



Multi-walled carbon nanotubes (MWCNTs)-reinforced ceramic nanocomposites for aerospace applications: a review

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ABSTRACT

Advances in the nanotechnology have been actively applied to the field of aerospace engineering where there is a constant necessity of high durable material with low density and better thermo-mechanical properties. Over the past decade, carbon nanotubes-based composites are widely utilised owing to its fascinating properties resulting in series of multidisciplinary industrial applications. Carbon nanotubes (CNTs) are rolled up sheets of carbon in nanoscale which offers excellent thermal and mechanical properties at lower density which makes them suitable reinforcement for composites in aerospace applications. Owing to its high Young's modulus and chemically inert behaviour, CNTs are forefront of material research with applications varying from water purification to aerospace applications where applicational sector remains a mystery. Although there has been numerous research on the CNTs-based materials, there are only limited studies focusing on its utilisation for the field of aerospace engineering. As a result, in this review, we intend to cover the processing and synthesis techniques, thermal and mechanical properties as well as few industrial applications of CNTs-reinforced ceramic composites. Further, any potential development in additive manufacturing-based technique for fabricating CNT/ceramics and its applications in aerospace industries have been highlighted.

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Abbreviations

CNTs	Carbon nanotubes
SWCNTs	Single-walled carbon nanotubes
MWCNTs	Multi-walled carbon nanotubes
CVD	Chemical vapour deposition
Fe	Iron
Co	Cobalt
TCCVD	Thermal catalyst chemical vapour deposition
C ₂ H ₂	Acetylene
H ₂	Hydrogen
CH ₄	Methane
Sol-gel	Inorganic colloidal suspension (sol) and gelation of the sol in a continuous liquid phase (gel)
SrTiO ₃	Strontium titanate
Mg	Magnesium
CMCs	Ceramic matrix composites
TEOS	Tetraethyl orthosilicate
TiO ₂	Titanium oxide
HPP	Hot pressing process
SPS	Spark plasma sintering
YSZ	Yttria-stabilised zirconia
CTAB	Cetyl-trimethyl ammonium bromide
SiO ₂	Silicon oxide
Al ₂ O ₃	Aluminium oxide (alumina)
SiC	Silicon carbide
SDS	Sodium dodecyl sulphate
GA	Gallium
MnO ₂	Manganese dioxide
Ni	Nickel
Ar	Argon
MgO	Magnesium oxide
FeCl ₃	Ferric chloride
Sc ₂ Si ₂ O ₇	Thortveitite
Ni (NO ₃) ₂ · 6H ₂ O	Nickel(II) nitrate hexahydrate crystals
Fe (NO ₃) ₃ · 9H ₂ O	Iron(III) nitrate nonahydrate
TiB ₂	Titanium diboride
Si ₃ N ₄ + Lu ₂ O ₃	Silicon nitride + lutetium oxide
ZrO ₂	Zirconium dioxide
AM	Additive manufacturing
TPI	Thermosetting polyimide
DSM Novamid® ID1030 CF10	New PA6/66 grade of carbon-reinforced polyamide
C	Carbon
FDM	Fused deposition modelling
ZrO ₂	Zirconium oxide

NASA	National Aeronautics and Space Administration
JAXA	The Japan Aerospace Exploration Agency
ISRO	Indian Space Research Organization
TiC	Titanium carbide
EMI	Electromagnetic interfaces
HAP	Hydroxyapatite
UAV	Unmanned aerial vehicle
VR	Vacuum radiation
UV	Ultraviolet radiation
Cr ₂ O ₃	Chromium(III) oxide
UHTCs	Ultra-high-temperature ceramics
Hf	Hydrogen fluoride
Zr	Zirconium

Introduction

Interest towards the field of nanotechnology remains elevated in several disciplines of science due to its higher potential in areas involving engineering, agriculture and medicine. While the term “nanotechnology” has no approved definition, the products of nanotechnology are said to be measured at least less than 100 nm in any one dimension [1]. Being a specific component of nanotechnology, nanomaterials have attracted various researchers owing to its promising properties in mechanical, thermal and electronic disciplines. The multidisciplinary expansion of the nanomaterials depended on the support from industrial and academic sectors which included extremely funded research projects including National Nanotechnology Initiative (NNI) by US government in 2001. The NNI research supported the commercialisation, development and research on the field of nanotechnology with a budget of more than a \$1 billion [2]. Over past few decades, discovery and analysis on the properties of the nanomaterials have gained constant worldwide growth [3].

Carbon nanotubes (CNTs) are allotropes of carbon which exists in a quasi-one-dimensional structure which consists of graphite (carbon atoms) arranged in hexagonal structure creating a nanotube forming multiple layers [4]. CNTs are tube structured

materials made from layers of graphite in nanoscale with varying outer diameter of 3–30 nm. Some researchers consider the CNTs to be a derivative of both carbon fibres and fullerenes arranged in a muffled tube structure [5]. CNTs are generally classified by the number of carbon layers combined to form out the surface, i.e. single-walled carbon nanotubes (SWCNTs) where hexagonally structured single graphene layer varying diameter range of 0.4–5 nm and multi-walled carbon nanotubes (MWCNTs) which comprises of two or more sheets of the carbon with diameter range of 1–20 nm [6–8]. Though classification of CNTs depends on different layers of carbon, this allows them to have varying attributes. For instance, SWCNTs are suitable for electronic devices, sensors and field transmitters whereas mechanical-based applications prefer MWCNTs [9–11]. Being distinguished by the properties, synthesis techniques of the CNTs also vary as MWCNTs are simpler to produce compared to SWCNTs which require controlled growth and particular atmospheric conditions leading to difficulty in bulk synthesis [12, 13]. Further, synthesis technique also determines the purity of CNTs as most of the techniques are dependent on the catalysts and gases utilised during production. Several synthesis methods such as arc discharge, chemical vapour deposition (CVD), plasma rotation and hydrothermal processes are being developed to produce high-quality CNTs as per the requirements of the research [14]. Chemical vapour deposition is one of the most common techniques which is utilised to synthesis CNTs. The CNTs formed through the technique depend on the various factors including temperature, atmospheric conditions, hydrocarbon source, catalysts and reactor type (i.e. horizontal or vertical). Typically, CVD technique involves agents like metal catalysts such as iron (Fe) or cobalt (Co) and hydrocarbon sources such as methane and ethylene [15].

Various researchers have utilised CVD-based techniques to synthesis CNTs by varying parameters such as catalysts, hydrocarbon sources which allowed them to control the growth and structure of CNTs [16–18]. For instance, Arunkumar et al. synthesised MWCNTs using thermal catalyst chemical vapour deposition technique (TCCVD) as illustrated in Fig. 1 by decomposition of C_2H_2 with Fe/MgO as catalyst which was prepared by liquid impregnation. The technique resulted in MWCNTs with diameter of ~ 20 nm and purity of $\sim 97\%$ [14]. Alternatively,

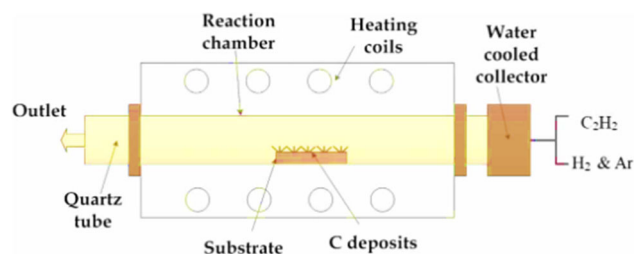


Figure 1 TCCVD technique set-up utilised for production of MWCNTs adapted from Arunkumar et al. with permission through rights link [14].

techniques like plasma rotation and arc discharge has also been utilised to synthesis CNTs along with CVD-based techniques. For instance, MWCNTs were first reported by Iijima et al. through arc discharge method while controlling the parameters to form fullerene [4]. Arc discharge method utilises high temperature (> 3000 °C) to evaporate carbon atoms into plasma forming SWCNTs or MWCNTs based on the atmospheric conditions. On the other hand, there has also been development with other techniques like plasma rotation and in situ emulsion polymerisation which have been capable of synthesising high-quality CNTs [19, 20]. Flame synthesis is one of the recent techniques for producing CNTs in large volume compared to other techniques. Metallic-based catalyst particles, heat source and a source of carbon are the three main components for the synthesis of CNTs in flame synthesis. This process has potential to produce CNTs on any required surface, especially in highly controllable way [21, 22]. Through flame synthesis, single-walled carbon nanotubes (SWCNTs) have been observed formed in a premixed acetylene/oxygen with 15 mol% argon flame doped with 6100 ppm iron pentacarbonyl vapour and operated at a pressure of 50 Torr (6.7 kPa) [23]. The parameters of this process are flexible and can be modified based on the required particle size and the growth of CNTs. These parameters include flame temperature, concentration of carbon specimen used, catalyst type, equivalence ratio and fuel type. The alignment is often desired and high purity of CNTs can be produced by using this process. Under the time frame of 1 min, CNTs are formed with a typical length of 1–5 μ m and a mean diameter of 30–85 nm [24]. Carbon nano-onions were produced using enhanced acoustic modulation process in flame synthesis [25]. Flame synthesis is used and a substrate of iron-doped Al_2O_3 – ZrO_2 was used which is then catalysed

decomposition of methane [26]. However, even with these techniques on place, CVD and its by techniques are considered to be the best way to produce CNTs owing to its cost effectiveness and high purity.

From its discovery, significant research has been constantly carried around CNTs on its synthesis, reinforcements and coating areas to explore the unique properties and applications. The distinctive properties and characteristics of the CNTs make them a potential reinforcement material in the field of engineering. CNTs have been utilised as reinforcing materials for metal and ceramics to improve its properties in various applications [27]. Nowadays, ceramics are preferred over metals and alloys owing to its thermal stability and lower density which make them a potential candidate in for structural, aerospace and industrial applications [28–30]. Although brittle nature of ceramics reduces its suitability in field of structural and aerospace applications. Various research has been conducted to enhance the fracture toughness of the ceramics by reinforcing them with various materials including metal and ceramic additives. Although with the potential of CNTs being a suitable reinforcement material, research work on incorporating CNTs has proved to be effective to ceramics composites as studies have demonstrated enhancement in fracture behaviour [31–33].

Although over the decade, there has been constant research on incorporating CNTs into by focusing on the processing techniques and properties of the composites. There is limited research on the proving the potentiality of CNTs-reinforced ceramic nanocomposites in the field of aerospace engineering by detailing fabrication technique and properties associated with the composites. Thus, this review intends to provide a comprehensive review on the processing and densification techniques associated with MWCNTs-reinforced ceramic composites with highlights towards the future manufacturing process, i.e. additive manufacturing. Further, potential applications of the CNTs-reinforced ceramics composites

in aerospace and industrial sectors. The utilisation of the CNT/ceramic nanocomposites focused on the current concerns on the aerospace field such as structural, shielding, icing and barrier coatings.

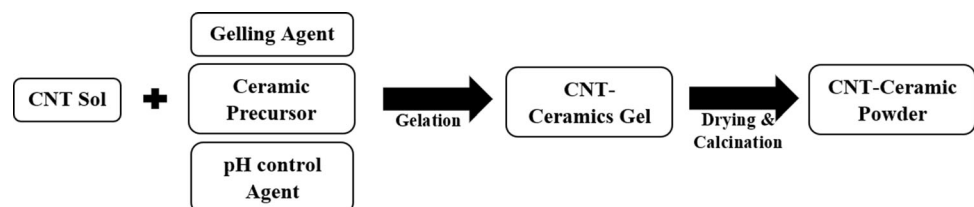
Processing techniques

The remarkable stiffness, axial strength of CNTs with its stability in chemical and thermal environment makes them a unique and a crucial reinforcement to improve the mechanical properties of ceramics. Many studies reported marginal improvements mechanical properties after the CNT reinforcement [34, 35]. These results are dependent of CNTs synthesis techniques, composite processing method utilised by various researchers during their research. This section provides an overview on the different CNTs processing techniques associated with pre-processing of ceramic composites. The common CNTs processing techniques include sol–gel, powder metallurgy and in situ growth techniques. However, a colloidal route is also being followed by researchers around the world which is cheaper and time efficient compared to other processing techniques [33].

