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Review Article

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Recent advances in carbon and ceramic composites reinforced with nanomaterials: Manufacturing methods, and characteristics improvements

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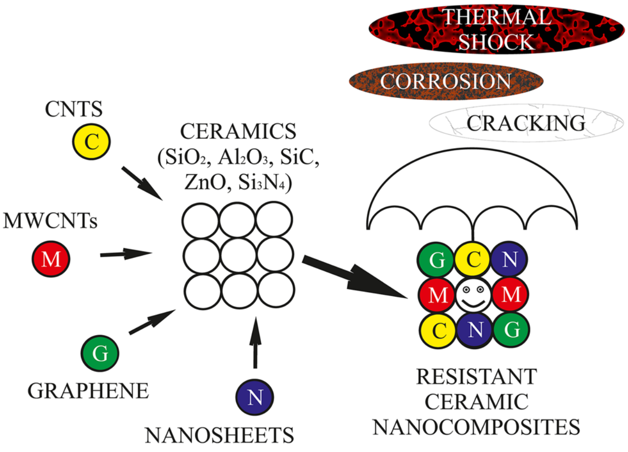
Abstract: Recent years have witnessed a considerable increase in the production of novel types of nanomaterials and nanomaterial-modified matrices and surfaces. This article highlights the latest achievements (predominantly over the past 5 years) in the properties and manufacturing methods of nanostructured composites, with a particular focus on carbon and ceramic materials. The key parameters determining the effectiveness of nanofillers for reducing brittleness and increasing corrosion resistance, including that at high temperatures, in ceramic composites are discussed. Interphase effects play a decisive role in shaping the properties of nanocomposites. Research into the relationship between the structure of composites, particularly those based on ceramic materials as a matrix, and the addition of carbon nanotubes, is promising. Thus, it has been shown that carbon nanomaterials significantly reduce the brittleness of ceramic and carbon composites, increasing their strength and durability.

Keywords: ceramic matrix composites, nanoparticle dispersion, nanofillers, carbon materials, carbon nanotubes, mechanical properties, dispersion assessment methods, crack resistance



Graphical abstract

1 Introduction

Nanotechnology studies the fundamental properties of substances that are smaller than 100 nm in at least one of three dimensions; this field of science is related to physics, biology, and chemistry. The upper limit of the size of nanomaterials or nanosystems is the point beyond which further increase does not lead to a change in properties. Morphologically, nanomaterials can be films, rods (tubes, wires), quantum dots, *etc.* Nanoscale dimensions cause changes in the physical and chemical properties (electrical conductivity, magnetism, ability to absorb and emit light, optical refraction, thermal stability, strength) of materials and lead to catalytic activity or reactivity that are not observed in macro- and microscopic bodies of the same chemical nature [1,2]. From a qualitative point of view, the special properties of nanomaterials are associated with both the unusually developed surface of the particles and the electronic and quantum effects inherent in these nanoparticles.

Various nanoscale materials are used as fillers to improve the properties of polymer (thermoplastic, elastomeric), ceramic or metal matrices [3–5]. The reason for preferring nanofillers to macrofillers is that nanofillers not only improve the desired mechanical properties of composites, but also improve their functional properties,

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such as self-lubricating surfaces [6]. Composites consist of two phases: matrix and reinforcement. It has been demonstrated that the introduction of nanofillers in various ratios improves the structural or functional properties of matrices, such as mechanical, thermal, electrical, electrochemical, and others [4,7,8]. In this case, nanoparticles are used as connecting links between molecular structures in bulk materials [9]. A particularly important aspect is the use of solid micro- and nanofillers as compatibility enhancers or to increase the strength and flexibility of matrices when only a limited amount of filler can be used.

Obtaining new types of materials with advanced performance characteristics is important for the development of the aviation industry, energy, construction, and biomedical technologies [10–13]. Numerous studies have shown improvements in the mechanical properties of composite materials through the introduction of different forms of carbon materials [14–19] (Table 1).

However, this area remains insufficiently studied. The main problems arise from the inherent characteristics of carbon nanotubes (CNTs) as nanofillers, in particular their strong tendency to aggregate and their flexibility, which often leads to pronounced bending of nanotubes during the manufacture of polymer nanocomposites [20]. A similar effect is also known for composites containing metal wires, where it has a significant impact on the properties of the material [20]. The effectiveness of mechanical reinforcement of ceramic composites with carbon allotropes remains a subject of debate, due to the heterogeneity of carbon nanomaterial dispersion, weak adhesion at the phase boundary between carbon allotropes and ceramic matrix grains, and the location of carbon allotropes at grain boundaries [21,22]. In this regard, the analysis of recent studies presented in this article, which addresses the aforementioned shortcomings, is highly relevant. A large number of literature reviews are devoted to polymer and/or metal matrices reinforced with carbon nanomaterials, and there are not enough publications on changing the properties of ceramic matrices using CNT.

