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Review Article

Carbon nanotube characteristics and enhancement effects on the mechanical features of polymer-based materials and structures – A review

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ABSTRACT

Carbon Nano Tubes (CNTs) are cylindrical structured rolled up sheets of graphene. The use of CNTs is observed as increasing immensely nowadays due to its tremendous characteristical properties. The low thermal expansion coefficient (CTE) of carbon nanotubes (CNTs), their high mechanical strength, high specific surface area (SSA), and high thermal conductivity strongly idealize them as a nanostructure for enhancing the physical properties of the fibre-reinforced polymer (FRP) materials for advanced applications such as microelectronic, chemical sensors, electromagnetic interference shielding (EMI), microwave absorption, hydrogen storage and aerospace. This review is comprised of the recent advancement in the fabrication of FRP by incorporated CNTs. Moreover, different functionalization techniques are analyzed to ensure uniform dispersion of CNTs and various fabrication routes were opted to enhance the overall performance of the CNTs incorporated FRPs composites. Common characterization techniques such as scanning electron spectroscopy (SEM), Differential scanning calorimetry (DSC), Thermal gravimetric analysis (TGA) and mechanical testing are discussed to study the influence of CNTs on physical and mechanical properties of CNTs incorporated FRP composite. Furthermore, comprehensive and descriptive overview of complex characterization techniques including Brillouin scattering, Raman spectroscopy, transmission electron microscopy (TEM), Dynamic light scattering (DLS), X-ray photoelectron spectroscopy (XPS) and Fourier transform infrared spectroscopy (FTIR), is also covered in the scope of this paper which are used for studying influence of CNTs on the properties of FRP composites. This review will facilitate upcoming researchers to choose the most appropriate functionalization method of CNTs, optimized fabrication route for composite synthesis, essential characterization techniques for evaluating the performance as per required applications.

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1. Introduction

Superior mechanical properties of a material can greatly enhance its industrial performance. The manufacturing of materials with improved properties depends on designing composites that harness additive or synergistic properties of component materials [1]. In this regard, nanotubes are a potential candidate to be employed in composites, as they possess structural perfection, high electrical conductivity, nanosize, and chemical stability which can be integrated into flat panel displays appropriate for electron field emitters [2,3]. Owing to these exceptional properties, carbon nanotubes can be employed in various applications, such as in oxygen reduction reaction that is electro-catalyzed through multiwall nanotubes. The Li-intercalated single-walled nanotubes (SWNT) used as battery electrodes are useful to observe large irreversible capacities and voltage hysteresis [3]. The unexpected reversible and high hydrogen adsorption has encouraged researchers to use nanotubes as high-capacity hydrogen storage media in SWNT materials. Previous studies have narrated that this unusual behavior has allowed the use of carbon nanotubes as essential products of electronic devices such as single-electron transistors, rectifying diodes, and field-effect transistors for logic circuits [2,4]. When nanotubes are employed in composites, they can improve the required features for a particular application.

Composites may consist of two, three, or more elementary materials arranged into a matrix, which determines the general shape of the composite and also provides reinforcements for the components to serve technical functions [5]. However, often the matrix of a composite is the first to fail because of relatively lower material strength. For example, while carbon fiber-reinforced composites are known for their excellent mechanical properties, the polymer, i.e., epoxy in a carbon fiber composite, is often the weakest link (CF/ECs) [6]. Several studies have reported nanoparticle integration into epoxy and its fiber-reinforced composites [7,8]. However, the modification of the matrix is idealized through the low thermal expansion coefficient of carbon nanotubes (CNTs), high specific surface area (SSA), thermal conductivity as well as high mechanical strength because of its high aspect ratio [6]. Strengthening polymer matrix by dispersing tiny particles, i.e., nanoparticles, is becoming increasingly popular among material engineers. Therefore, strengthening the matrix could improve the general performance of CF/ECs under various loading conditions.

The performance of the polymer matrix can be observed with the selection of appropriate material design that leads to mutual supplement and inter-relationship, hence producing a new superior performance. This property is essential in differentiating general materials from mixed materials. This improvement in the performance of the polymer matrix using composite materials emphasizes the importance of designable composite materials.

The selection of material is also an essential component of the composite design, as most industries prefer materials that are easily available, cheap, and convenient to use [9]. Moreover, in recent times, the material selection also relies on the material being biodegradable as well, along with all other properties. The elemental material carbon delivers all of these required properties therefore, many companies prefer carbon-based composite material designs [10]. In this regard, a rolled graphene sheet is considered a carbonaceous cylindrical nanomaterial in CNTs. Based on the number of graphene sheets and tube rolling, single-walled carbon nanotubes (SWCNTs) and multi-walled carbon nanotubes (MWCNTs) are the two most important forms of CNTs. The SWCNTs consist of one graphene sheet, whereas, MWCNTs are formed when multiple graphene sheets are rolled into making one tube having multiple nested cylinders with varying diameters [7]. Due to the differences in their structure, MWCNTs are more suited to reinforce polymer composites, including carbon-reinforced epoxy resins. However, more recently, a different form of MWCNTs, i.e., helical MWCNTs, is increasingly being used and preferred as a CNT for reinforcement [11].