Sol–gel route

Sol–gel is one of the oldest processing methods available in the material science where highly pure composite powder could be produced owing to the controlled parameters in sol–gel operations [36, 37]. The technique could be utilised to distribute CNTs homogenously throughout the ceramic matrix with uniform crystalline size with help of ceramic precursors and control agents [38]. Figure 2 shows the schematics of the sol–gel process for forming CNT–ceramic powder through drying and calcination techniques. Though sol–gel is cheaper and sustainable method, the dispersion of CNT into ceramic is non-compatible owing to the hydrophobic nature of CNTs. Therefore, surfactants are required for dispersing CNTs into the aqueous solution [39]. Further

Figure 2 Schematic of CNT–ceramic processing through sol–gel technique.



for uniform dispersion of the CNTs onto the matrices, external techniques are employed which enhances higher homogeneity on the composites. These external dispersion techniques involve physical methods such as ultrasonication, planetary ball milling and plasma and irradiation and chemical methods using organic and inorganic compounds which can interact with MWCNTs leading to dispersion [40]. Silva et al. created MWCNTs/zirconia and alumina-based ceramic composites by dispersing MWCNTs with help of dabcosil stearate and established that the MWCNTs were dispersed uniformly in high quantities on the sol [41]. Likewise, Lopez et al. prepared the sol–gel derived silica-reinforced MWCNTs coating using Tetraethyl orthosilicate (TEOS) as precursor and ethanol as gelling agent and physical techniques like ultrasonic probe and magnetic stirrer for enhancing distribution throughout the surfaces [42]. Similarly, Geo et al. prepared CNTs/TiO₂ composites through sol–gel process by dispersing MWCNTs using a sodium dodecylbenzene sulfonate surfactant and ultrasonication for uniform distribution of the MWCNTs [43]. However, few reports of Arunkumar et al. reported that the physical techniques were not able to be satisfactory in providing only through single physical techniques therefore he utilised two or more techniques to support higher homogeneous distribution. The report also specified that with increase in CNTs content, the rate of uniform distribution reduces owing to increase in Vander Waal force of attraction between CNTs particles [31].

Powder metallurgy

The simplest technique to fabricate any ceramics is said to be through powder metallurgy process. The techniques depend on different densification

techniques including conventional techniques like pressureless sintering and non-conventional techniques like hot pressing process (HPP) and spark plasma sintering (SPS) by measuring the weight/volume fractions of different materials required to form the final composite structure. Although conventional techniques of densifications are available, recent manufacturing trends have given raise to techniques like HPP and SPS which enhance the density of composites by compacting with high pressure. Powder metallurgy mostly depends on the mixing process where different techniques such as manual milling, ball milling, ultrasonication and other processes are involved [44, 45]. Arunkumar et al. fabricated MWCNTs/yttria-stabilised zirconia (YSZ) nanocomposites using SPS process as shown in the flow chart (Fig. 3). The authors mixed the MWCNTs with YSZ creating a slurry through ultrasonication, followed by magnetic stirring and further ball milling before drying using rotary evaporation [31]. Different researchers have worked with powder metallurgy for developing desired ceramic composites followed with various densification techniques [46]. Though powder technique is followed for synthesis of composite powders, the strength and properties of the powders depend on densification techniques which could lead to fatal effects to the composites leading to cracks on the samples or reduced density. Therefore, there is a constant demand to enhance the densification technique throughout the research area of materials science.

Along with powder processing, colloidal preparation route in which CNTs are dispersed into solvent using physical and chemical techniques is also followed to produce CNT-based nanofluid. The colloidal route of nanofluid preparation is a part of different fabrication techniques as it is applied to

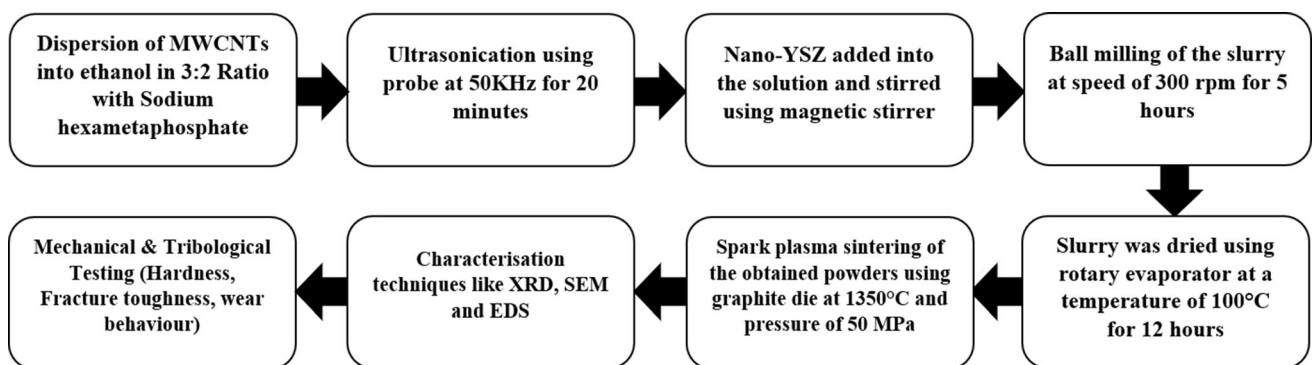


Figure 3 Powder processing technique schematics.

Table 1 Colloidal route for dispersing ceramic matrix into MWCNT nanofluids

Dispersion medium	Physical/chemical method	Ceramic matrix	Results/outcome	References
CTAB (cetyltrimethylammonium bromide)	N/A	SiO ₂	97% relatively dense ceramic composites of MWCNTs/SiO ₂	[49]
Distilled water	Polyethyleneimine and sonication for two minutes	YTZP	Higher dispersion with enhanced density, toughness	[50]
Deionised water	Polyacrylic acid (PAA) and vigorous stirring	Alumina	Increased bending strength due to MWCNTs pull-out	[51, 52]
Ethanol	Sodium hexametaphosphate and ultrasonication	SiC/YSZ	Improvement in fracture toughness with reduction in other mechanical properties	[31, 32]
SDS solution/distilled water/gum Arabica	Ultrasonic bath for 6 h followed by ball milling for 8 h	Alumina	GA and SDS mixture-based composite shown improved electrical properties	[53]
Distilled water/acetone/ethanol	Sonication and electrophoretic deposition	SiC	Higher dispersion and homogenous distribution of MWCNTs	[54]
Deionised water	PAA with ammonium salt with ultrasonication and mechanical agitation	YTZP	Fracture toughness of the samples increased with reduction in hardness was observed	[55]
Distilled water	Ultrasonication	Alumina and SiC	Superior hardness with high stability between SiC and MWCNTs were observed	[56]
Ethanol	Phosphate ester and cresolsulfonphthalein were surfactants with ultrasonication	MnO ₂	Electrical conductivity of the composites increased with promising electrochemical performance	[57]
Distilled water	Sonication for 1 h followed with stirring	Alumina	Enhanced electrical conductivity	[58]

distribute the nanotubes homogeneously. In this route, a nanofluid of CNTs is formed through dispersing CNTs into a solvent along with dispersing agent before addition of the ceramic to the solution and dried leading to enhanced CNT distribution. This is followed by processes such as drying and densification leading to the formation of a pellet. Goyny et al. utilised the colloidal route for fabricating MWCNT-reinforced epoxy composites by dispersing MWCNTs into an acetone solution before sonication for 30 min and slow addition to heated epoxy resin [47]. Likewise, Arunkumar et al. utilised colloidal route for dispersing the MWCNTs into nano-SiC with help of chemical dispersing agent and ethanol as medium [32]. Table 1 shows the colloidal route followed by various researchers for dispersing MWCNTs into the ceramic matrix. Even though various colloidal techniques are available for ready usage electrophoretic deposition is widely used due to the higher dispersion rate onto the ceramic matrices [48]. As reported

in Table 1, the dispersion medium and physical or chemical method is essential for enhancing the properties of MWCNT-reinforced ceramic matrix composites. Researchers utilise different surfactants which enhance the homogeneous distribution and dispersion of CNTs into the medium. These processes (sol-gel, colloidal and powder metallurgy) require external action and further densification processes to be effective. The in situ growth technique only allows the CNTs to be grown directly on the surfaces of ceramic matrices which reduces the steps compared to CVD. The following section details the in situ growth process of MWCNT-reinforced ceramic matrix composites (Fig. 4).

In situ growth

The powder technique reported by many researchers for fabricating CNT/Al₂O₃ and CNT/MgO-based composites through mixing or compacting ceramics with pre-grown CNTs powders followed by

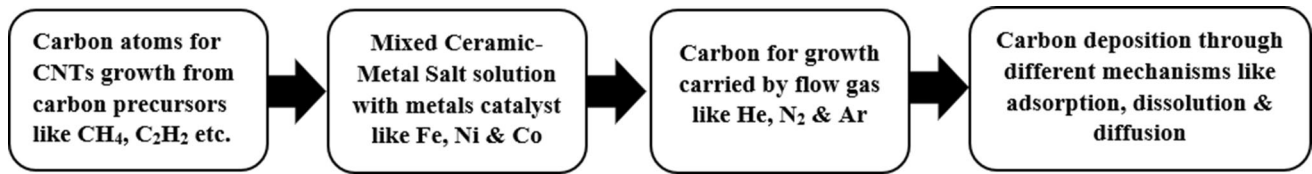


Figure 4 Schematics of in situ growth of CNTs onto the ceramics.

densification techniques. However, powder-based techniques require chemicals for enhancing diffusion such as surfactants, dispensing agents and physical methods like ball milling and ultrasonication to attain high order homogeneity. On the other hand, in situ growth of CNTs into the ceramics is advantageous as number of synthesis steps diminishes. This process utilises the nanopores on the ceramic body formed during sintering at high temperatures. These nanopores can be utilised as CNTs growing medium with help of a metal catalyst and a hydrocarbon source with a controlled growth temperature range of 700–1300 °C which must remain closer to the temperature of ceramic densification [59]. The growth technique enhances the homogenous dispersion of pure and high crystalline CNTs onto the ceramic substrate which tends to improve the mechanical properties and bonding between ceramics and CNTs. Chen et al. reinforced SiN by in situ growth of CNTs through chemical vapour infiltration which resulted in enhanced electrical conductivity as pores of SiN

were connected through CNTs [60]. Sun et al. prepared CNT-reinforced SiC_f/SiC composites through CVD by utilising a polymer impregnation pyrolysis process. The growth method involved weaving SiC fibres into a 3D fibre format with nickel, aluminium and lanthanum nitrates used as catalysts for growing the CNTs. Figure 5 shows the microstructure of the growth of CNTs at different stages of the synthesis. Figure 5a shows the SiC fibres and Fig. 5b the metallic catalysts covering SiC fibres in the preform. Figure 5c shows the feather growth of MWCNTs and Fig. 5d the CNTs on single strand of SiC fibres. The in situ growth enhanced the mechanical properties of SiC_f/SiC which were due to the CNT pull-out mechanism [61].

Ding et al. grew CNTs for electromagnetic wave absorption by dissolving FeCl₃ onto polysiloxanes in ethanol and calcined for 2 h in an argon atmosphere. The grown CNTs were characterised using Raman spectroscopy and it was determined that at 900 °C, the size of CNTs was higher and with increase in

Figure 5 Growth of CNTs: **a** SiC fibre surface, **b** SiC preform covered with metallic nanoparticles, **c** feature like CNT growth on SiC and **d** CNTs on SiC fibres adapted from the Sun et al. with permission [61].