Since composite manufacturing is an important area of materials science and mechanical engineering, it is relevant to analyze the various components of matrices and fillers. For example, Al–Si alloy is widely used in mechanical engineering to manufacture parts such as pistons and cylinder liners due to its high strength. The size of silicon particles in different areas of the casting system varies significantly, leading to significant variability in the mechanical properties of the sample [23]. Primary silicon crystals can be purified to increase wear resistance and hardness. For example, it has been found that silicon carbide (SiC) particles synthesized *in situ* play a positive role in increasing hardness, density, wear resistance, and thermal expansion coefficient. In addition, the ultimate tensile strength of a SiC-reinforced alloy can be increased to 132 MPa at a test temperature of 350°C [24]. It has been demonstrated that when the content of SiC nanoparticles is increased from 1 to 10 vol%, the yield strength and tensile strength increase from 296 and 343 MPa to 545 and 603 MPa, respectively [25]. It has been reported that the elastic modulus of a composite reinforced with 10 vol% SiC nanoparticles is 97.1 GPa, which is significantly higher than that of pure aluminum (72.6 GPa) [26]. The strengthening effect of SiC nanoparticles is reduced due to their aggregation in the metal matrix [17]. In addition, fully dense composites (titanium boride [TiB_2] + SiC) reinforced with Ti_3SiC_2 containing 15 vol% TiB_2 and 0–15 vol% SiC were obtained by *in situ* hot pressing. The increase in SiC content contributed to compaction and significantly suppressed the growth of Ti_3SiC_2 grains. It was found that the maximum flexural strength of such composites is 881 MPa, and the fracture toughness is 9.24 MPa at 1/2 at 10 vol% SiC. The Vickers hardness of the composites increased monotonically from 9.6 to 12.5 GPa [27]. In the work of Li *et al.* [28], composites made of ZA27 alloy reinforced with Ti_3SiC_2 were synthesized by hot pressing mechanically alloyed mixtures of Ti_3SiC_2 and ZA27 powders. It was shown that composites containing 20% Ti_3SiC_2/ZA27 have the highest tensile strength at room temperature, flexural strength,

Table 1: Comparison of mechanical properties of composite materials

Materials	Experimental technique	Young's modulus	Reference
Monolayer graphene	Nano-indentation in AFM	1 TPa	15
Chemically rGO sheet	An AFM tip-induced deformation	250 GPa	16
Monolayer GO	AFM imaging contact mode	207 GPa	17
Free standing GO	Nano-indentation on a dynamic contact tool	697 ± 15 GPa	18
Graphene paper	—	31.7 GPa	19
Graphene fiber (hybrid of graphene ribbons and sheets)	Tensile tester at a strain rate of 1 mm min^{-1} with a gauge length of 5 mm	—	20

and Vickers hardness, equal to 339 MPa, 593 MPa, and 1.13 GPa, respectively. The improved mechanical properties of the Ti_3SiC_2/ZA27 composite are mainly due to the effects of fine-grained and dispersion strengthening [28,29]. Friction stir processing (FSP) was used as a method for producing Al 7075 composites (SCs) doped with three different nanoparticles: multi-walled carbon nanotubes, graphene, and titanium dioxide [30]. All SC samples demonstrated uniform dispersion of filler nanoparticles in their microstructure. The increase in the hardness and strength of the composites was due to grain refinement and the pinning effect, which was accelerated by the circulation of the cooling liquid. The hardness values in SCs increased by approximately 95–110%. The wear rate in SCs also decreased due to the increase in hardness and the formation of a lubricating coating in the case of carbon-containing composites. In addition, scanning electron microscopy demonstrated that three different phases can be distinguished in the composite containing 10% SiC, namely, the white phase Ti_3SiC_2, the non-white phase TiB_2, and the dark phase SiC. The scanning electron microscopy (SEM) tomography results were in good agreement with the results of X-ray and electron analyses. The SEM tomography results showed a uniform distribution of both TiB_2 and SiC in the composites, with fine grains approximately 1–2 μm in size for SiC and 2–4 μm for TiB_2. Increasing the SiC content from 0 to 15 vol% initially led to an increase in the flexural strength of the composites, followed by a decrease. The effect on fracture toughness remained insignificant [26,31].

In addition, various nanofillers are used in matrices to change the interphase interaction of components, in addition to reinforcement and increased wear resistance. For example, hydroxyapatite was synthesized in the form of nanofibers and introduced into a polylactic acid (PLA) matrix [32]. The authors note that, despite poor interphase adhesion between hydroxyapatite nanofiber (HANF) and PLA, the destruction of composites was slowed down due to the binding effect of the fibers. The elastic modulus and tensile strength of PLA/HANF with an HANF content of 5 wt% increased by 38 and 13%, respectively, compared to PLA. An increase in the tensile strength of PLA/HANF compared to PLA resin was observed up to 10 wt% of HANF. Hydroxyapatite fibers were then combined with PLA, which further improved the adhesion to the interface between HANF and the PLA matrix, as well as the dispersion of HANF in the composites.

It should be noted that the choice of matrix is determined by the intended application and performance characteristics of the materials. The materials used are diverse, and this article will cover several important groups, with a particular focus on ceramic and carbon materials.

2 Characteristics of CNTs and approaches to forming CNT-based composites

CNTs are a type of crystalline carbon and, depending on the synthesis method, can have a crystalline, amorphous, or mixed structure. These nanoparticles are widely known for their unique physical and chemical properties and practical applications. The classification of CNT is usually based on the number of carbon layers forming their structure, which is equivalent to the number of walls in the nanotube [33,34].

CNTs have attracted considerable attention since their discovery in the 1990s due to their exceptional tensile strength and electrical conductivity, which make them valuable components in the aerospace, automotive, and computer industries. The morphology of single-walled nanotubes (SWNTs) can be determined using various approaches, including atomic force microscopy (Figure 1).