Among various CNTs types, MWCNTs are the most ideal CNTs candidate to be incorporated into the polymer matrix [12,13]. The higher the percentage of CNT in a composite, the greater the strength improvement. However, there is an effective content threshold beyond which the further increase does not improve mechanical properties, primarily owing to the aggregation of CNTs. Considering the above-mentioned facts, the present study aims to focus on the mechanical characteristics of fiber/epoxy composites enhanced by carbon nanotubes. It examines the integration of CNTs and how this integration might enhance the mechanical efficiency of CF/Ecs. Moreover, the study compares various CNTs dispersion techniques to achieve maximum fracture toughness in a CNT-integrated polymer composite. It is observed that CNT-based composites, including carbon fiber/epoxy, exhibit an extremely high flexural and tensile strength as well as stiffness [12,14], Compression After Impact (CAI) [15], effective compression modulus, and the compression strength, and Young's modulus tensile and strength [16,17]. The improved properties can be credited to the improved dispersibility (bridging effect) as well as the extraordinary load-carrying capability of CNTs, which is also explained in this review. The present study also explains the effects of employing carbon nanotubes based on the existing literature and uses different papers combined.

The utilization of Carbon Nano Tubes for fabrication of Fiber Reinforced Polymer Composites have been observed increasing day by day. As in Graph 1, the increasing trend of CNTs utilization may be seen. Before 2010, the research studies related to CNTs based composites were not be

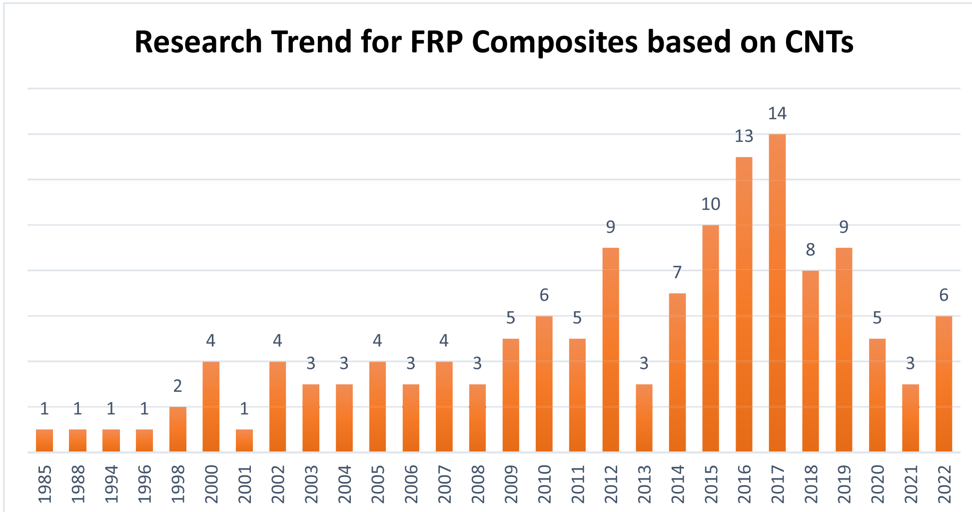

Fig. 1 – Research Trend for fiber-reinforced polymer (FRP) Composites based on CNTs 1985–2022.

considered as hot research topic. The data labels on peaks are number of publications addressed in this review. However, a great up fall in the utilization of CNTs for research and industrial purposes may be seen after 2011, according to the trends in Fig. 1.

CNTs has number of industrial applications relevant to the fields i.e. electronics, plastics, composites energy storage & harvesting etc. The total market share of CNTs is about $ 685.3 Million, which is classified in Fig. 2. The utilization of CNTs for industrial applications is also presented in Fig. 2. The optimistically excellent properties of CNTs make them suitable to be incorporated in Fiber Reinforced Polymer Composites specialized for industrial applications. This review is addressing all the aspects related to the characteristics of CNTs required for their incorporation in FRP composites. How these factors may be considered and tackled during fabrication of composites is also covered in this review which may help tremendously exploring new and innovative industrial application characteristics of CNTs/FRP composite.

This review also addresses the need of lighter weight CNTs based FRP Composites, delineating their mechanical behavior and their stability in response of material testing and characterization. The testing and characterization of a specific material is a core standard that can lift your material at upper level of significance gaining industrial/market stability and acceptability. The addition of CNTs is also considered as challenge as it surely effects the micro or nano structure of the base polymer. This behavioral change is not general and at industrial scale, the requirement of scale up production makes more difficult to avoid the change in the structural morphology and this review is addressing the structural changes, their causes, relevant properties and their possible methods of solution. Conclusively, this review study is covering almost all the required set of characteristical aspects or parameters, which may be a helpful database according to research and industrial point of view as well. This article highlights the recent advancements in the incorporation of Carbon Nano Tubes (CNTs) in FRP composites. Although there

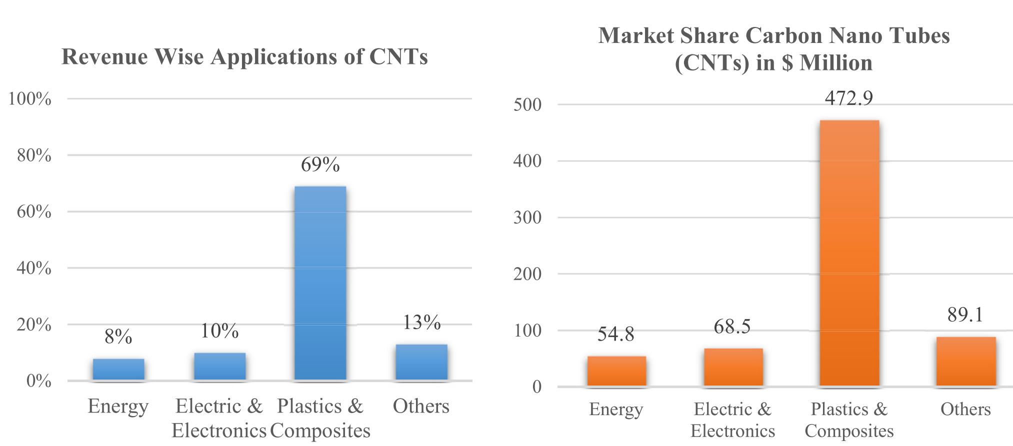

Fig. 2 – Market Share & Applications based Classification of CNTs.