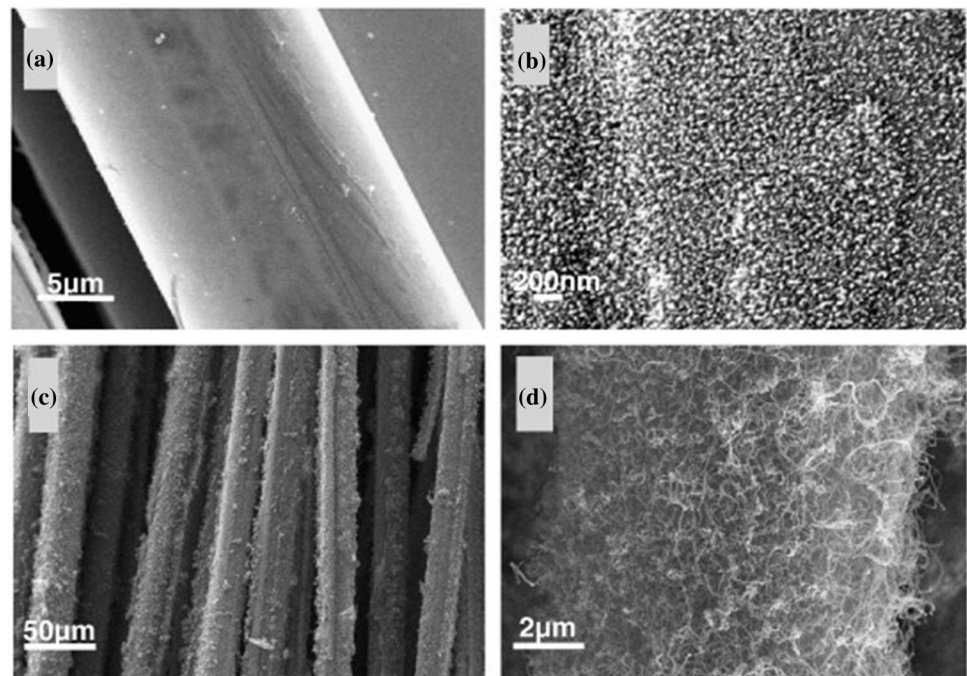


Table 2 Parameters and outcomes reported by researchers for *in situ* CNTs growth onto ceramics

Atmosphere	Time and temperature	Catalyst/medium	Precursor	Ceramic medium	Outcomes	References
Nitrogen	700 °C and 120 min	Tetramethyl ammonium hydroxide & Sodium hydroxide	Acetylene	Cobalt–zeolites	19.46% CNTs were produced with ID/Ig ratio 0.88	[18]
Argon (Ar)	800–1500 °C and 120 min	Iron(III) Chloride (FeCl ₃)	Ethanol	SiC/SiOC	Electromagnetic properties improved drastically	[62]
Argon/H ₂	780 °C for 30–120 min	Nickel(II) nitrate hexahydrate crystals (Ni (NO ₃) ₂ ·6H ₂ O)	Camphor & acetone	Porous ceramics	Prepared for air filtration and shown good reusability for filter applications	[63]
Hydrogen	700 °C for 20 min	(Ni (NO ₃) ₂ ·6H ₂ O)	Ethene	Sc ₂ Si ₂ O ₇	Electromagnetic waves absorption material was prepared with high bonded CNTs	[64]
Hydrogen	800 °C for 150 min	Fe (NO ₃) ₃ ·9H ₂ O	Distilled water + NaOH	TiB ₂	CVD method was utilised for CNTs growth onto TiB ₂ matrix where increasing Fe catalyst led to density and diameter increase in CNTs	[17]
Argon	550 °C and 60 min	Cobalt acetate tetrahydrate	Acetone	Si ₃ N ₄ + Lu ₂ O ₃	CNTs grown Si ₃ N ₄ composites shown enhanced shielding effectiveness for electromagnetic shielding	[60]
Nitrogen	2 h and 700 °C	Ferrocene	Cyclohexanol	α-Alumina	Modified ceramic–alumina composites have shown enhanced copper absorption from water	[65]
Hydrogen & nitrogen	750 °C and 60 min	Nickel nitrate, aluminium nitrate and lanthanum nitrate	Ethanol	SiC	Improved mechanical properties due to CNTs pull-out mechanism	[61]

temperature (1500 °C), there was a steep decrease in size of CNTs up to 6.84 nm [62]. Table 2 displays the parameters and outcomes observed by various researchers. These studies reported that the *in situ* growth technique can be a viable method for CNT/ceramic composite formation. Though the technique has been successful in growing CNTs over ceramic mediums as reported in Table 2. The technique was unable of growing CNTs in large surfaces.

Densification techniques

High-temperature sintering techniques are required for forming ceramic-based composites. The densification techniques enhance the bonding between the different powders present inside the composites leading to enhancement in mechanical and thermal properties. These treatments are critical for enhancing the interaction between the ceramics and CNTs by compressing and shaping the powders by supplying energy onto the matrices. This heat energy involved in the densification is utilised for breaking the bonds between the ceramics for superior bonding

with the reinforcing material. Though the concept of the densification has been followed by different techniques, i.e. conventional and non-conventional to enhance the density of the composites. There are still developments being considered to achieve a high order densified sample. The sub-section below provides a brief on various densification techniques including the famed techniques like pressureless sintering, microwave-assisted sintering, hot press technique and SPS.

Pressureless sintering

Pressureless sintering is a thermal technique for densifying ceramic composites [38]. This sintering technique utilises the application of uniaxial hydraulic pressure to compress CNT/ceramic composites pellets followed with sintering of the pellets high-temperature furnace under varying temperature and atmospheric conditions depending on the material compressed as shown in schematics in Fig. 6. In case of CNT/ceramic nanocomposites, the atmospheric condition is preferred to be in vacuum environment with flow gas being He, Ar and nitrogen rather than oxygen owing to the lower oxidation of CNTs in room environment [32]. The sintering temperature of the CNT/ceramic composites depends on the dominating material or matrix material in the composites, i.e. ceramics in case of ceramic/CNTs.

Zhang et al. improved the flexural strength of an alumina substrate by reinforcing with MWCNTs through the pressureless sintering process. MWCNTs were compacted into alumina at different compositions and uniaxially pressured and sintered at 1500 °C for 2 h using a tubular furnace with an argon atmosphere. The 1% MWCNTs-reinforced alumina composites demonstrated ~ 540 MPa whereas pure alumina exhibited ~ 400 MPa for similar conditions [66]. Ahmad et al. fabricated MgO tailored alumina with 2% MWCNTs using pressureless sintering with an external pressure of 40 MPa and sintered at 1600 °C for 1 h under vacuum environment. The fabricated composites have shown a meagre mechanical response compared to hot pressed

samples ostensibly due to poor microstructure formation. However, the pressureless sintered samples shown a slight increase in the mechanical properties compared to hot press technique. Figure 7a–f illustrates the fractured surface of pressureless sintered composites which shows numerous open pores representing the reduced densification on the surfaces (Fig. 7a–c) whereas the pores are reduced with addition of MgO along with MWCNTs (Fig. 7d–f) [67].

Likewise, Bakhsh et al. utilised pressureless sintering to fabricate CNT-alumina nanocomposites at 1700 °C for 15 min under an argon atmosphere and reported the fracture toughness of nanocomposites increased by 10% due to crack deflection, better dispersion and bridging mechanisms of the CNTs. However, densification of the composites decreased with increasing CNT content, leading to increased pore density and reduced hardness [68]. Further studies on the CNT/ceramic composites using pressureless sintering also exhibited improvement in the fracture toughness with reduction in density and hardness properties resulting in need of new sintering technique for increasing density along with reduction in porosity.

Hot press technique

The hot press technique is a non-conventional sintering method which involves high pressure for compaction the powder with subsequent high-temperature sintering [69]. The technique is also known as pressure-assisted sintering where pressure compresses the powder of CNT/ceramic into densified pellets or films. This technique increases the density of the composites due to the action of high pressure and temperature leading to enhanced bonding between the ceramics and reinforcement material which could increase the mechanical properties [70]. Mullite/MWCNTs-based composites were fabricated using the hot press method at 1600 °C which exhibited a 10% enhancement in bend strength and 78% increase in fracture toughness compared to monolithic mullite [71]. Numerous studies on the hot

Figure 6 Schematic of the pressureless sintering process.

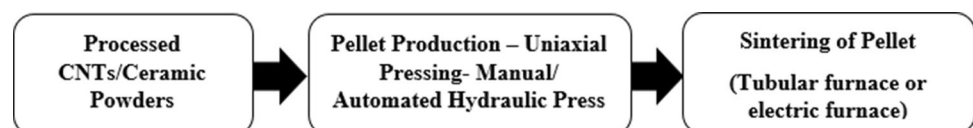
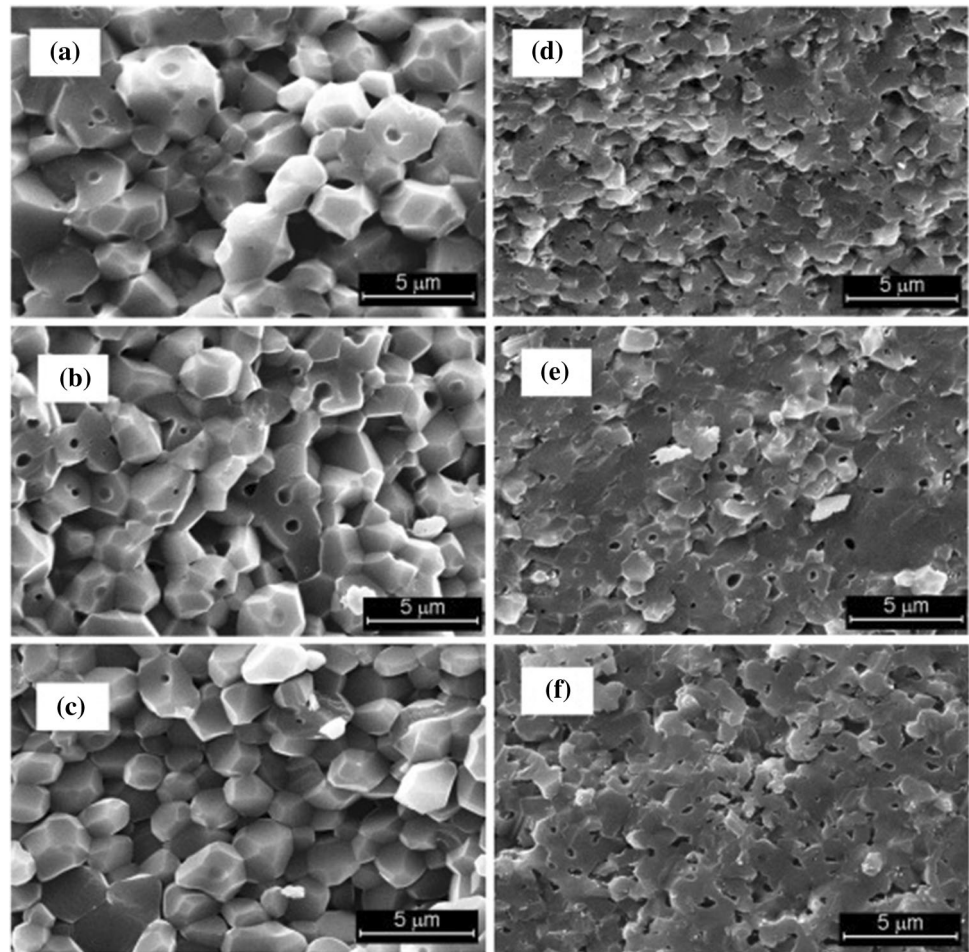


Figure 7 Microstructure of pressureless sintered: **a** Al_2O_3 , **b** 300 ppm $\text{MgO}-\text{Al}_2\text{O}_3$, **c** 600 ppm $\text{MgO}-\text{Al}_2\text{O}_3$, **d** nanocomposite Al_2O_3 / MWCNTs, **e** 300 ppm MgO nanocomposite and **f** 600 ppm MgO nanocomposites adapted from Ahmad et al. with permission [67].



pressing of MWCNTs and ceramic nanocomposites have been carried out by researchers and few are reported in Table 3. The technique is said to enhance the density of the composites compared to the pressureless sintering and further provide with higher bonding between CNTs and ceramics due to continuous pressure being applied. The formed nanocomposites are said to be crack-free and dense owing to constant pressure and temperature. However, the technique could also damage the microstructure of the nanocomposites which may result in affecting the bonds between CNTs and ceramic present in the nanocomposites. Further, report studies till date have shown that the incorporation of CNTs into ceramics through hot pressing techniques only led to partial improvement in the properties. Therefore, there is a need to improve the densification behaviour of composites.