Arc discharge and laser ablation methods allow the production of SWNTs with a high-quality graphite lattice, but they are energy-intensive and yield a small amount of product. In contrast, chemical vapor deposition and catalytic growth in the gas phase are more economical, although the resulting nanotubes often have an imperfect graphite structure. As a result, chirality and geometric parameters play a decisive role in determining the optical, electrical, and mechanical properties of CNT [36,37]. The prospects for using CNT as a reinforcing component in composites are due to their unique combination of physical and mechanical characteristics, including an abnormally low coefficient of thermal expansion (CTE), exceptional

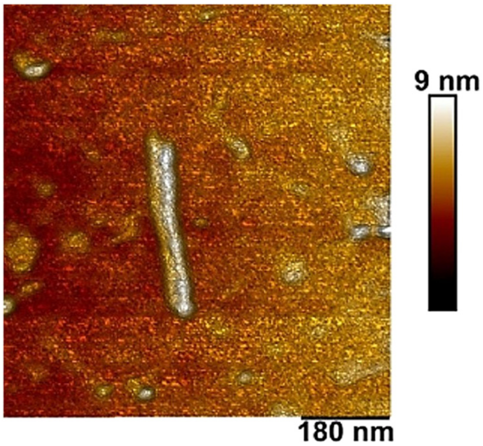

Figure 1: Visualization of MWCNT using atomic force microscopy. Reproduced with permission from [35].

mechanical strength (tensile strength up to 100 GPa), and high thermal conductivity (up to 3,000 $\mathrm{W\,m^{-1}\,K^{-1}}$) [38].

Experimental studies show that adding just 3 vol% of CNT to an aluminum matrix allows achieving an elastic modulus of 90 GPa and a strength of 600 MPa, which is comparable to the characteristics of traditional Al-SiC composites containing 20–25 vol% silicon carbide [8]. At the same time, optimizing the CNT content to 4.5 mass% leads to an increase in hardness and tensile strength by 2.3 and 2.4 times, respectively, compared to an unreinforced matrix. A critical factor determining the performance characteristics of such composites is the degree and uniformity of dispersion of nanotubes in the metal matrix [4]. Numerous studies confirm that nanotubes effectively suppress the thermal expansion of the matrix material, and the degree of CTE reduction depends on both the volume fraction of the strengthening phase and the spatial orientation of the nanotubes [9]. The greatest effectiveness in terms of thermal expansion control is observed when using oriented structures based on CNT, which is associated with the anisotropy of their physical properties and a more sophisticated mechanism of stress transfer at the phase boundary [39,40]. In addition, dispersion, especially uneven dispersion of CNT in the matrix, plays a decisive role in the mechanical and thermal properties of the composite. A number of studies have shown that CNT reduce the CTE, for example, of aluminum composites [38]. The degree of reduction in the coefficient depends on the content of CNTs and their orientation (with oriented CNTs being more effective [40]. Over the past two decades, several materials have been developed using CNT that can store electrical energy, withstand lightning strikes, detect damage, and have the ability to self-repair [41]. Zhu *et al.* [42] reported in their work that incorporating SWCNT into a polymer matrix can increase Young's modulus by up to 20%. Some studies report that the use of SWCNT can increase Young's modulus and shear strength by more than 1,000% [43]. With the introduction of curvature into SWCNT, the elasticity of CNT slightly increases under tension and decreases under compression [44].

Various methodological approaches are used to improve mechanical, electrical, thermal, and barrier properties due to the unique characteristics of CNT. The two main types of matrices for obtaining composites are polymer and ceramic matrices. Polymer nanocomposites with CNT include thermoplastics (*e.g.*, polyethylene, polypropylene, polycarbonate), thermosetting resins (epoxy, phenol–formaldehyde), and elastomers (silicone, polyurethane) [45,46]. The main methods for incorporating carbon nanoparticles are (1) melt mixing, which is used, for example, for polyamide-6 or polypropylene, where shear forces at high temperatures ensure the dispersion of CNTs [47]; (2) mixing solutions, which is used for polyaniline, PET, and PVC. CNTs are pre-dispersed in solvents (dimethylformamide, chloroform) followed by polymer precipitation [48,49]. In addition, ultrasonic treatment [50], mechanical mixing [51], and probe dispersion [52]. Resin transfer molding deserves special mention, as it is used for epoxy matrices and provides a high degree of CNT wetting and the production of large-sized composites [53].

When ceramic matrices are used, CNT-reinforced ceramic composites (CNT-CMC) can be obtained, which are used in the aerospace, defense, and energy industries due to their high thermal stability and strength [54]. According to an analysis of the literature, the following methods of incorporating CNTs into ceramic matrices are most in demand: (1) mechanochemical mixing, in which matrix powders and CNTs are subjected to high-energy mixing in planetary mills [55]; (2) the sol–gel method is often used for SiO_2 matrices and provides an optimal level of CNT distribution [56]; (3) hydrothermal synthesis is used to create hybrid nanostructures and improve interphase interaction [57,58]; and (4) spark plasma sintering (SPS) provides dense packing and control over microstructure, actively used for Al_2O_3 and SiC matrices with CNTs [59,60]. Hot pressing is a classic method for creating dense composites based on ZrO_2 and Si_3N_4 [61,62]. The manufacturing parameters and some characteristics of composites based on polymer and ceramic matrices and CNTs are presented in detail in Table 2.

Thus, the combination of CNT with various polymer and ceramic matrices using adapted synthesis methods provides a wide range of mechanical and functional characteristics, paving the way for the creation of a new generation of multifunctional nanocomposites. At the same time, the most preferred method for obtaining composites is FSP (Figure 2) with the introduction of dispersed nanofillers, as it combines technological versatility, a high degree of strengthening, and a wide range of applications [63–65].

