirconia, is the mineral of baddeleyite and is chemically inert. In its pure form, ZrO_2 exhibits a polymorphic configuration and exists in three distinct phases at ambient pressure. The monoclinic phase is stable from room temperature up to 1170 °C. Tetragonal ZrO_2 with a distorted fluorite structure is stable at intermediate temperatures between 1170 and 2370 °C, while the cubic fluorite phase is stable at very high temperatures from 2370 °C up to its melting point (2750 °C) [16][17]. The higher the temperature, the better the symmetry. The phase transition ability makes zirconia one of the most-studied ceramic materials over the last few decades and an important candidate for diverse areas of structural applications [18].

The structure of cubic zirconia can be represented by a face-centered cubic (FCC) structure similar to that of fluorite. The eight oxygen ions occupy the tetrahedral interstitial sites and are surrounded by Zr^{4+} cations, which occupy the summits of the cube and the centers of each face. The tetragonal phase of zirconia is represented by a straight prism with rectangular sides. Similar to the cubic structure, zirconium (Zr) ions are coordinated to eight oxygen atoms, and the oxygen atoms are coordinated to four Zr ions. However, the distorted shape of the tetragonal phase is linked to the alternative oxygen ions' displacement along the c-axis, resulting in the formation of two tetrahedron plans where one is elongated and rotated by 90° due to the different distances of each of the four oxygen ions from Zr^{4+} (2.065 and 2.455 Å, respectively) [19]. In its natural form, zirconia exhibits the monoclinic phase at low temperatures known as baddeleyite and contains about 2% HfO_2 (hafnium oxide), with high chemical and structural similarity to zirconia. In the monoclinic phase, Zr^{4+} ions are coordinated to seven oxygen ions residing in tetrahedral interstices. The Zr ions are coordinated to three and four oxygens at average distances of 2.07 and 2.21 Å, respectively. Therefore, one of the angles (134.3°) differs considerably from the tetrahedral value (109.5°) [20].

3. Carbon Nanotubes (CNTs): A Valuable Second Phase in Zirconia-Based Composites

The discovery of carbon in the form of graphite was achieved in 1779, followed by diamond 10 years later. These two enormous discoveries in the field of nanotechnology generally and carbon structure specifically stimulated researchers worldwide to increase their interest in finding other forms of more stable and structurally ordered carbon. In 1985, a new form of carbon known as fullerene was accidentally discovered by Kroto, Smalley, and Curl (Nobel Prize in Chemistry in 1996) [21]. The structure of fullerenes is almost similar to that of a single sheet of graphite (graphene), with a planar honeycomb lattice, in which each atom is attached to three neighboring atoms (hexagonal rings) via a strong chemical bond. However, fullerene sheets exhibit additional pentagonal or sometimes heptagonal rings and are not planar like graphene, and can also generate three-dimensional (3D) structures similar to a football.

A few years later, in 1991, the Japanese scientist Iijima [9] discovered multi-walled carbon nanotubes (MWCNTs) with an outer diameter ranging from 3 to 30 nm and at least two layers. Later, in 1993, he discovered a new class of CNTs, single-wall carbon nanotubes (SWCNTs). SWCNTs tend to be curved, rather than straight, with a typical diameter between 1 and 2 nm. CNTs are cylindrical fullerenes with a nanometric diameter and micrometer-scale length, leading to a high length-to-diameter ratio exceeding 10^7 . CNTs self-align into chains by van der Waals forces, where the carbon atoms are sp^2 -bonded with a length of approximately 0.144 nm. In MWCNTs, the interlayer distance between two successive CNTs is similar to the interspaces between two successive graphene layers in graphite (about 3.4 Å).

Since their discovery, MWCNTs have opened up an incredible range of promising applications in nanocomposites, nano-electronics, medicine, energy, and construction. Indeed, CNTs exhibit novel and unique properties, namely very high tensile strength (≈ 100 GPa) and Young's modulus (≈ 1500 GPa), high thermal conductivity and chemical stability, and excellent electrical conductivity similar to those of silver and platinum [6][22][23].

4. Processing Techniques Used to Produce CNT-Reinforced Zirconia-Based Nanocomposites

Different processing routes have been proposed over the last few years for the preparation of CNT/ceramic-matrix composites, as comprehensively reviewed by Chan et al. [24]. The techniques may greatly vary depending on the type of ceramic used, but, in general, a sintering step is always necessary to consolidate the final product. Sintering may be combined or not with a pressing stage; recently, spark plasma sintering (SPS) has attracted significant interest to reduce the risk of thermal damage to CNTs owing to the low temperature required for sintering and short sintering time.

In the case of CNT/zirconia composites, processing is typically based on ball milling and SPS; therefore, the focus is on these methods. Other strategies, which have not been specifically applied to producing CNT/zirconia composites so far, but that could be of interest for future research on this topic, are shortly mentioned in a dedicated subsection.

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