Microwave-assisted sintering

Microwave-assisted sintering is an emerging pressureless sintering method for fabricating CNT-CMCs with the help of electromagnetic fields (microwave). In this technique, powder metallurgy is utilised for processing CNT/ceramic pellets through physical and chemical process processes and pressed using hydraulic press at high pressure. Microwaves irradiate in the furnace allow ceramics to generate heat within the matrix due to joule's heating effect leading to internal interaction between the reinforcement and ceramics as illustrated in Fig. 8. Egorov et al. reported the fabrication of alumina ceramics reinforced with MWCNTs using microwave sintering at a temperature of 1500–1600 °C with a heating rate of 50–100 °C/min with no hold time and determined that up to 2.5 wt.% of CNTs did not affect the thermal stability of the composites during rapid sintering of alumina [81]. Similarly, Ghobadi et al. densified

Table 3 Various research on hot pressing technique of CNT/ceramic nanocomposites

Ceramics	Temperature/pressure	Processing technique	Outcomes	References
SiN	1700 °C for 60 min with 2 MPa	Ball milling & sonication	MWCNTs survived after 2 MPa with 15–37% improvement in mechanical properties	[69]
Fe–Al ₂ O ₃	1355–1535 °C for 15 min	Selective reduction	High dense composites with marginal increase in Fracture toughness	[72]
BAS (Barium Aluminosilicate)	1600 °C for 60 min	Ball milling & sonication	Reinforcement with MWCNTs increased mechanical properties than whiskers	[73]
Metal–magnesium aluminate spinel	1200–1500 °C for 20 min with 43 MPa	Ball milling & sonication	Hot press temperature was not enough to densify the composites	[74]
Alumina	1800 °C for 60 min with pressure of 40 MPa	Ball milling & sonication	Wear resistance of composites increased up to 4% CNT but after 4% mechanical properties reduced	[75]
Alumina	1500 °C under vacuum at 40 MPa	Purification of MWCNTs and ultrasonication	Percolation increased by 3.89 wt%, enhanced electrical properties	[76]
ZrO ₂	1300 °C for 30 min	Colloidal route	Decreased hardness and increased elastic resistivity of 13 order of magnitude compared to monolithic zirconia	[77]
SiC	2000 °C at 25 MPa for 60 min	Ultrasonic shaker	Bending strength of the composites and fracture toughness increased over 10% than monolithic SiC through same purpose due to strengthening and toughening role	[78]
Borosilicate glass	750 °C in vacuum for 60 min at 5 MPa	Ultrasonication for 4 h & calcination at 350 °C	Monolithic ceramics were found to have higher mechanical properties than fabricated composites	[79]
TiB ₂ –SiC	1800 °C at 30 MPa	Ball milling with different CNTs compositions	Addition of CNTs did not significantly affect the composites in fracture toughness however, Vickers' hardness reduced to 21 GPa	[80]

alumina/MWCNTs composite through microwave sintering coated with boehmite nanoparticles. The samples were cold pressed at 180 MPa and sintered at 1520 °C for 45 min. The reinforced CNTs improved the fracture toughness by ~ 37% with highly relative density [82]. However, like the pressureless sintering technique the density of the composite reduced with increase in CNTs content and further addition of CNTs led to decreased mechanical properties. Even though there have been studies on microwave-assisted sintering in different ceramic and metal matrix composites, the studies on CNT-reinforced ceramics are limited and further research is required.

Spark plasma sintering (SPS)

SPS is an emerging sintering technique for fabricating highly dense ceramics-based composites with help of electric pulse and continues pressure and

temperature. The technique utilises three factors, i.e. pulse electric current, temperature and pressure to enhance the densification on the samples as represented in Fig. 9 where typically graphite-based die is employed with internal water-cooling circuits and DC pulse generator to generate heat. The SPS utilises uniaxial pressure and temperature simultaneously to increase the compression rate of the composites to enhance densification through providing the bond material with joule law of heating onto the surfaces [31]. The Joule's heating of the composites helps in improving the bond between the ceramics and CNTs. Various excellent reviews have been published on fabrication of CNT/ceramics through SPS processes along with its merits and demerits [38, 84]. Zhang et al. utilised SPS process for fabricating SWCNTs-reinforced alumina composites with temperature of 1150 °C and pressure of 63 MPa for dwell time of

Figure 8 Microwave-assisted sintering furnace model adapted with permission [83].

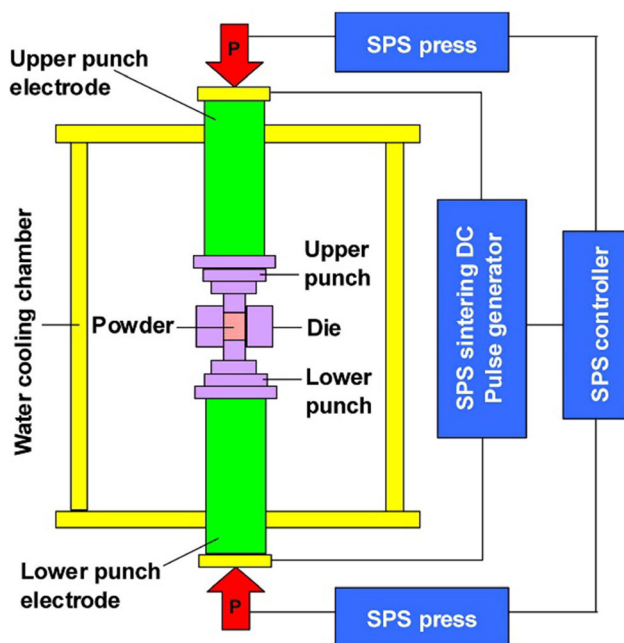
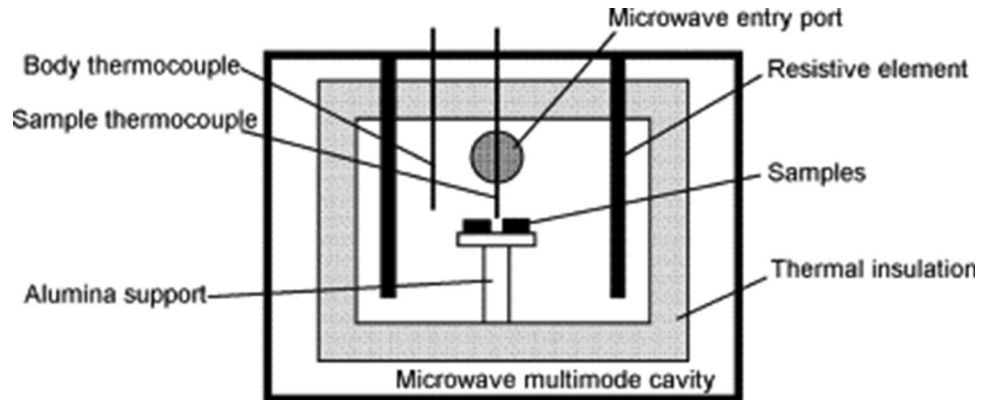


Figure 9 Schematics of spark plasma sintering adapted from Zhang et al. with permission [84].

3 min which enhanced the electrical conductivity and fracture toughness from 3.3 to 9.7 MPa.m^{0.5} [85, 86]. These works inspired various researchers to concentrate on the SPS technique for fabricating CNT-reinforced ceramics as reported in Table 4. However, reports from Zhang et al. showed agglomeration of the CNTs and recent studies tried different powder processing techniques to avoid this problem [85, 86]. Arunkumar et al. have studied the fabrication of MWCNT-reinforced SiC and YSZ through SPS at various compositions by dispersing MWCNTs into the ceramic matrix through techniques like ultrasonication and ball millings process reported enhancement in fracture toughness of the nanocomposites [31, 32]. In contrast, Lopez et al. utilised SPS

with carbon-based (graphene) reinforcement with YSZ and reported improved the fracture toughness and hardness along with significant improvement in electrical and thermal properties which contradicted hardness results reported by Arunkumar et al. [87]. Likewise, Momohjimoh et al. compared MWCNTs and SiC-doped alumina-based matrix and achieved lower-density composites with higher electrical conductivity (~ 101.118 S/m) [88]. Arunkumar et al. higher weight percentage MWCNTs (15 wt.%) study with nano-sized SiC reported an enhancement in the fracture toughness, but a drastic drop in hardness and impact strength [32]. In summary, an extensive study on SPS is still necessary to understand the optimisation of heat and properties of MWCNT-reinforced ceramics. Though SPS is an emerging technique, it's been widely utilised by researchers for fabricating composites with higher density. However, the technique tends to produce complex geometries and fabrication of large models through the technique is still impossible. So, there is a need for advance techniques which can produce complex models.

Additive manufacturing of MWCNTs composites

Even with significant advancements in the fabrication of MWCNT/ceramics composites, some investigations reported a partial increase or drastic drop in the density and other properties. Therefore, demand for advanced fabrication techniques which impart high densification and superior mechanical properties is highly sought-after. Additive manufacturing (AM) or 3D printing is one of the recent manufacturing processes which is one of the most promising future

Table 4 Various reports on spark plasma sintering for ceramic/MWCNTs composites

Ceramics	Temperature/pressure	Powder processing technique	Outcomes	References
Al ₂ O ₃	1500 °C with 20 MPa	Ball milling and drying	Negative effect on densification and better mechanical properties	[89]
Al ₂ O ₃	1375 °C in vacuum for 5 min	Two-step process with ultrasonication	Fracture toughness of the composites increased by 2.5%	[90]
Al ₂ O ₃	1000–1200 °C and 5–20 MPa	Sonication and slurry technique	Porous composite membrane was fabricated, and best combination was revealed	[91]
Al ₂ O ₃ –SiC	1500 °C for 10 min at 50 MPa	Slurry and ultrasonication	Increase in electrical conductivity making it suitable for electric discharge machine	[92]
Al ₂ O ₃ –SiC	2000 °C and 50 MPa with dwell of 10 min and Ag atmosphere	Sonicator and slurry drying	Increase in densification and fracture toughness	[93]
SiC	1800 °C and 50 MPa with dwell time of 10 min	Ultrasonication and rotary evaporator	Improvement in fracture toughness up to 5 wt.% CNTs addition	[32]
SiC	2000 °C and 73 MPa	Aqueous slurry and drying	Density and hardness increased with CNTs pull-out mechanisms	[94]
SiC	1800 °C	Diffusion process	Outstanding thermal and electrical properties were increased with CNT addition	[95]
YSZ	1200–1500 °C at 50 MPa	Ball milling	High CNTs dispersion could increase fracture toughness	[96]
YSZ	1350 °C and 50 MPa with dwell of 10 min	Ultrasonication and rotary evaporator	Fracture toughness increased by 21% than YSZ ceramic	[31]
YSZ	1300 °C and 30 MPa	Ultrasonication and ball milling	Addition of CNT increase densification of YSZ ceramic	[97]

avenues for research involving the commercial markets of the aerospace for preparing composites with high structural stabilities [98]. AM is a viable process for manufacturing complex customised prototypes with various developments in the machines and printable materials for small-scale production. Various research has been carried out on the AM of composites and its products [99, 100]. Likewise, research on 3D printing of CNT-based components is also being carried out around the world. Yu et al. created a novel 3D printing procedure for fabricating CNT-based micro-supercapacitors which uses solid-containing CNT-ink. The printed micro-capacitors have shown excellent stability and significant areal capacitance [101]. Ye et al. printed CNT-reinforced thermoplastic polyimide composites by producing filament as illustrated in Fig. 10 for usage in aerospace structures by investigating the tensile and bending properties. The study reported that the CNT-reinforced composites showed superior performance and with further optimisation the complex aerospace components can be fabricated [102].