The comprehensive analysis of ceramic matrices modified with CNT is a priority area in modern materials science. It focuses on the development and fundamental understanding of new classes of nanostructured composites with improved performance characteristics. The relevance of this area is due to the need to create a new generation of structural and functional materials that are simultaneously high-strength, low-density, thermally and chemically resistant, and capable of integrating additional functions such as electrical conductivity, thermal stability, or crack resistance. Reinforcing ceramic matrices with CNT not only significantly expands the strength characteristics

Table 2: Characteristics and features of the production of composite materials reinforced with CNT, with polymer and ceramic matrices

Criterion	Polymer matrices with CNT	Ceramic matrices with CNT
Processing temperature	Low (usually <300°C)	High (up to 1,600°C and above)
Methods of dispersing CNT	Simple: Ultrasound, dissolution, extrusion	Complex: Ball milling, sol–gel, SPS
Preservation of CNT structure	Good (at low temperatures)	Risk of destruction during sintering
Compatibility with CNT	Often poor, chemical functionalization required	Very poor without modification, especially with oxide matrices
Interfacial adhesion	Moderate, improved by functionalization	Complex, but can be high with good chemical compatibility
Alignment and orientation of CNT	Easily achieved by extrusion, centrifugation	Difficult to implement, requires special methods (magnetic field, freezing)
Technological simplicity	High, easily scalable	Low, requires complex equipment
Mechanical strength of the composite	Moderate, limited by polymer properties	High, especially with optimal sintering
Crack resistance	Insignificant improvement	Significant improvement due to CNT bridges
Thermal conductivity and electrical conductivity	Can be improved (depending on CNT content)	Significant improvement with CNT alignment
Material density	Low	High
Application	Electronics, biomedicine, packaging, flexible electronics	Aerospace, tools, thermal barriers

of brittle ceramics, but also increases the resistance to destruction and durability of materials under extreme loads.

3 Ceramic composites and nanocomposites

Ceramic materials and ceramic matrix composites (CMC) are of particular interest for a variety of applications. Ceramics are known to consist of metallic and non-metallic elements and are valued for their hardness and resistance to high temperatures. However, ceramic materials are very brittle, which limits their use [66]. They also have low tensile strength [67], but they are good thermal and electrical insulators [68]. Due to strong ionic or covalent bonds in ceramic materials, plastic deformation is difficult, resulting in their brittleness. There are traditional methods to reduce the brittleness of ceramic materials such as (i) grain size reduction, based on increasing the total area of grain boundaries to make it difficult for cracks to propagate; (ii) surface toughening, dispersion toughening by adding a second phase; and (iii) texturing by a given grain orientation [69]. Modern methods are aimed at obtaining CMC in which the ceramic material forms a continuous matrix and nanofillers of various types are used as a reinforcing component. These nanoparticles or nanofibers make it difficult for cracks to spread and enable composites to withstand sudden temperature changes without breaking. The ceramic matrix, in turn, enables the composite material to be resistant to corrosion and oxidation. Continuous or long and discontinuous or short fibers are used to increase the strength of CMC. Discontinuous or short-fiber composites consist of non-oxide alumina, as well as oxide alumina materials [70,71]. The utilization of zirconium oxides, SiC, TiB_2, and aluminum nitride in the reinforcement of a ceramic matrix is outlined elsewhere [72,73]. Continuous or long fibers exhibit superior strength, which endures even when the ceramic matrix fractures, thereby mitigating the propagation of cracks. Conversely, short fibers are employed to enhance resistance to cracking. Monofilament fibers provide a better interfacial connection, which makes matrix materials more durable [74,75].

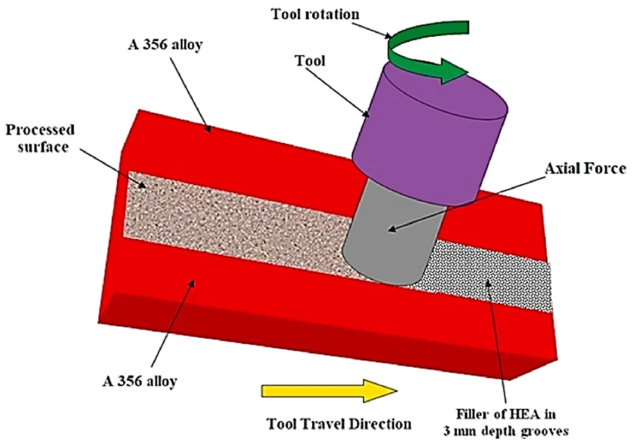

Figure 2: Composite creation procedure. Reproduced with permission from [63].

Depending on the matrix used, ceramic matrix nanocomposites are divided into oxide and non-oxide composite systems [76]. Nanofillers can additionally be classified by origin, composition, and size [77] (Figure 3).

Nanofillers, even at a level of less than 1%, can endow nanocomposites with desired properties. This is in contrast to higher loading levels of approximately 15–40 vol% for conventional microfillers in traditional composites [78].

Karbalaei Akbari *et al.* [79] introduced two types of TiB_2 particles, namely micro- and nanocomposite reinforcements, into a molten A356 aluminum matrix using a mechanical stirrer. Nanocomposite elements allowed obtaining materials with enhanced tensile strength and viscosity. In particular, the addition of optimal amount of the TiB_2 nanocomposite (1.5% by volume) resulted in a 43% increase in tensile strength and a 27% increase in elongation compared with a non-reinforced alloy.

In the majority of composite systems, it has been observed that nanofillers such as fibers, filamentous crystals, and plates at an optimal load level increase the fracture toughness of ceramics. These nanofillers have been shown to delay the onset of cracking and prevent crack propagation [80].

It should be noted that ceramic nanocomposite materials are of high practical importance, since they can be used in the aircraft industry to increase the performance of aircraft systems [81], as well as in mechanical engineering to increase the wear resistance of piston mechanisms [82].