are several works that investigate the CNT-addition effect on polymeric-based materials such as; electrical and magneto-resistance features [18], thermal conductivity properties [18,19], efficient microwave absorption in composite sheet, etc. [20,21], the authors here discuss the use of various functionalization techniques and fabrication routes to improve the mechanical properties and physical features of fibrous composite materials. The study delves into the characterization techniques used to investigate the influence of CNTs on the properties of FRP composites, including simple methods like Scanning Electron Microscopy (SEM) and Differential Scanning Calorimetry (DSC) and more sophisticated techniques like Transmission Electron Microscopy (TEM), Raman spectroscopy, Brillouin scattering, Dynamic Light Scattering (DLS), X-ray Photoelectron Spectroscopy (XPS), Fourier Transform Infrared Spectroscopy (FTIR), and Thermal Gravimetric Analysis (TGA). Moreover, the authors highlight the potential applications of CNT-based FRP composites in various fields such as microelectronics, chemical sensors, electromagnetic interference shielding (EMI), microwave absorption, hydrogen storage, and aerospace. This review aims to provide guidance to researchers in selecting the most suitable functionalization technique, fabrication route, and characterization method for their specific application. The article contributes to the advancement of knowledge in the field of materials science and engineering, specifically in the development of advanced materials for high-performance applications.

2. Research design

A descriptive and investigative research design has been used in this study. A combination of intensive and systematic literature review (SLR) has been employed as the research instrument. The rationale for using a systematic review was to identify, assess, and synthesize all the empirical evidence that fulfills a pre-quantified eligibility criterion to respond to a research question [8]. Furthermore, this study has used additional supporting literature gathered through an intensive literature review to reinforce findings through SLR.

For SLR, studies on CNTs and their combinations with composites, different dispersion techniques, CNT content, and an increase in mechanical performance were chosen. A systematic review of research articles and case studies obtained from different databases, such as Google Scholar and Sciencedirect was conducted. Table 1 displays the list of phrases that were used to search the publications along with hits per source, which is providing the insight of content requirement around the globe and demand of further research and summarization of previous research work carried out in that particular field. For data analyzes, NVivo software was employed for content analysis and/or thematic analysis [22]. This qualitative analysis software offered one-click access to word frequencies and keywords in the sample, while also permitting distinguishing patterns in the content across several texts and data sources.

Table 1 – Phrases employed to search the publications.

Search Phrases	Google Scholar	Science Direct	Total
Carbon “nanotubes” “epoxy” modulus “strength” “fracture”	26 700	4889	31 589
“Carbon nanotubes” “dispersion” “epoxy”	71 000	11 233	82 233
“Carbon nanotubes” epoxy “Raman spectroscopy”	19 000	3319	22 319
“Carbon nanotubes” “Brillouin spectroscopy”	15	4	19

2.1. Structure, characteristics, and properties of CNT

2.1.1. Structure of CNT

The effectiveness of CNTs is shown in different aspects, such as high surface area, mechanical characteristics, and nanotechnology. CNT's tubular structures, with a diameter ranging from one to a few nanometers, offer unique properties as compared to their non-nano homologs. Fig. 3 shows the pictograph and SEM image of single-walled carbon nanotubes (SWCNTs) and multi-walled carbon nanotubes (MWCNTs) taken from the literature [23]. CNTs are fundamentally an allotropic form of carbon, like diamond, graphite, or fullerenes. The internal geometry and diameter rely on the method of manufacturing and the variable approaches employed to reach the end product [24].

The synthesis, physicochemical properties, and types of CNTs are well-documented in almost all the literature that was obtained and reviewed based on the set criteria. In this regard, Iijima [25] presented the first observation of CNTs. The different types of CNTs can be characterized depending on the graphite layers, which can be considered as concentrically coiled graphite sheet layers where each carbon atom is joined with three other atoms by sp^2 hybridization [26], as shown in Fig. 3. Due to the curvature of the leaflets, the atoms of carbon also have a partial sp^3 character, which becomes more prominent with the reduction of the cylindrical radius. The space between leaflets is the same as in the graphite carbon, which is around ~0.34 nm in the normal direction. All CNTs are unique because of their size diameter (d), of the order of a nanometer, and length (L), of several microns. These parameters lead each CNT to possess a form factor, $a = L/d$, between 100 and 1000 [27]. Beyond these geometric characteristics, nanotubes have impressive electrical, thermal, mechanical, and magnetic properties [27,28]. Table 2 compares the different theoretical and experimental characteristics of CNTs and graphite. CNTs are lighter than graphite and possess approximately similar yield strength, which makes it the desired candidate for the applications where high strength to weight ratio is required. As far as thermal stability concerned, CNTs provide superior properties in comparison with graphite (see Table 3).