Li et al. printed carbon-reinforced polylactic acid composite and reported that the printed material showed increased interfacial strength along with 13.8% and 164% increased tensile and flexural strength than original carbon-fibre-reinforced samples [100]. 3D printing of C/SiC composites material is one of the greatly researched areas with various researchers focusing on SiC-based 3D printable materials. Zhu and co-workers developed carbon-fibre-reinforced SiC composites by combining 3D printing and infiltration processes. The green bodies (unsintered) were fabricated through 3D printing using phenolic resin-coated carbon fibre composites and vacuum infiltration was used to fabricate C/C preforms. Liquid silicon was used for infiltrating C/C_f preforms to attain the final C/SiC composites [103]. The number of recent studies on 3D printing ceramics has increased exponentially over the last few years, mirroring trends seen metals and polymers [104]. 3D printing technologies for ceramics have been classified based on the feedstock utilised for printing, such as: slurry, powder or bulk techniques [105]. SiC reticulated porous ceramics were prepared by 3D

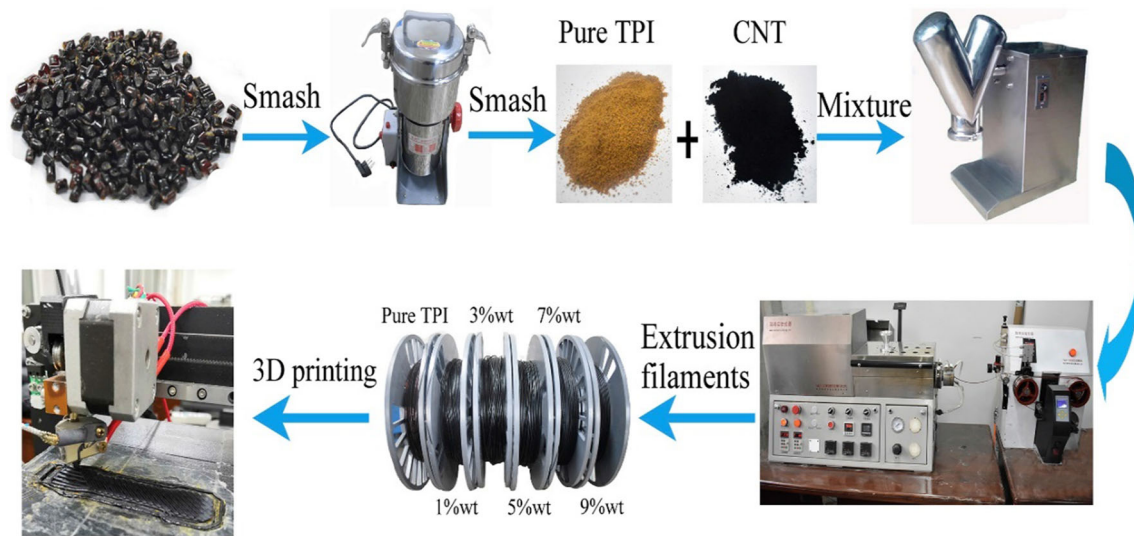


Figure 10 3D printing filament preparation schematics of CNT/TPI composites adapted from Ye et al. with permission [102].

printing with help of a polyvinyl alcohol mould, before the slurry was gel-casted into the surfaces [106]. Allahverdi and co-workers successfully printed ceramic-based transducers in different shapes through fused deposition modelling (FDM) technique, with help of polymers/piezoelectric ceramics (lead–zirconate–titanate and lead–magnesium–niobate) composites as feedstocks and demonstrated enhanced electrical properties [107]. Also, Blazdell et al. used ink-based feedstock for 3D printing of ZrO_2 and TiO_2 ceramics using small volume ink

fractions led to poor surface quality [108]. To the best of knowledge and despite recent developments in the field, there have been no studies related to additive manufacturing of MWCNT/ceramic-based composite products. Further studies are necessary in AM for incorporating CNT-based ceramic composites for engineering or biomedical fields. Prior to developing advanced manufacturing process, there is a need to optimise current fabrication techniques to improve the densification behaviour and properties of the MWCNTs/ceramic nanocomposites. Table 5 details

Table 5 Advantages and disadvantages of various fabrication techniques

Fabrication techniques	Type of processing techniques	Advantages	Disadvantages
Pressureless sintering	Powder metallurgy	Easy to utilise Customisable to user	Lack of pressure during sintering leads to cracks and reduction in density
Microwave-assisted sintering	Powder metallurgy	Complete bonding between composite elements is possible Increased sintering effect	Lack of pressure during sintering leads to cracks and reduction in density
Hot pressing method	Powder metallurgy, colloidal route	High densified samples can be achieved Even surface throughout the composites	Chance in microstructure Propagation of cracks
Spark plasma sintering	Powder processing techniques	Highly dense sample with less cracks can be achieved Durability of samples are high	Small-scale samples Traces of carbon in analysis due to presence of graphite die
Additive manufacturing	Filament-based technique Powder-based technique	Any shape can be achieved as per user needs Optimised to enhance density and other properties	Still under development Time taken for final products are higher Expensive

the summary of advantages and disadvantages of different fabrication techniques reported in this review. By optimising the disadvantages of current fabrication techniques could lead to enhancement of the various properties associated with MWCNTs-reinforced ceramic composites.

Properties of MWCNTs/ceramic composites

Properties of MWCNT-reinforced ceramic composites have been a fascinating area of research with numerous groups focusing on enhancing the properties of the ceramics for various applications. Though there have been many reviews on the properties of the MWCNTs-reinforced ceramics composites including details on the failure mechanisms for diverse applications. Nevertheless, there is still a need to understand failure mechanisms and other physical behaviours in detail [38, 109]. The subsequent sub-section provides a brief overview on the properties and studies conducted on MWCNT-reinforced ceramic composites.

Thermal properties

Thermal behaviour of the materials plays an essential role in determining its usage in high-temperature applications such as aerospace and nuclear environments [38]. Studies have shown that the reinforcing CNTs can enhance the thermal properties of the material. This phenomenon is especially utilised for polymer and ceramic-based materials which are used to form composites designed to enhance mechanical and thermal properties. Use of ceramic-based composites has enhanced aerospace applications due to their superior thermo-mechanical properties compared to Ni-based superalloys and aluminium alloys which are currently the materials of choice in aerospace engineering for structural and turbine components. Basic thermal properties of any material involve conductivity, diffusivity, heat capacity and thermal expansion which highly depend on parameters like bulk density, sintering temperature and pressure on surface of the composites [110]. Kumari et al. prepared CNTs-alumina composites at varying composition and sintering temperatures and reported that the diffusivity of the material increased by 60% compared to monolithic alumina with 7.39 wt.% of

the composites for samples sintered at 1550 °C [111]. However, Ramachandran et al. reported a reduction of the diffusivity value with increase in CNT content with 1 wt.% showing the highest diffusivity value compared to the monolithic alumina fabricated via same technique [33]. Both research teams prepared the composites through SPS process with sintering temperature and dwell time playing a huge role in determining the diffusivity of the composites. However, the preparation aspect of the CNT/alumina differed from one another. On the other hand, Shah et al. fabricated a hybrid composite of graphene/CNT/alumina through SPS (as shown in Fig. 11) and reported enhancement in the conductivity for 1 wt.% CNTs and 0.4 wt.% graphene [112]. However, with SiC and CNTs as reinforcements in hybrid composites, thermal conductivity dropped at higher SiC content resulting in inferior thermal properties than CNT-reinforced composites [92].

Although there have been many literatures focusing on the thermal properties of the composites reinforced with CNTs as reported in Table 6, most of them concentrated on the effect of the wt.% of CNTs and sintering temperature on the thermal properties, none reported on the mechanism which caused this enhancement [113]. According to Ramachandran et al., the twirled structure of the MWCNTs incorporated into the alumina matrix may block the travelling phonons. This leads to the reduction in thermal conductivity along with agglomeration of MWCNTs onto the matrix could reduce the phonon scattering due to Van der Waals forces of attraction between the MWCNTs which was also observed by Hazel et al. [33, 114]. In contrast, Kumari et al. stated that the uniform dispersion of the MWCNTs onto the ceramic matrix could be reason for the increase in thermal conductivity [111]. Although researcher like Kumari et al. reported that homogeneous distribution of MWCNTs on the ceramic composites could have been the mechanism behind enhancing the thermal behaviour but that could be the basic mechanism behaviour the improvements. It is noteworthy that the mechanism proposed by Ramachandran et al. and Hazel et al. concentrates on the phonon scattering which is still an area of ongoing research.

Mechanical properties

Mechanical properties including hardness, fracture toughness and flexural strength are desirable

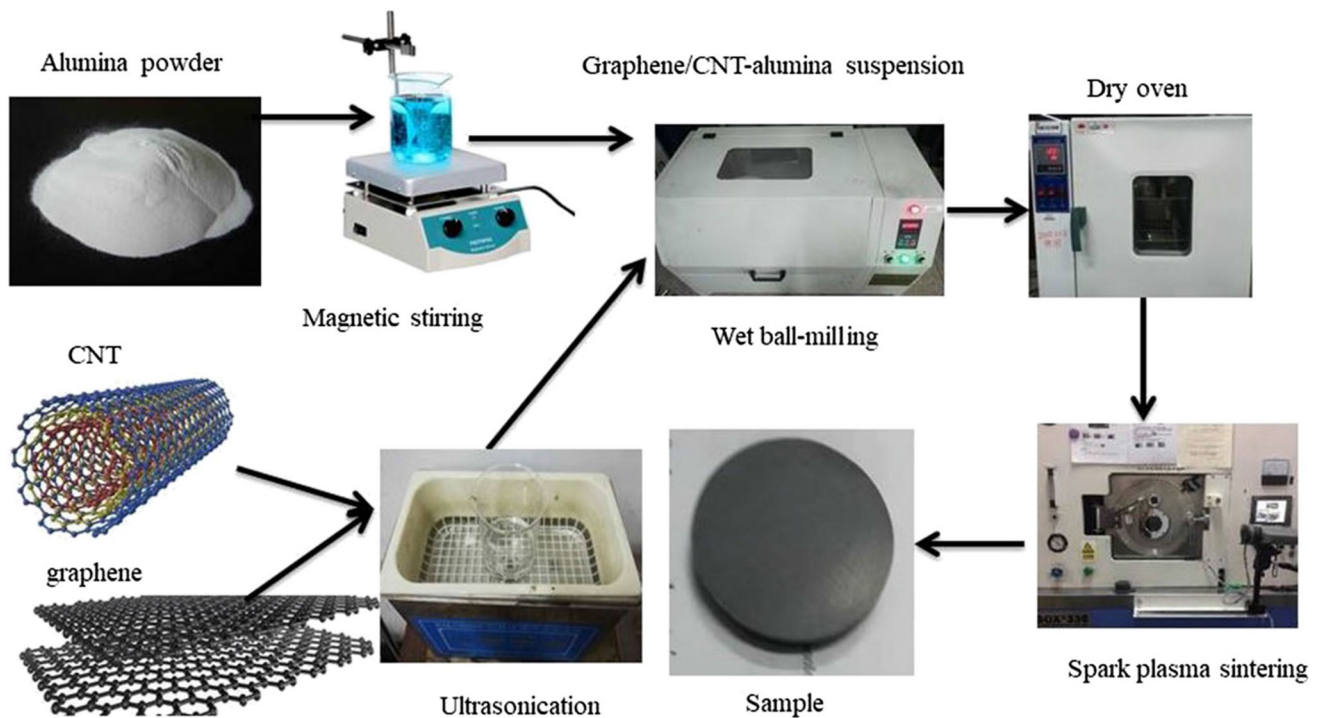


Figure 11 Graphene–CNTs/alumina nanocomposites preparation by Shah et al. adapted from Springer with permission [112].

properties for any aerospace materials and ceramics being brittle in nature requires reinforcement to enhance its brittle behaviour. CNTs are one such reinforcement which could be used to enhance the fracture toughness of the ceramics. Several literature studies have focused on the effect of CNTs on the mechanical properties of ceramics and it has been determined that appropriate CNTs compositions, densification techniques and homogenous distribution of CNTs onto the ceramic matrix are required to enhance the properties of the composites [31–33]. However, there have been also anomalies in mechanical properties noted by various researchers which highlights the importance of CNT/ceramic composition. Literatures on CNT composites with SiC, YSZ and alumina with various compositions showed excellent properties at 1 wt.% MWCNT reinforcement [31–33]. Figure 12 illustrates the indentation on the surface (Fig. 12a) and fracture mechanisms like pull-out and bridging (Fig. 12b) on the alumina/MWCNTs surfaces which were dominating failure mechanisms. This was also reported in a previous work of MWCNTs/YSZ nanocomposites which have shown formation of pores with increase in MWCNTs due to agglomeration on the surfaces and a reduced heating effect on the sintered surfaces

[31]. Ghobadi et al. prepared alumina-based CNT composites using cold pressing technique and reported a flexural strength of 465 ± 31 MPa whereas Ramachandran et al. reported flexural strength of 515 ± 33 MPa using SPS [33, 121]. These results from the researchers prove that the mechanical properties depend on the fabrication technique and bulk density of the composites.