Many new CMCs with improved properties have been developed. For example, a ceramic composite consisting of aluminum oxide, glass frit, and microencapsulated phase change materials was obtained, which could maintain stable heat storage characteristics after 200 melting-solidification cycles with minor latent heat losses [83]. In the study by Wang *et al.* [84], a high-temperature phase change material made of a ternary salt/porous Si_3N_4 composite was fabricated. The skeleton material was composed of Si_3N_4 porous ceramics, while the filler was a ternary salt consisting of 50 wt% sodium chloride, 30 wt% potassium chloride, and 20 wt% magnesium chloride. The thermal conductivity of the composite was found to be six times higher than that of pure ternary salts, and the thermophysical properties of the composite remained stable even after 300 thermal cycles.

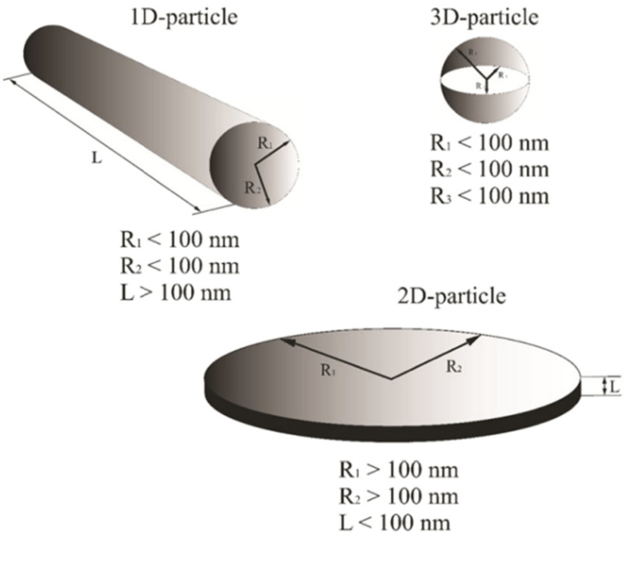

Figure 3: Classification of nanofillers in terms of size and morphology.

In a recent study, multiphase ceramics composed of corundum, spinel, and mullite were obtained, exhibiting high resistance to thermal shock [85]. Furthermore, novel high-entropy disilicate (Y0.25Yb0.25Er0.25Sc0.25)2Si_2O_7 ((4RE0.25)2Si_2O_7) materials were also obtained through a two-step process, which demonstrated favorable phase stability during 1.5 h of sintering at 1,600°C, in addition to demonstrating excellent corrosion resistance [86].

It is noteworthy that carbon materials are among the most widely employed nano-sized additives in ceramic composites, and their utilization is described in more detail below.

4 Carbon reinforcing elements

Composite materials created by mixing different components have improved properties compared to the original constituents. For example, the powder metallurgy method makes it possible to combine metal powder with reinforcing material (fiber, ceramics or other metal), and the ratio of components is important for achieving the required characteristics. This method is cost-effective and allows producing complex shapes of composite products. In the field of materials science, nanocarbon has been identified as a particularly effective toughening phase in CMC. In recent years, there has been a surge of interest to the use of nanocarbon materials for the strengthening of polymer composites [87]. Promising nanofillers for this application include graphene oxide and graphene [88], pyrocarbon [89], and graphite [90], which have the potential to mitigate the limitations of ceramic materials due to their superior thermal, electrical, and mechanical properties. In addition, composites based on carbon materials and aluminum are lightweight, which is advantageous, for example, for the aerospace industry, where it is important to save fuel and reduce the emission of harmful compounds into the atmosphere.

A significant difficulty in obtaining ceramic composites with carbon nanofillers is to reach the uniform filler distribution. Due to the strong electrostatic interaction of the surfaces of nanomaterials, they are prone to aggregation in the composite material. Surface modification of carbon materials, addition of bonding agents and addition of surface active compounds are often used to optimize the interfacial interaction in matrices. The synthesis of SiCN(CNT) composite ceramics has been achieved through the polymer-derived method [91]. For SiCN(CNT) pyrolyzed at a temperature of 1,100°C, an optimal reflection loss of −38.40 dB was obtained at a frequency of 18 GHz in the sample with a thickness of 4.4 mm, and an effective absorption bandwidth (EAB) was 1.3 GHz (from 16.7 to 18 GHz). Besides, with a thickness of 4.7 mm, a wider EAB of 2.16 GHz (from 15.76 to 17.92 GHz) was achieved. Thus, SiCN composite ceramics containing CNT demonstrate an attractive potential for applications in the field of microwave radiation absorption [37]. Using the one-step sintering method, C/$MgAl_2O_4$ composites were obtained, and graphene with 6–10 layers was distributed in the agglomerates, increasing the materials' resistance to oxidation, thermal shock, and slag corrosion [92].

A considerable number of studies have demonstrated that various types of carbon materials have the capacity to enhance the mechanical properties of ceramic matrices to a significant degree [93–95]. For instance, it has been shown that CNT can provide enhanced interlayer and intralayer reinforcement, as well as an increased resistance of composites to delamination [91]. The utilization of nanofibers as a reinforcing agent in materials is attributable to two principal factors: the high ratio of surface area to the volume of the fibers, and their high aspect ratio. Nanofillers have been shown to exhibit superior mechanical properties in comparison to micro-reinforcing materials; however, it is necessary to ensure good dispersion of nanoscale fillers. The analysis of literature data shows the problems in improving matrix properties when the use of equal volumes of nanofillers leads to incomplete changes in the properties of the resulting composite, which emphasizes the relevance of the study of properties and mechanisms of deformation of composites [96].