The bonding of carbon can be seen as a defining factor to develop materials with entirely different properties. There are several common methods used for CNT synthesis nowadays, as shown in Fig. 4 [45]. A layered construction with weak out-of-plane bonding is developed through the sp^2 hybridization of carbon. Similarly, an ordinary central hollow is surrounded by concentric cylinders with regular periodic interlayer spacing.

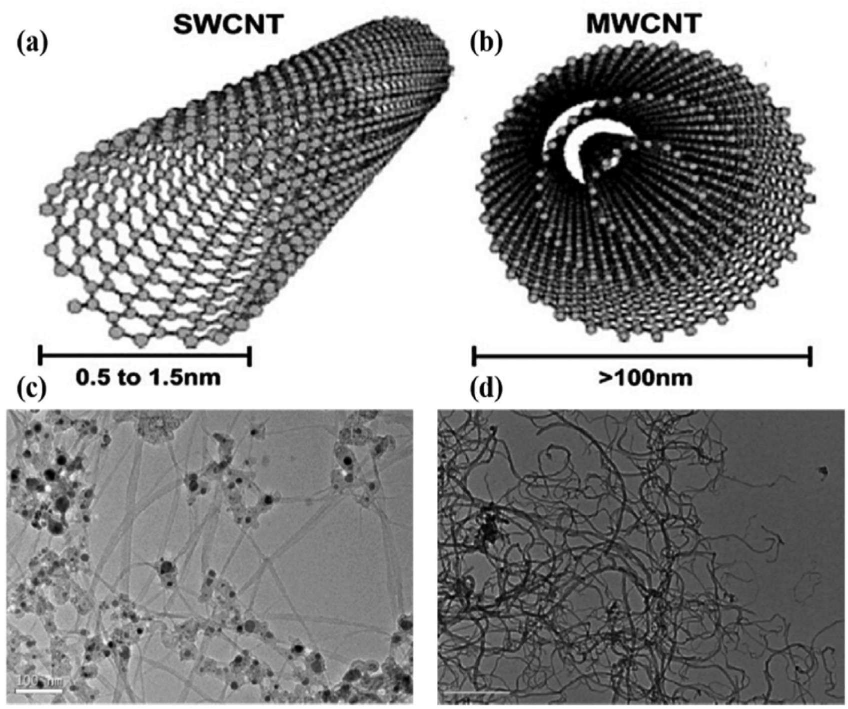

Fig. 3 – Pictorial Graph of (a) Single-Walled Carbon Nanotube and (b) Multi-Walled Carbon Nanotube and SEM image of (c) MWCNT and (d) SWCNT [23].

Another significant property of carbon nanotubes is their elasticity [46]. This means that the carbon nanotubes will bend, buckle, kink, and twist instead of breaking the nanotube under high force and press sitting. The nanotube will return to its original structure once the pressure element is removed, but nanotube elasticity does have its limit [47]. It might also be possible to temporarily deform to a nanotube shape under strong physical forces [48].

2.2. *CNT characterization technique*

Throughout extensive literature on carbon nanotubes spanning decades of extensive research, the complex methods of their characterization can be found which span from the most evident microscopy, for the size and shape study, to different types of spectroscopies. In Table 4, the general techniques of nanotube characterization are mentioned.

Table 2 – Comparison of previous researches & this study.

Past Research Factor	Comparison with this Research	Ref.
This previous research only incorporated the structural modifications occurred due to variation in parameters while fabrication.	This research report is covering all structural changes in response of change in parameters with complete characterization tools along with the optimization of polymer compounding.	[29,30]
The previous research are devoid of presenting complete algorithm for composite fabrication based on CNTs.	This research is clearing all methods of graphene/CNTs deposition/ synthesis for FRP Composite complete step by step algorithm.	[31–33]
Some researches has been found optimizing the process of composite fabrication but didn't addressed their compatibility with graphene/CNTs or any other organic material upon combination.	This report is addressing the optimization tools for the fabrication of composites well suited for combination with CNTs.	[34–36]
A Number of researches have covered generalized synthesis and its combination with composite, not addressing the effect of different types of CNTs.	The fabrication of multi walled CNTs with proper parameters optimization is covered under this study.	[37–39]
Generally, separate synthesis of CNTs followed by combination with composites is adopted by research studies.	This research study is also addressing the in-situ synthesis of CNTs based FRP Composite.	[40,41]
The proper distribution and dispersion is very challenging for researchers and is overlooked many times.	The distribution and dispersion properties of composites are delineated in this research incorporating limited but specialized CNTs and FRP composite compatibility factors.	[42,43]

Table 3 – Characteristics of the CNT compared to indicative carbon graphite.

Properties	CNT	Graphite	References
Density	0.8 g/cm^3 for SWCNT 1.8 g/cm^3 for MWCNT (theoretical)	2.26 g/cm^3	[16,22]
Young's module	~1 TPa for SWCNT ~0.3–1 TPa for MWCNT	1 TPa (in the plan)	[23,24]
Stress at break	50–500 GPa for SWCNT 10–60 GPa for MWCNT		[16,25]
Resistivity	5–50 μΩ cm	50 μΩ cm (in the plane)	[44]
Thermal conductivity	3000 W.m-1.K-1	3000 W.m-1.K-1 (in the plan) 6 m-1.K-1 (c-axis)	[22]
Susceptibility	22 × 106 EMU/g (perpendicular to the plane)		[22]
Magnetic	0.5 × 106 EMU/g (parallel to the plane)		[16]
Thermal expansion	Negligible (theoretical)		[16]
Thermal stability	>700 °C (in the air); 2800 °C (under vacuum)	450–650 °C (in the air)	[22,44]
Specific surface	10–20 m^2/g		[22,44]

The search volume for the keywords carbon nanotube mechanical and technique name consists of a range of specific methods and techniques which are shown in Fig. 5. The most used technique for CNT characterization is Scanning Electron Microscopy (SEM.