Table 7 reports various mechanical properties adapted from different investigations which use CNT reinforcement at different compositions to enhance the properties of the ceramics. As reported in Table 7, most studies reported the MWCNT reinforcement onto ceramics enhanced the mechanical properties to certain extent. Most of the studies indicated that mechanisms like fibre pull-out, fibre bridging and crack deflections on material surfaces due to the presence MWCNTs on the composites were responsible for the enhancement of the mechanical properties. In contrast, some show that the mechanical properties of the CNT-reinforced composites also depend on the densification techniques and porosity of the material. Arunkumar et al. reported increase in CNTs content resulted in increase in the porosity of the composites as there was a transition from open pores to closed pores as illustrated in Fig. 13.

Table 6 Thermal properties of ceramic/CNT composites reported by various researchers

Composite	Preparation technique	Densification Technique	CNT content	Diffusivity mm ² /s	Thermal conductivity (W/mK)	Heat capacity J/gK	References
MWCNT/ Al ₂ O ₃	Powder processing	Spark plasma sintering at 1500 °C	1 wt%	8.77	29.62	0.87	[115]
MWCNT/ Al ₂ O ₃	Powder processing	Spark plasma sintering	7.39 wt% and 19.10 wt%	13.98 at 25 °C and 5.24 at 25 °C	90.44 at 100 °C and 36.77 at 100 °C	2.09 at 60 °C and 3.31 at 60 °C	[111]
SWCNT/ Al ₂ O ₃	Powder processing	Spark plasma sintering	10 vol% and 15 vol%	–	11.4 and 7.3	–	[116]
MWCNT/ TiN	Powder processing	Spark plasma sintering	1 Wt%	3.25 at 375 °C	14.75 at 375 °C	0.9 at 375 °C	[117]
MWCNT/ SiO ₂	Hot pressing	Spark plasma sintering	5 vol.% & 10 vol.%	–	3.48 ± 0.01 and 4.08 ± 0.01	–	[118]
CNT– graphene/ Al ₂ O ₃	Powder processing	Spark plasma sintering	0.4 Wt% graphene and 1Wt% CNT	7.56	23.25	1.0606 and 1.04691 at 300 °C	[112]
C/SiC	Laser-assisted chemical vapour infiltration (LA-CVI)	Vacuum impregnation	0.5 Wt%	–	150.42	–	[92]
ZrB ₂ -20 SiC– CNT	Powder processing	Spark plasma sintering	10 vol%	0.240 at 50 °C 0.121 at 1200 °C	61.8 at 50 °C 51.4 at 1200 °C	0.485 at 50 °C 0.802 at 1200 °C	[119]
ZrB ₂ -CNT	Powder processing	Spark plasma sintering	10 vol%	0.204 at 50 °C 0.130 at 200 °C	48.9 at 50 °C 46.6 at 1200 °C	0.415 at 50 °C 0.632 at 1200 °C	[120]

However, increase in MWCNTs content could lead to reduced solid state diffusion which could inhibit the transition of open pores. This could result in reduction of bulk density which weakens the composites leading to poor mechanical properties [31]. This effect was also previously observed on the alumina/MWCNTs composites sintered through SPS providing evidence of pores transition from open to closed at temperature of 1050 °C [122]. Though the concept of open and closed pores is provided in detail with only these two researchers there is a very low possibility that the drop in mechanical properties with increase in the MWCNTs content could be a mechanism for the reduction. On the other hand, it is feasible to state that the porosity mechanism could have an effect at high MWCNTs content but possibly would not be a governing mechanism.

Like mechanical properties, tribological behaviour also plays a crucial role in determining the service life of the structural materials in various applications [33]. Various reinforcements and coatings are being considered to reduce the friction and wear behaviour of the materials [42]. Ceramics-based composites have limited usage in contact-based applications such as bearings, valves and nozzles owing to its high frictional and wear behaviour [128]. Thus, there is a need for protective coatings and reinforcements to enhance the wear behaviour of the ceramic matrix composites [42, 75]. CNTs are one of the major reinforcement materials in the wear applications owing to its failure mechanisms and lubricating behaviour [33]. Ramachandran et al. studied the wear behaviour of the alumina/MWCNTs composites under varying load and time and determined that wear depended

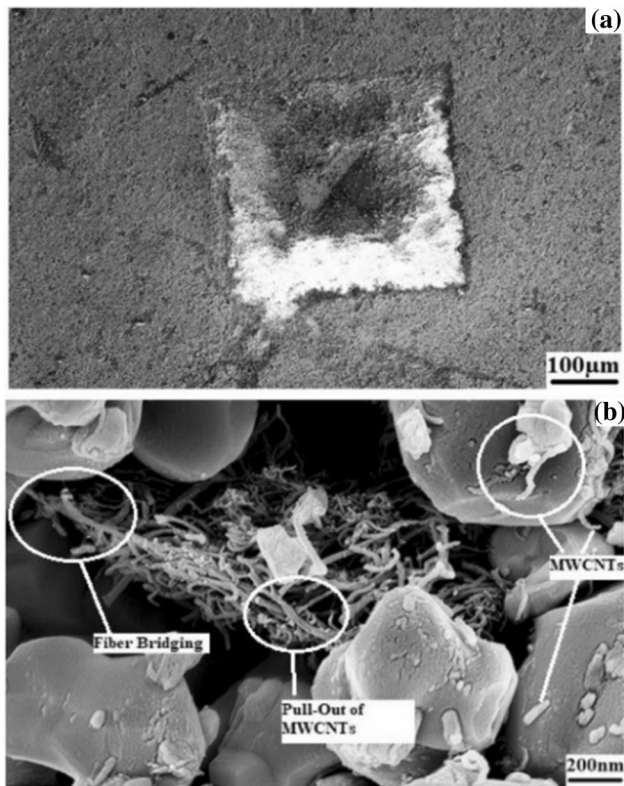


Figure 12 Surface indentation and fibre pull-out on $\text{Al}_2\text{O}_3/\text{MWCNT}$ nanocomposites adapted from K. Ramachandran et al. with permission [33].

on the MWCNTs content, time and load as shown in Fig. 14 [33]. However, Sharma et al. provided a contradictory report for the alumina/MWCNTs fabricated through SPS technique showing a lower wear rate at 3 vol.% MWCNT reinforcements compared to 1 vol.% MWCNT which increased the wear rate at 5 vol.% [46]. Other sources have provided evidence of reduction on the wear behaviour of the composites by reinforcing CNTs onto the ceramic bodies which were independent of densification techniques [75]. The lubricating behaviour of MWCNTs along with its different mechanisms including fibre pull-out and bridging on the surfaces enhanced the wear behaviour of the composites until its threshold limit. But with an increase in CNT content, increased agglomeration on the composite surfaces inhibited the wear behaviour [33]. Figure 15 adapted from Hazel et al. illustrates the microstructure of the composites reported that the failures on the wear run surfaces were due to the debriefs and scaling formed on the composites. The colour variation in the microstructure could have been due to the formation of mosaic like grains with varying grain sizes on the light

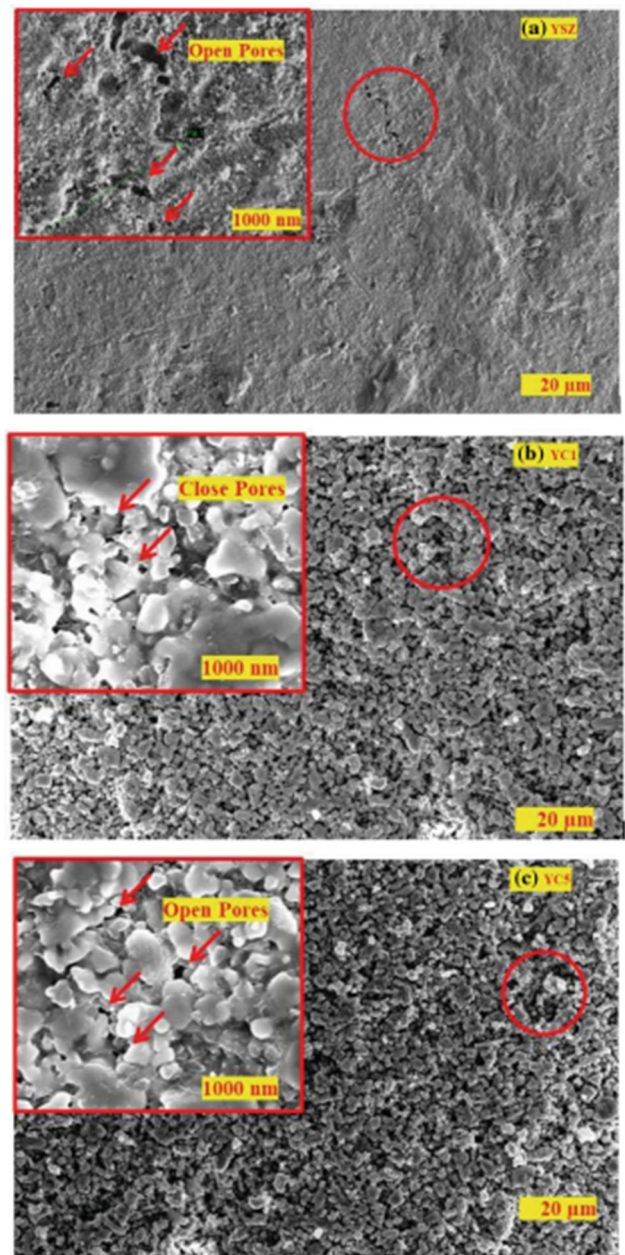


Figure 13 Open pores and closed pores transition with MWCNTs content on YSZ adapted from Arunkumar et al. with permission from corresponding author [31].

patched area which could avoid large wear whereas dark regions are stated to be mostly exposed with high amount of debris and scaling than the light region [114]. On the other hand, Ramachandran et al. stated that the load and sliding distance could have had higher impact on the wear behaviour as the materials temperature could increase inhibiting interfacial bonding between the CNT and alumina nanoparticles [33]. Few of the highlighted work in