Tungsten carbide matrix composite reinforced with a hybrid nanocomponent of CNT, SiC nanowire (SiCnw), and graphene (G) (G/CNT/SiCnw-WC) was developed [97]. All the conventional toughening mechanisms, provided by utilization of separate G, CNT, or SiCnw, were operational, and a synergistic toughening effect was obtained as a result of improved uniform dispersion of G/CNT/SiCnw, which facilitated the maximization of the toughening effect. The augmentation of the number of toughening mechanisms is illustrated in Figure 4 [97].

CNT can improve the thermal properties of polymers and ceramic materials. Shah *et al.* [98] produced a graphene/CNT/alumina hybrid composite and reported an increase in thermal conductivity of the composite at 1% CNTs and 0.4% wt% graphene. CNTs can be used to increase the crack resistance of ceramics [99]. Mirkhalaf *et al.* [100] compared the polymer-derived ceramics (PDCs) toughened with three nanofillers (CNT, Si_3N_4, and Al_2O_3 nanoparticles) and found that CNTs were less efficient than Si_3N_4 and Al_2O_3 nanoparticles (Figure 5a), because of the formation of CNT agglomerates (Figure 5c).

The incorporation of nanotubes resulted in a marked enhancement of the ceramics mechanical properties, with the addition of 1 wt% CNT leading to a 50% increase in the modulus of elasticity. However, the modulus of elasticity of the material remained almost constant with further addition of CNT. The hardness of the material increased from approximately 4.1 GPa for a specimen without filler to approximately

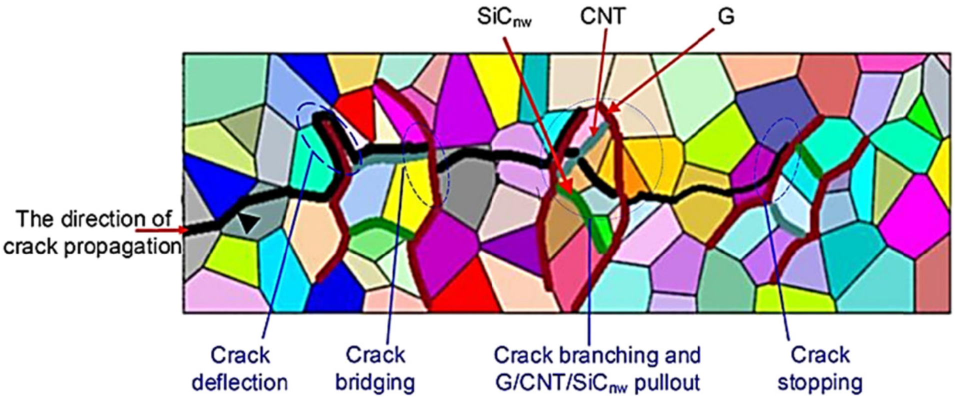

Figure 4: Scheme of toughening mechanisms in a ceramic composite, provided by a hybrid reinforcing agent G/CNT/SiCnw. Reproduced with permission from [97].

9.8 GPa (an enhancement of 2.5 times) for a sample containing 1 wt% CNT, after which it no longer increased [100]. The incorporation of nanotubes results in an enhancement of the overall mechanical properties, which may be attributed to the formation of microscopic cracks observed in several studies of CNT-reinforced PDCs [101].

Thus, carbon materials, both individually and as part of complex reinforcing compounds, can significantly enhance the properties of the initial matrix, provided that their uniform distribution in the matrix is ensured.

5 Dispersion of nanocarbon in a ceramic matrix

In addition to the advantageous properties of carbon materials, there are certain limitations that must be considered. A significant disadvantage of using carbon fibers is their inert surface, which results in the poor compatibility of the interface surface with polymer matrices, and, consequently, to numerous defects of the interface surface and pores in finished carbon fiber composites. The dispersion of nanocarbon in CMC is challenging due to its significantly large specific surface area, surface energy, van der Waals forces resulting from the intermolecular electric dipole moment, and interactions between functional groups, as well as its tendency toward aggregation and entanglement [101]. The interfaces between nanocarbon and ceramics are considered suitable due to disparities in surface tension and density. It is important to note that currently, there is no single method for measuring the strength of ceramic matrix nanocarbon composites. Traditionally, strength is defined as the energy required to propagate a crack per unit length. However, high anisotropy of composites makes the measurements difficult.

It is noteworthy that the characteristics of composites largely depend on the method of their manufacture [102]. Wet or dry mixing of carbon material with ceramic powder is most often used to produce composites [103], but this results in agglomeration and loss of the original structure of the materials. However, a novel method for the fabrication of nanocarbon/ceramic composites utilizing a combined gel casting and reduction sintering process has been proposed [104]. This novel one-step approach proposes a promising strategy for the production of aluminum oxide and nanocarbon composites. It is noteworthy that