Aquel et al. [29] summarized the data type about the properties of different kinds of nanotubes, obtainable by TEM and SEM in their review article. It includes size distribution, shape, outer and inner radius, layer spacing in MWCNT, etc. Due to the small size of carbon nanotubes, the characterization of their mechanical properties directly is complicated. However, the same characterization of mechanical properties was performed by numerous researchers by employing experimental methods, SEM, TEM, AFM, and Micro-Raman Spectroscopy, and respective theoretical models. Soni et al. [29] in their exhaustive review summarized existing approaches to such measurements for single-wall and multi-wall CNTs. It was shown that the elastic modulus (E) of nanotubes depends on the synthesis technique, for example, the MWCNT obtained by Arc Discharge (AD) has an average E value of 2,0 Tpa and for the MWCNT obtained by Chemical Vapor Deposition technique (CVD), had an average E value of 0.4 TPa.

Raman spectroscopy is a powerful tool for the CNT geometry characterization, i.e., inside and outside diameter variation and interlayer distance in the case of MWCNT [61]. The article considers the use of Raman spectroscopy to define the geometry configuration of synthesized nanotubes [62]. The surface defects and the degree of crystallinity of CNT may be determined by the analysis of the D band shape on the Raman spectrum [63], which relieves the wide possibilities of the functionalization effectiveness detection. Goudaec [64] provided an explanation of the micro-Raman spectroscopy principle, and applications to the study of nano-objects' size, shape, and geometry configuration. It not only includes individual particles but, may be used for aggregates as well.

3. Nanocomposite fabrication based on CNTs dispersion techniques

3.1. *Common dispersion techniques*

CNTs exist in the form of agglomerates, therefore they are dispersed using different dispersion techniques. Fig. 6 shows the agglomerates of CNTs [5]. The uniform dispersion of CNTs in a polymer matrix plays a key role in determining composite mechanical strength. The selection of a certain technique is defined by the properties and state of the polymer matrix and the required performance of an article thereof.

The most important techniques of CNTs dispersion are listed in Fig. 7. Previous studies have emphasized the mixing techniques and the effectiveness of the dispersion of nanotubes in unsaturated polyester resin [15,41,65–67].

The main challenge in mixing techniques was to accomplish the exfoliation of large stacks of nanotubes into single layers, all the while incorporating the manufacturing process restrictions. Similarly, the main process parameters were also

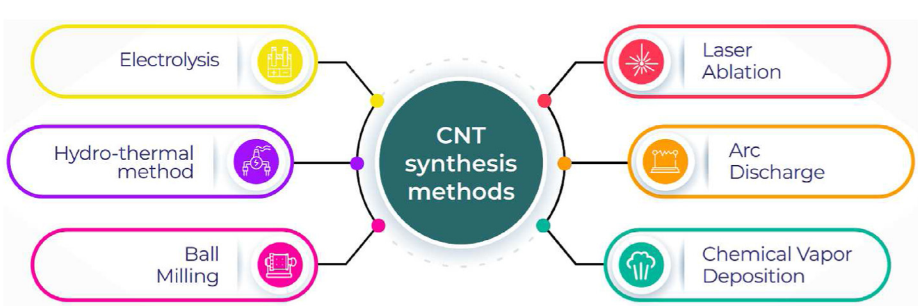

Fig. 4 – Various synthesis techniques of CNTs.

Table 4 – CNT characterization techniques.

Techniques	Properties	Refs
TEM	Size distribution, wall structure, defects, geometry	[31,49]
SEM	Size distribution, shape, topography	[50,51]
Atomic force microscopy (AFM)	Surface morphology, shape, structure	[52,53]
DLS	Particle size distribution	[42,54]
XPS	Surface functional groups	[37,55]
XRD	The phase structure, purity, and crystallinity degree	[56]
Energy Dispersive Spectroscopy (EDS)	Elemental composition	[32]
Raman spectroscopy	Diameter distribution, interlayer distance, chirality	[57]
FTIR	Surface functional groups	[58]
DSC	Thermal behavior	[59]
TGA	Thermal behavior	[60]

identified by considering appropriate nanotube injection and infusing natural fibers. On the contrary, this review deems to show flammability and improved mechanical properties of CNTs. In this regard, several dispersion methods were examined in this review, such as mechanical mixing, ultrasonication technique, solvent spraying technique, chemical functionalization [34], spark plasma sintering, and DNA-assisted dispersion [68,69]. Each of these ways of dispersion has its own distinct advantages: Mechanical mixing is a simple and inexpensive technique for combining materials, although consistent mixing may be difficult to obtain and particles can be damaged. Ultrasonication is a quick and effective process for dispersing particles in a liquid, and it may yield particles of very tiny diameters. Nevertheless, it may not be appropriate for all substances and might be costly. Solvent spraying is a simple and efficient approach for generating thin films, although it is not appropriate for all materials and might result in uneven coatings. Chemical functionalization may enhance the dispersibility and stability of particles in a liquid and is applicable to a broad variety of substances. Yet, it may be a complicated procedure that may affect the particle's characteristics. Spark plasma sintering is a process that uses a high-pressure electric current to generate dense and homogenous materials with great mechanical strength and distinctive microstructures more quickly and with less energy consumption if compared to conventional sintering processes [40,70,71].