Table 7 Mechanical properties of CNT/ceramics reported in various literatures

Ceramics	Preparation techniques	Densification technique	CNT content	Hardness (GPa)	Fracture toughness (MPa.m ^{0.5})	Flexural strength (MPa)	References
Al ₂ O ₃	Colloidal route	Spark plasma sintering	1 wt.%	17.26 ± 0.4	Indentation 5.6 ± 0.3 SEVNB 5.2 ± 0.6	515 ± 33	[33]
Al ₂ O ₃	Suspension technique	Isotropic cold pressing	1 vol.%	–	Indentation 4.2 ± 0.2	465 ± 31	[121]
Al ₂ O ₃	Colloidal route	Hot pressing	1 wt.%	–	4.0 ± 0.5	536 ± 44	[51]
Al ₂ O ₃	Powder processing	Spark plasma sintering	0.8 wt.%	–	3.85	–	[46]
Al ₂ O ₃	Precursor technique	Spark plasma sintering	2 wt.%	–	3.6	315	[123]
Al ₂ O ₃ –Whisker SiC	Powder processing	Hot pressing	1 wt.%	20.59	3.8–4.5	700 ± 50	[124]
SiC	Colloidal route	Spark plasma sintering	15 wt.%	8.81	10.21	–	[32]
SiC	Powder processing/tape casting	Hot pressing	0.5 wt.%	23.8 ± 2.5	6.1 ± 0.6	755 ± 106	[125]
SiC	Powder processing	Aqueous tape casting	0.5 wt.%	19.77 ± 2.5	4.38 ± 0.15	706.7 ± 16.9	[126]
Al ₂ O ₃ –SiC	Colloidal route	Cold pressing	2 wt.%	22 ± 2.0	6.5 ± 0.4	–	[56]
SiC	Powder processing	High pressure reaction sintering	3:2 ratio	20 ± 2.0	6.8 ± 0.8	–	[127]
YSZ	Colloidal route	Spark plasma sintering	1%	12.96 ± 0.3	Indentation 6.58 ± 0.3 SEVNB 5.22 ± 0.3	–	[31]
YSZ	Powder processing	Spark plasma sintering	5%	–	15.2 ± 0.4	–	[96]

this section explore the mechanical and tribological behaviour of MWCNTs-reinforced ceramic composites along with its failure mechanisms and quantity of CNTs being utilised for the composites. Although there has been constant enhancement in the properties with CNTs reinforcements, there is still a necessity to correlate the failure mechanisms with process parameters including densification time, temperature and testing environments which are yet to be developed in future along with few simulation tools.

Applications of MWCNT/composites

Nanocomposites are of great interest for various applications owing to their superior properties, surface to volume ratio and resistibility [33]. Currently,

various research organisations such as NASA, JAXA and ISRO are actively working on the development of nanocomposites for various structural and aerospace applications [129]. Applications related to CNT-reinforced ceramic nanocomposites are discussed in detail in this section.

Industrial & automobile applications

Nanocomposites have initiated a revolution of replacing the traditional alloys on the automobile composites [130, 131]. Steel-based components on automobiles provided high corrosion resistance and strength, but for high-performance vehicles, there is a need to reduce the weight/power ratio. Ceramics could act as possible replacement due to its lower weight to thrust ratio and high thermo-mechanical

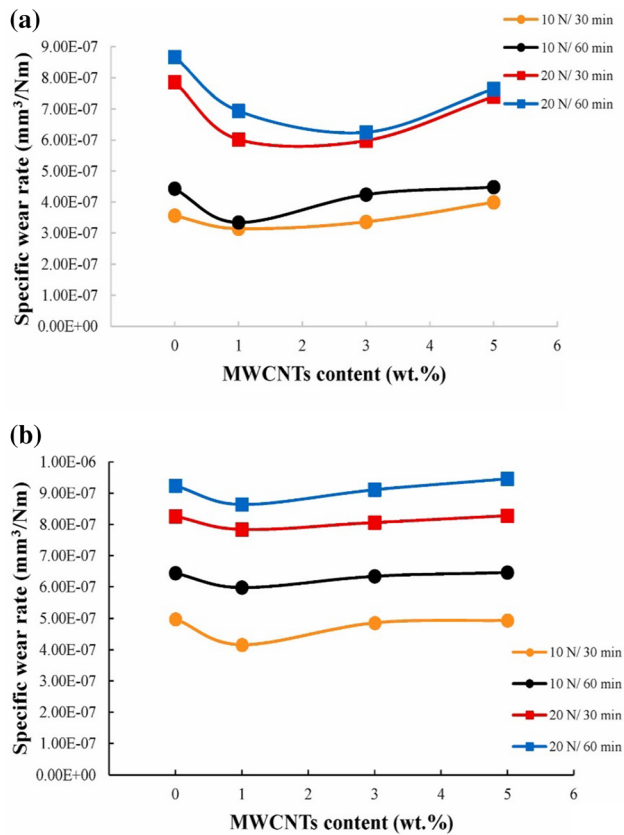


Figure 14 Wear behaviour of alumina/MWCNTs composites at varying sliding distance of **a** 100 m and **b** 200 m adapted from K Ramachandran et al. with permission [33].

behaviour. On the other hand, owing to its brittle behaviour, the life expectancy of ceramics is lower when compared to steel-based alloys [132]. Hence, using CNTs to reinforce ceramics enhances its fracture toughness to values greater than the highest-

grade steel components. This allows the ceramics to act as possible replacement in automobile applications by providing with hardness, lightweight and high temperature capabilities. Reinforced ceramics have potential to be used in engine parts, brake discs, valves, cylinder liners, spark plug, sensors, isolators, filters and pistons—all are subjected to higher load and wear which could lead to deformation and wear and are examples where ceramic-based nanocomposites could be efficient [130, 131]. Along with the load bearing capabilities, wear is also an important factor in the automobile and industrial-scale applications. CNT-based ceramic composites exhibit excellent wear-resistance behaviour by avoiding crack propagations and debriefs on the surfaces of the composites at various loads and temperatures [33]. CNT-based ceramic coatings have enhanced strength, wear resistance and higher fracture toughness [133]. Though, aluminium and its alloys are extremely popular in automobile applications for inner surfacing and ducting. Nowadays, aluminium-based metal matrix with ceramic reinforcements has also created new roles in automotive materials [130, 131]. Nayim et al. examined stir casted aluminium matrix composite reinforced with CNTs/TiC and reported a reduction in the density and increase in hardness due to the volatile nature of reinforcing material [134]. Though reinforcement of metals provided high strength and better properties, few articles reported low scale corrosive nature on the metal matrix composites which could be a concern for researchers. Therefore, there is a need for ceramics such as alumina, zirconia, SiC which are highly chemically inert and have reduced density

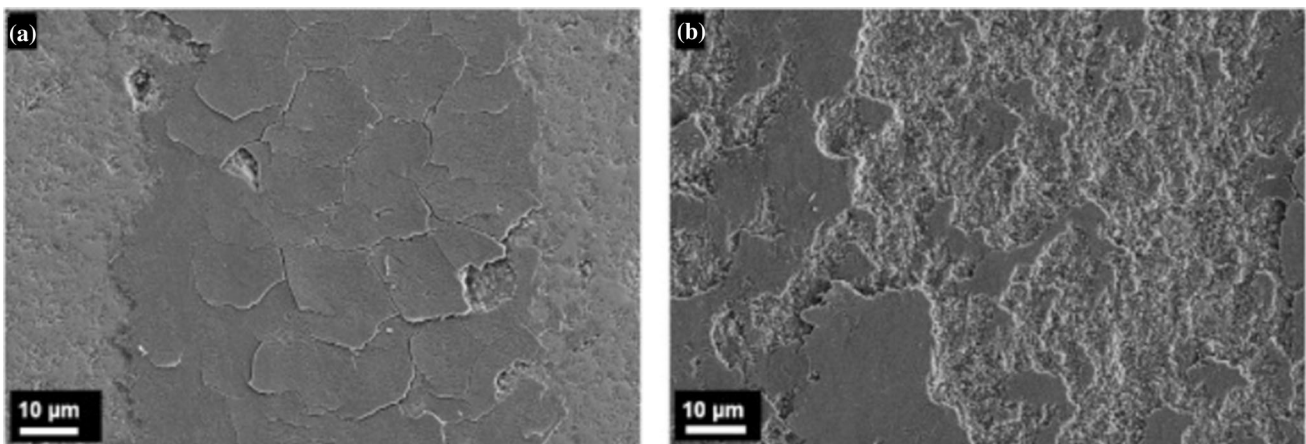


Figure 15 Wear topography of alumina with 10 vol.% CNTs adapted from Hazel et al. with permission [114].

than the metal matrix composites are preferred. Leonov et al. fabricated alumina reinforced by MWCNTs through SPS and determined increase in fracture toughness of $4.93 \text{ MPa}\cdot\text{m}^{0.5}$ and microhardness of 23.26 GPa [135]. Likewise, reinforcing SiC which is generally non-conductive with 6 vol.% MWCNTs enhanced electrical conductivity of the resulting composites enhanced its use in sensors, heat exchangers and automotive parts [136].

Aerospace applications

In recent decades, the aerospace industry has enjoyed rapid growth and in turn forces innovation in new materials. The possibility of combining desired material properties using nanocomposites has many potential applications in the aerospace industry. For example, ceramic materials have significant advantages in high-temperature applications. Various ceramics have been studied for its suitability in the thermal barrier coatings, structural areas and as reinforcements [32, 130, 131]. However, due to the lower fracture toughness and unable to satisfy required strength, ceramics require a potential reinforcement for its utilisation in aerospace industries. CNT reinforcements onto the ceramics could lead to improvement in mechanical and thermal properties of the composites along with satisfying high strength to weight ratio and fracture toughness which are crucial for the aerospace applications. Apart from structural strength, CNTs have potential to act as radiation shields for space vehicles [137]. Various research has been carried out for by researchers to determine the suitability of nanomaterials for radiation shielding including galactic cosmic radiation, solar particle events and neutron generated from interaction of solar and cosmic radiations [138]. Li et al. fabricated polydimethylsiloxane (PDMS)/SWCNTs through curing technique under 80 °C for proton radiation shielding and reported that under 105 MeV, the PDMS/SWCNT has shown enhanced proton stopping properties and was lighter in weight compared to PDMS and aluminium metal [139]. Likewise, CNT/polymer composites have also shown enhanced electromagnetic interference compared to polymer under same conditions [140]. The aerospace industry is classified into many categories such as commercial aircraft, unmanned aerial vehicle (UAV), rotorcraft, military aircraft and spacecraft (or) space shuttle as illustrated in Fig. 16. Despite this

often-disparate categories, general problems includes weight, lightning strikes, icing, electromagnetic interference shielding, stealth applications and hypersonic vehicle problems such as air resistance, high temperature capabilities and structural failure [141]. In space propulsion, aircraft are directly exposed to high-intensity ionising radiation along with exposure to debris formed due to thermal cycling and micrometeorites [142]. The aerospace vehicles are also subjected to various conditions involving moisture variations and extreme temperatures which require effective solutions including materials to overcome these problems [143]. CNT-based ceramic nanocomposites have excellent future in the aerospace applications which were studied by Ames Research Centre and Johnson Space Centre of NASA which have conducted investigations on thermal radiation and impact protective systems using CNT-reinforced materials [144].