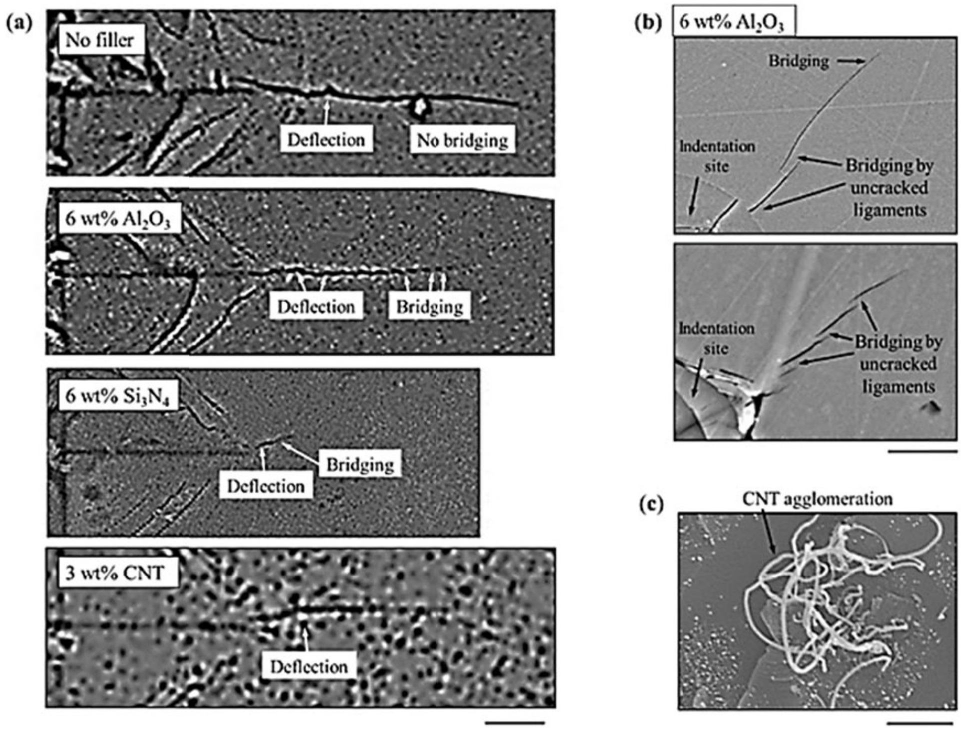

Figure 5: Visualization of the mechanism of composite toughening using nanofillers. (a) Comparison of crack propagation in cases without fillers, with 6 wt% Al_2O_3, 6 wt% Si_3N_4 and 3 wt% of CNT. (b) A more thorough examination of the process of toughening by filling cracks in PDC modified with Al_2O_3. (c) Agglomeration of nanotubes that has been observed on the surface of specimens modified with CNT. All cracks were produced with the same force on the PDC. Reproduced under the terms of the Creative Commons Attribution License from [100], Copyright © 2021, Crown.

the nanocarbon-ceramic composite fabricated using Al_2O_3 exhibits excellent p-type semiconducting properties and ultrahigh carrier mobility in the holes, contrasting with the metallic property of a conventional composite [105].

Technologies for producing composites with improved properties are being developed. For instance, the creation of an integrated composite that can be used at an operating temperature of ≈1,900°C was described. This is approximately 300°C higher than that of the previously described porous materials that can be used for aerospace vehicles in oxidizing environments. The resulting material has the properties of ablation resistance (ablation rate of $0.38\ \mu m\ s^{-1}$), thermal insulation (temperature difference of 1,050°C with a thickness of 10 mm), thermostability (shrinkage of 0.08%), and high load-bearing capacity (compressive strength of 90.5 MPa) at a temperature of 1,900°C. In addition, the material exhibits a substantially higher specific strength of 119 MPa per 1 cm^3, which is 12–34 times greater than that of rigid insulation boards, which are the most widely used materials for wear-resistant and heat-insulating applications. Such a carbon-ceramic composite should facilitate the design and increase the operating temperatures of thermal protection systems for critical components, thereby significantly enhancing the performance of future high-temperature thermal protection systems for aerospace vehicles [106].

Homogeneous distribution of nanocarbons in the ceramic matrix is fundamental for improving composite properties. Three common methods are employed for the optimization of the dispersion of solids in aqueous solutions, namely surface wetting, electric double layer, and colloidal protection [107,108]. Additionally, the volume fraction of CNT within the matrices significantly impacts their dispersion and wettability. Therefore, it is recommended to select a low volume fraction to enhance dispersion and promote high wettability. Optimization of the volume fraction should not be compromised prior to developing a CNT-reinforced composite. The maximum tribological enhancement of composites through CNT reinforcement can only be accomplished under the condition of uniform dispersion and complete wettability of CNT [109].