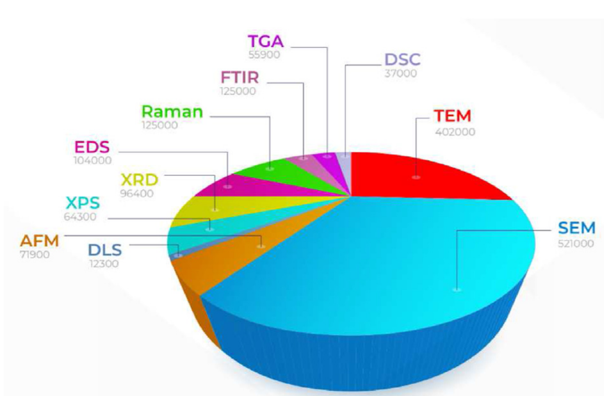

Fig. 5 – Search volume for keywords carbon nanotubes mechanical + "technique name'.

Within the matrix, homogenous/identical dispersion of CNTs is crucial to induce expected characteristics and obtain optimum performance of CNT-reinforced composites [72]. Many studies have efficiently improved the properties of the composite matrix by integrating nanotubes into epoxy and its fibre-reinforced composite. Several different strategies have been presented in the literature for the CNTs dispersion in epoxy, like mixing and sonication. However, most of these approaches are either restricted in capability or are not effective enough for uniform dispersion. The following section conducts the comparison of these techniques concerning the improvement in fracture toughness.

3.1.1. *Mechanical mixing in melt*

One of the most direct methods in the shear mixing arrangement without a solvent or a priori dispersant is mechanical mixing in the melt. This method yields composites with CNTs dispersed by a molten route using a mixer or extruder [69]. Fig. 8 taken from the literature shows the mechanical mixing of CNTs [69]. The principle is to overcome the attractive potential due to intense mixing [73]. The quality of the CNTs dispersion can be modulated by factors such as temperature, rotational speed, and residence time. The mechanical method is employed by manufacturing units for mass-scale production and is considered one of the most effective strategies for mechanical mixing [74]. Dispersion via mechanical mixing has been used for most thermoplastics such as polyethylene (HDPE and LDPE), poly methyl methacrylate (PMMA), polystyrene (PS), polypropylene (PP), polyamide 6 (PA6), polycarbonate (PC) or still thermoplastic elastomers; the list is not exhaustive [73–75].

Yokozeki et al. presented the experimental results on the mechanical properties of the epoxy matrix and carbon fibres via cup-stacked carbon nanotubes (CSCNTs) [76]. The study found no adverse effects on mechanical properties, but instead found improvement in stiffness and strength based on the experimental verification of CSCNTs dispersion. Significant enhancements in the mechanical properties of the modified resin and carbon fibre composites were observed via well-controlled processing. This study focused on the mechanical properties of compression, particularly after fracture toughness and impact strength, and impact resistance [77]. Giovannelli, Maio, and Scarpa employed mechanical mixing to obtain MWCNT epoxy resins [6]. The authors ran compressive and ASTM-derived tensile tests and found an

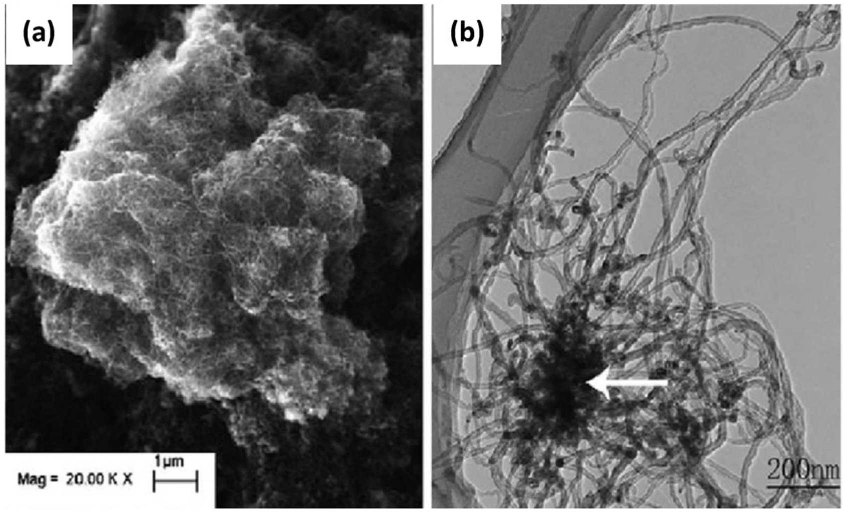

Fig. 6 – Images of CNTs agglomerates obtained by (a) SEM and (b) TEM [5].

increase in both compressive strength and Young's modulus. The study yields the same results with carbon fiber-reinforced epoxy of mode-I fracture toughness. The novel nanocomposite displayed superior mechanical performance in comparison to the average competitors. The method of dispersion highlighted a substantial potential for use in the industrial production of CNT-reinforced epoxy.

3.1.2. *Ultrasonication technique*

The goal of any dispersion technique is to generate enough local shear stress to overcome aggregation forces between aggregates. Ultrasonication is a technique that provides this shear stress in form of energy from sound waves to vibrate the particles, i.e., allows CNTs to overcome the binding energy between two or more nanotubes. From this perspective, ultrasonication uses a unique mechanism to deliver the required shear stress to disperse CNTs aggregates as compared to mechanical mixing [78,79].