Icing is a major concern in the field of aerospace as water droplets on the surfaces are supercooled and affect the outer surface components of the aircrafts including compressors which could damage the lift and angles of attacks in the fixed wings aircrafts. Icing also has a huge impact on the propulsion efficiency due to the increase in drag value [145]. Peck et al. investigated icing problems in rotor aircraft and stated ice adhesion results in vibrations of rotor blades that can lead to forced landings along with blurred visions to the pilots and unreliable instrumental readings [146]. To overcome icing issues, there are few techniques including anti-icing coatings and thermal insulators which could be employed on the surfaces. However, adding in a thermal insulator could lead to increase in weight which could reduce the efficiency of the aircraft. Chu et al. developed self-heating CNT-reinforced composites which provided excellent electrical heating at ambient temperature [147]. MWCNTs-based thin and transparent films were developed by Yoon et al. for de-icing applications [148]. These CNTs-based web laminated acted as electrothermal heating for the anti-icing where the structure of web is aligned perfectly with negligible weight gain. The web structure could produce uniform heating with less energy consumption [149]. These coatings provided protection against icing edges and enhanced barrier resistance on the surfaces without compromising the thrust to weight ratio. Like icing, nullifying lightning strikes are a big challenge to every aircraft with statistical data

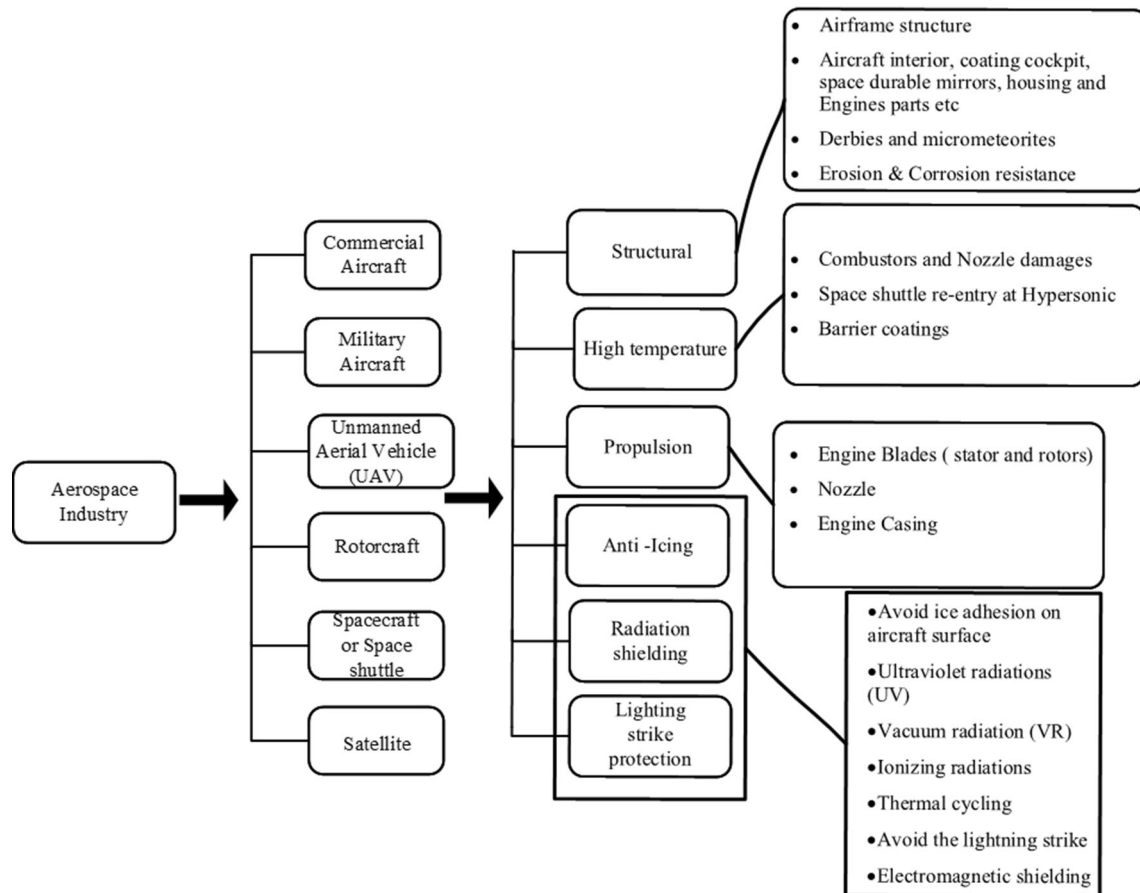


Figure 16 Schematics of classification of aerospace industry and its major concerns.

reporting most of aircrafts are subjected to lightning strike for 1000–10,000 h [150]. Lightning strikes on aircraft typically occur at an altitude of 5000–15,000 ft with a power of 200,000 A and heat generated close to 30,000 °C and impact force of 500 psi [144]. Lightning strikes can lead to damage, melting or burning of airframes. Conventional airframes are made of aluminium and its alloys which are highly conductive and reactive towards lightning strikes. Therefore, these materials tend to provide a threat towards the flying of the aircraft in the lightning climate. So, researchers have focused to form structural materials which can replace the aluminium-based alloys. Researchers have come across materials like fibre-reinforced polymers (FRP) which are unable to conduct high electrical current and electromagnetic force sufficiently during lightning which results to aircraft structural failures and electromagnetic waves interfere which may affect the communications and electronics devices in the aircraft. So, it is necessary to

protect against lightning strike to prevent both lightning current and EMI phenomena. Recent studies conducted on CNT-based composites show that they can offer protection from lightning strikes. It reported advanced materials like polymer-based composites and SWCNTs could be utilised to improve lightning protection and electromagnetic interference resistance (EMI). CNTs and its carbonaceous structures-based composites could support the aerospace industry with superior shielding effectiveness and high specific surface area due to its high aspect ratios. Also owing to the high vaporisation rate of CNTs-based composites compared to metals, it is said that the material could withstand higher EM shielding and lightings [151]. However, there are still more studies required to understand a way to collaborate the lightning strike protection along with weight of the materials.

Weight of aerospace structures is a challenge towards the researchers working on the aerospace

industries as it has impact on the efficiency and emissions of aircrafts. Generally, aircraft are designed in a way to reduce the total weight. However till date, there is no stand-out material which can withstand the extreme thermal strain and low weight requirements in aircraft engines [152]. Currently, Ni-based superalloys are utilised in turbine areas owing to its high temperature stability and thermo-mechanical properties at 800 °C with help of thermal barrier coatings [153]. So, there is a need of materials which can reduce and enhance the temperature capabilities of the turbines to increase the efficiency of the composites [154]. CNT-reinforced ceramics and CMCs are deemed to be a potential replacement in such areas and studies have been carried out to verify its suitability. Companies like Rolls Royce have initiated the use of the CMCs (oxide and non-oxide) in their stator parts. Though owing to its reaction with the water vapour, the service life of the CMCs is affected. As CNTs are inert to the water environment, the use of CNT-based composites could be utilised as a replacement. A simulation study by O' Donnell et al. reported that by replacing aluminium in different commercial aircraft including Boeing 747-400, Boeing 757-200, Airbus A320 and Embraer E145 with CNT-reinforced polymers could lead to 14% weight reduction, resulting in 13.2% range increase and 9.8% reduction in fuel consumption [155]. Further study by Gohardani et al. to replace the copper wiring by CNT-polymer-based composite wiring could lead to weight reduction by up to 69% [144]. These reports by researchers indicate that there is a constant need for developing the structural material in the area of the aircrafts and aerospace applications. The need for ultra-high-temperature ceramics (UHTCs) for hypersonic vehicles has been an important driving force in the field as hypersonic flight which involves high aerodynamic forces and extreme heat flux generated by temperatures of ~ 2400 °C. These UHTCs are designed to overcome thermo-mechanical problems occurring on the surfaces of the vehicle [143]. Currently, materials such as Hf, Zr and other rare-earth-based ceramics and composites have been studied as UHTCs owing to their higher temperature resistance and extreme heat flux [156, 157]. Although there have been studies describing the need for ceramic-based composites for high-temperature applications, there is still a fundamental requirement for enhancing the thermo-mechanical capabilities of ceramics in UHTCs. Though most ceramics have

good thermal resistance, ceramic/CNT composites (thermal conductivity ~ 3000 W/mK) provide enhanced resistance towards thermal damage with improvement in the thermal behaviours of the composites [158]. Numerous research articles have been published on reinforcing different UHTCs with CNTs to enhance the thermal behaviour and fracture toughness of the materials [158, 159]. Upada et al. utilised CNTs as reinforcement and a coating for aerospace engine components which enhanced their corrosive resistance and service life [160].

Like the structural needs, stealth is one of the major requirements in the aerospace industry especially for military applications. Emerging additive manufacturing of aerospace components and high specific strength of the CNTs make them one of the key elements for improving the mechanical properties. Owing to the absorption structure of CNTs, they are highly capable of absorption EM waves and X band frequencies and could enhance the stealth characteristics of the aircraft. Kim et al. studied the absorption rate of CNTs-based sandwich composite and aluminium and reported that CNT-based sandwich absorbed 90% of the EM waves which was 3 times higher than aluminium [161]. Along with these issues in the aerospace, there is also a constant need to enhance the efficiency of aircraft turbine engines. This could be achieved by enhancing the gas inlet temperature. However, increasing the gas turbine inlet temperature could lead to melting of Ni-superalloys which is already supported by thermal barrier coatings. Therefore, there is a need for high-temperature material which can reduce the weight and serve as replacement in turbine blades. Ceramics matrix composites (CMCs) are currently being research for its usage in turbine blades [162]. However, due to their reaction with water vapour, there is a need of new coating techniques which could enhance protection under various environments [163]. Many researchers approach this with a multilayer coating consisting of a bond layer, intermediate layer and top layer [164]. Studies have been also conducted to understand the suitability of ceramic/CNT composites as bond layer materials and thermal barrier coatings [133]. Goyal et al. studied the behaviour of CNT-reinforced Cr_2O_3 composites coatings for a thermal power plant barrier at a temperature of 900 °C. The CNT composition ranged from 1 to 8% and it was determined that with increase in CNT content, the corrosion resistance enhanced with

reduced weight gain [165]. Furthermore, the substrates were also modified to enhance the fracture toughness and hardness of the composites. Choudhary et al. found that a modified Ni-based alloy with 1 wt.% CNT reinforcement through a selective laser melting technique enhanced the elastic modulus up to 2.7 times compared to a normal Ni-based alloy. The plasma coating of lanthanum zirconate on Ni/CNT composites showed suppression thermally grown oxide growth at 1800 °C which was stated to be 600–800 °C higher than current aero-turbine engines [166]. Though there has been various research to determine the suitable materials for the environmental and thermal barrier coatings including rare earth silicates and zirconates for top layer coatings. There are only limited studies to determine the adhesion behaviour of the bond layer with the substrates including Ni-based superalloys and ceramic matrix composites. Further, there is also a lot of research required to establish a definite correlation between the quantity of MWCNTs reinforcements essential for improving the mechanical and thermal properties of the ceramic nanocomposites with the physical parameters including fabrication techniques factors such as pressure and temperatures for preparing high-quality aerospace applications and other engineering sectors.

Summary and future work

The present review on the MWCNT-reinforced ceramic composites describes various processing and densification techniques developed to enhance the properties of the CNT-reinforced ceramic composites. These newly developed techniques have provided better and more consistent distribution of MWCNTs within the ceramic matrix leading to improved mechanical and thermal properties. However, there is still a need to improve the fabrication and processing techniques to produce highly distributed MWCNTs onto the ceramics composites which could enhance the mechanical properties and fracture toughness.

By reviewing the fabrication and processing techniques associated with MWCNTs/ceramics nanocomposite fabrications, it can be stated that the: (1) dispersion of MWCNTs onto the matrix, (2)

Interfacing bonding between MWCNTs and ceramics and (3) Novel techniques to prevent damage of CNTs are critical issues which are to be addressed to fabricate highly dense and distributed nanocomposites. Furthermore, the investigation of the thermo-mechanical properties of MWCNT/ceramic composites indicated the necessity of novel fabrication method which utilises sufficient MWCNTs content for various applications with toughness enhancing mechanisms like fibre pull-out, crack and fibre bridging and crack deflections. Though it could be stated the future developed novel method could enhance the toughening mechanisms, there is still a need to establish relationship between various structural parameters with toughening mechanisms to attain high order relationship between the CNTs and composites. Also, there is a need to determine the amount of MWCNTs required to enhance and further an optimal technique is necessary to understand the design of MWCNTs-reinforced ceramic composites. The additive manufacturing sector could be game changer in the field of fabricating MWCNTs-based ceramic composites. However, with current achievements and growth in the additive manufacturing sector it would be question of time and optimisation for developing such techniques.

On the other hand, the immediate need of materials in the field of aerospace where CNT-based composites could be utilised has been discussed in detail along with various applications of CNT-reinforced ceramics. Though CNT-reinforced ceramics have various potential applications in aerospace industries. There is still a gap to understand the requirements of aerospace industries where the composites based on CNTs could fill in. Further, the gap in understanding the need of aerospace materials which could enhance the efficiency along with structural improvements is still occurring research and understanding the requirements is still closing up, it can be said that the CNT–ceramic composites could act as potential replacements in the industry with further research.

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