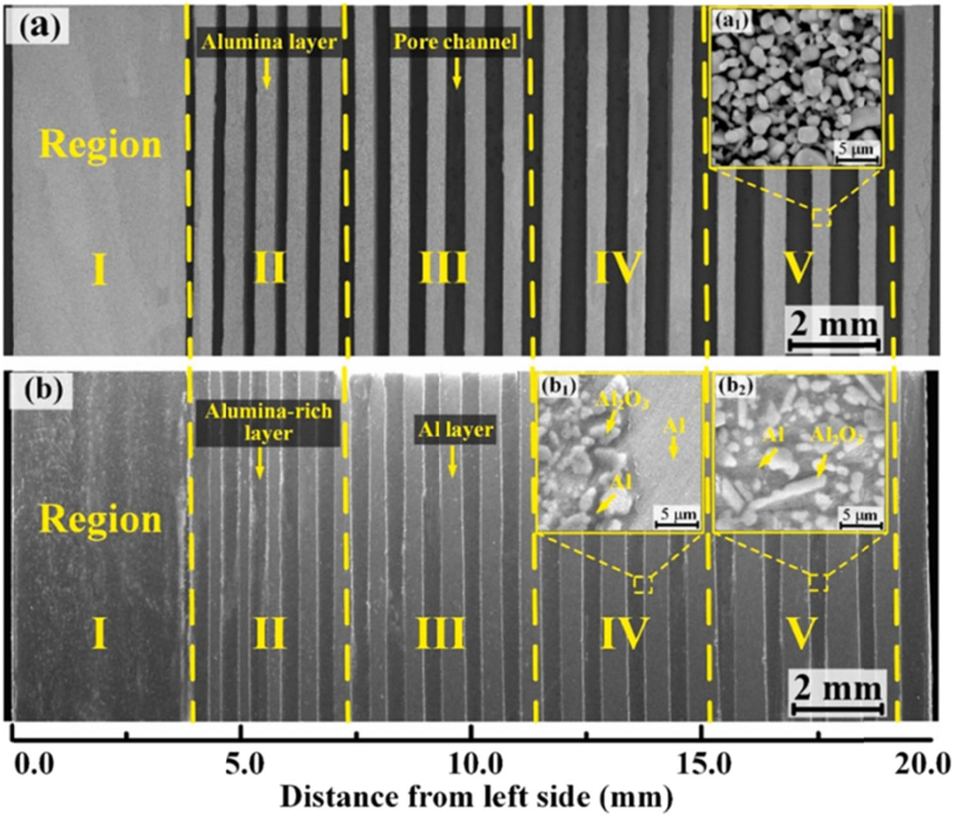

Figure 6: The macrostructures of (a) an Al_2O_3 scaffold and (b) an Al/Al_2O_3 composite visualized with SEM. Magnified views of the microstructures of a porous Al_2O_3 wall, an interface, and an infiltrated ceramic layer are shown in the insets (a1), (b1), and (b2), respectively. Reproduced under the terms of the Creative Commons Attribution License from [112], 2024 Elsevier B.V.

6 Methods for estimating the dispersion of nanofillers

Filler uniformity can be assessed using direct and indirect methods. Direct dispersion estimation includes image analysis methods such as SEM and X-ray computed tomography (CT) [110], but it is difficult to quantitatively evaluate the fiber dispersion using these methods. The indirect method typically relies on mechanical or electrical characteristics for macroscale estimation, which is not suitable for individual distribution checks, considering the influence of the matrix [110].

The study of composite morphology is important for understanding their physical properties, and electron microscopy methods such as SEM and transmission electron microscopy (TEM) are widely used for this purpose. These methods are very helpful for determining the shape and size of domains in composites, as well as the degree of their adhesion to the main component at the interface. The application of SEM facilitates the correlation of alterations in morphology with the processing parameters employed [111].

For example, the structure of the Al_2O_3 scaffold sintered at 1,400°C for 2 h was studied using SEM. It was shown that the thickness of the ceramic layers was approximately 360 μm (Figure 6) [112].

In order to conduct a more comprehensive study of the physical properties of composites, it is important to combine various methods, such as wide-angle X-ray scattering (WAXS), small-angle X-ray scattering, small-angle neutron scattering, light scattering, and differential light scattering. Scanning calorimetry [113] is also a useful method in this respect.

The use of theoretical models and numerical simulations to predict the physical properties of a composite can be an efficient way to save time and cost when developing a new composite, even if it cannot completely replace experimental research [113–115].

With the help of supercomputer methods, realistic modeling of large-scale tasks consisting of several objects has become faster and more accessible in recent years, for example, modeling the formation of a seepage network. A microstructural model has been developed for modeling the nonlinear behavior of the conductivity of composite dielectrics modified with metal oxide fillers, which allows taking into account the features of the destruction of the insulating matrix and the mechanism of internal conductivity, as well as the exact percolation threshold, which was 33% [116].

A new morphological model was used to understand the importance of CNT aggregation and electron tunneling. The influence of the geometry of the carbon nanofiller, aspect ratio, aggregation, and concentration has also been studied. The simulation results show that the degree of aggregation has a significant effect on the change in electrical conductivity depending on the concentration of nanoadditives. Well dispersed nanofillers are not always preferred for forming conductive networks [117]. Currently, machine learning is successfully used to predict the properties of composite materials, which involves analyzing and determining the nonlinear relationships between properties and related factors based on existing information. For example, a trained neural network can be used to predict Young's modulus and tensile strength of polyethylene nanocomposites based on the geometric parameters of the nanometric filler. The combined use of deep learning with traditional experimental approaches makes it possible to obtain more accurate predictions of the mechanical properties of a composite material [118].

7 Conclusion

The study of the structural characteristics of composites and nanostructures is of fundamental importance in materials science. Thanks to their unique properties, ceramics and their composites can replace traditional materials in various fields, such as gray cast iron in brake systems. This is largely due to their improved physical and mechanical properties, in particular high wear resistance and low friction coefficient, resistance to high temperatures and other factors that are crucial for reducing component losses during rotation and increasing component durability [119]. This article considers the use of carbon materials to enhance the properties of ceramic matrices. It is demonstrated that

1. The introduction of nanofillers with different morphologies and chemical compositions in various proportions allows for the targeted improvement of key properties of the base matrix.
2. An increase in the mechanical (strength, stiffness, and resistance to destruction), thermal, and electrochemical properties of composites has been demonstrated.
3. Thanks to their significantly larger specific surface area, nanofillers can achieve excellent interaction with the matrix, which leads to improved dispersion and, ultimately, more promising composites than when using macrofillers.
4. To evaluate filler dispersion, direct methods (microscopy [*e.g.*, SEM, TEM, AFM], spectroscopy, and tomographic methods) are used, as well as analysis based

on changes in physical, mechanical, or electrical properties. Each method has its advantages and limitations and can be selected depending on the type of matrix and the nature of the filler.

5. The use of computational tools, including finite-element analysis, multiscale modeling, and machine learning algorithms, opens up new possibilities for predicting the behavior of composites, optimizing the selection and distribution of fillers, and developing a new generation of high-performance nanocomposites.

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