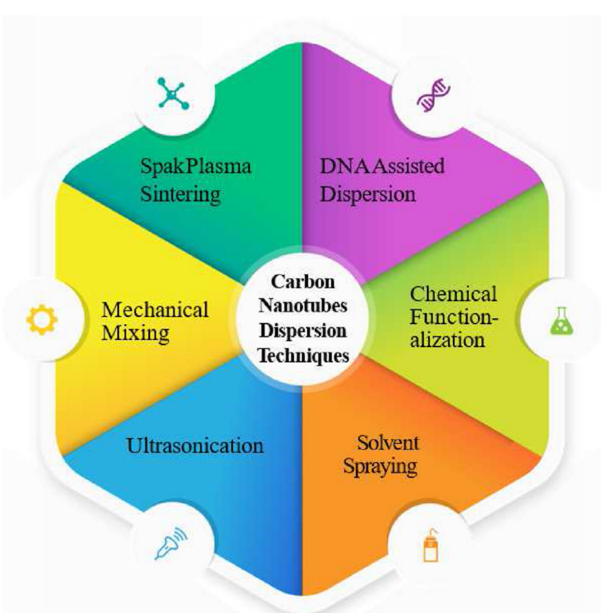

Fig. 7 – CNTs dispersion techniques in polymer matrix.

During sonication, a transducer irradiates the dispersion matrix with a sound field that creates cavities having localized pockets of high energy release. Sonication energy is typically delivered either using a bath sonicator or a tip sonicator. A bath sonicator creates a relatively weaker yet more uniform sound field compared to a tip sonicator. The sound field is closely attached to the standard, and the treatment is identically homogeneous for suspending a bath-type sonicator [80]. The sound tip sonicators can generate stronger fields but they induce a relatively turbulent mixing zone compared to bath sonicators. Required sonication energy and preferred sonicator type depend mostly on, the concentration of the nanotubes, the frequency of sonication, the temperature of the suspension, the viscosity of the suspension, the volume of the matrix, and the sonication time. Fig. 9 represents the dispersion of CNTs in ethanol with the help of ultrasonication [78].

Several studies have reported successful dispersion of CNTs in epoxy resin through ultrasonication. Fromyr et al. have investigated ultrasonic dispersion of commercially available MWCNTs where up to 1% (w/w) of nanotube was isolated through a horn sonicator and a bath sonicator [43]. The particle size distribution of the dispersed nanotube demonstrated a homogeneous and bimodal distribution of free and aggregate nanotubes, respectively. The authors reported an increase in the mass fraction of free, un-entangled nanotubes with an increase in acoustic intensity or diffusion time of the sonicators. Jamal-Omidi et al. have reported a multi-stage dispersion of CNTs, which includes ultrasonication as the final step of dispersion [81]. Unlike a stand-alone ultrasonication dispersion, the quality of dispersion obtained by the proposed method is not affected by the sonication frequency or temperature of the suspension.

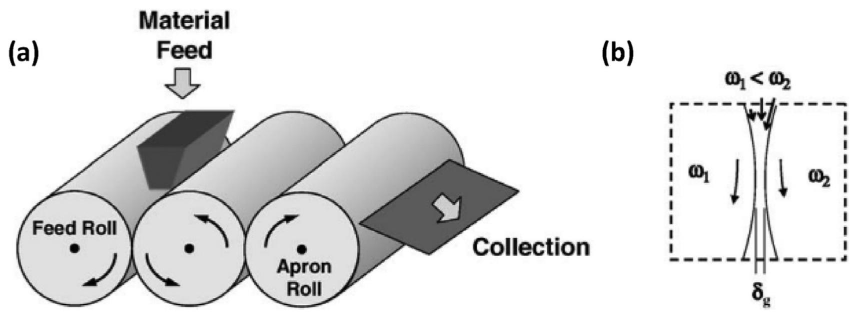

Fig. 8 – (a) Schematic diagram showing the general Configuration of a three-roll mill and (b) region of high shear mixing between the feed and center rolls [6].

3.1.3. *Solvent spraying technique*

The solvent spraying technique can be conceptualized in three stages: adding CNTs into the solution, combining the solution with the selected polymer, and finally, precipitation of the polymer or evaporation of the solvent to obtain the final composite. However, apart from some very acidic solutions like sulphuric acid or chlorosulfonic acid, CNTs have little to no solubility in solvents such as water or organic solvents. Thus, it is recommended to use an additive during the first dispersion step in the solution. Fig. 10 represents the fibers onto which CNTs were deposited using the spraying technique [82]. In Fig. 10, three different spraying sequences were opted to optimized the uniform dispersion of CNTs on fibers. In Fig. 10(a), extensive agglomeration was observed whereas in Fig. 10(c), slight agglomeration was noticed. Fig. 10(b), showed uniform dispersion of CTNs on fibers. Therefore, it was concluded that the spraying of CNTs-ethanol solution followed by E20-ethanol solution was selected as most appropriate sequence of spraying.

According to Bakshi, Tercero, and Agarwal, the solvent spraying method can help in obtaining stable dispersions owing to the adsorption of a homopolymer on the surface of CNTs [33]. This approach is based on a balance sheet of simple thermodynamics which states that the entropic cost incurred by the adsorption of the polymer is negligible compared to the free energy gain due to the screening of the solvophobic interaction between the solvent and the CNTs. Thus, stable solutions of nanotubes were obtained in water by sonication in the presence of polyvinyl pyrrolidone (PVP) and polystyrene sulfonate (PSS). After depositing these dispersions on a substrate, the nanotubes appear mostly individualized [33].

Rodríguez-González et al. have used a solvent spraying technique for the incorporation of MWCNTs on unidirectional carbon fiber/epoxy prepregs [7]. The authors have investigated the role of agglomerates in the reduction of oxidized MWCNTs on mode-I IFT (G_{IC}) of laminated composites utilizing a double cantilever beam (DCB) test. They reported that mode-I fracture toughness improved for all the laminated composites in contrast to the samples without MWCNTs. Furthermore, they found that a 52% substantial increase in the average G_{IC} initiation was achieved for laminated composites that were reinforced with oxidized and agglomerate-reduced MWCNTs prepared with only 0.05 wt% MWCNTs.

In addition, Guadagno, Kadlec, and Hron investigated the effects of flame retardant glycidyl POSS and MWNCNTs on mode-I IFT of carbon fiber-reinforced epoxy [83]. They found that the mechanical characteristics were reduced by 10% from the mutual effects of GPOSS and CNTs. Agglomerates or a change in fibre content volume caused a reduction that was influenced by the complex procedure of infusion due to high resin viscosity. Nevertheless, the authors resolved that the reduction of strength appears to be inconsequential compared to the benefits of the GPOSS in terms of added flame resistance and higher electrical conductivity of the resin.

3.1.4. *Chemical functionalization*

As aggregation is a surface phenomenon, chemical changes on the surface of the nanotubes may facilitate their dispersion into the solvents and/or polymers [34,84]. The common methods of surface functionalization include two groups: chemical functionalization and physical functionalization as stated in Fig. 11. Chemical surface treatment of CNTs includes oxidation, chromatography, acid treatment and functionalization (attachment of functional groups on the surface). In the case of grafting short molecules, a modification in surface energy makes it easier to disperse. Studies have reported that this method improves the interfacial interaction between the matrix and CNTs, and the respective dispersibility of CNTs into the matrix [85]. For example, amino-functionalized CNT shows improved dispersion in the epoxy, and MWCNTs display more improved dispersibility in polymer rather than SWCNT or DWCNT [70].

Fig. 12 represents the chemical functionalization of CNTs showing that there are two methods for functionalization, one is the covalent method while the other one is the non-covalent method [86]. Covalent methods include the sidewall attachments and end and defects while non-covalent methods include the polymer wrapping and surface attachments.

The impact of amino functionalization of MWCNT on mechanical and thermal characteristics of E-glass composite was examined by Rahman et al. and Garg, Sharma & Mehta [86,87]. Rahman and colleagues mentioned a 21% increase in strain tolerance (before failure), modulus, and a 37% increase in flexural strength for 0.3 wt% loading of MWCNT. The study also emphasized that to effectively employ CNT as structural

Fig. 9 – CNTs Dispersions in Ethanol after (a) 1 min, (b) 3 min, and (c) 5 min Sonication [78].

reinforcement and to have the adequate transfer of stress to the higher CNTs from the lower matrix, there is a need for robust interfacial adhesion between the polymer and CNTs. This adhesion can be accomplished by using amino-functionalized CNTs [86].

3.1.5. *Spark plasma sintering*

Spark plasma sintering (SPS) employs pressure-driven consolidation of powder by driving the direct electric current using a compressed sample in a matrix of graphite [40]. This technique is considered an energy-conservation approach because of its small processing time and limited process stages. Spark plasma sintering has been used to increase the fracture toughness of Al_2O_3/CNT nanocomposites. Several studies examined various amounts of CNTs (1–7 wt %) in Al_2O_3 and described a full value of fracture toughness of 4.7 MPa $m^{1/2}$ [5,83]. Fig. 13 shows the Al_2O_3/CNT nanocomposites synthesized using the spark plasma sintering technique [85].

Bakhsh et al. [70] have emphasized the development of CNT-reinforced alumina nano-composites by employing a novel SPS approach for the dispersion of CNTs and mixing them in alumina. The approach involved using differing concentrations of CNTs (1, 2, and 3 wt%) in the matrix. The authors have studied fracture toughness, hardness, and densification behavior of the nano-composites and conducted a comparison of mechanical properties. The study has shown an enhancement in mode-I and mode-II IFT of about 14% at 1 wt% CNT-alumina composite over non-reinforced composite. Such improvement in IFT was accredited to the enhanced dispersibility of CNTs in the matrix that eventually assisted in "grain growth suppression" to offer better grain in the CNT-reinforced composites. Similar findings were also reported by Azarniya et al. [88]. In comparison to conventional sintering processes, it is quicker and more energy-efficient and can be used to a broad variety of materials. Spark plasma sintering is better for conventional sintering procedures because it can generate dense and homogenous materials with high mechanical strength and distinctive microstructures more quickly and with less energy consumption. It is also applicable to a broad variety of materials and provides exceptional control over processing parameters, resulting in improved mechanical qualities and usefulness. Nevertheless, it takes specialized equipment and knowledge, and it may not be appropriate for all materials or applications [40,70,71].

3.1.6. *DNA-assisted dispersion*

Dispersion of CNTs through the DNA is one of the most enhanced techniques that is used to separate and disperse the SWCNTs and MWCNTs. In this technique, single-stranded DNA is used along with the sonication technique to mix the carbon nano-tubules evenly in the mixture. This technique is introduced when no other technique could provide efficient results. Fig. 14 represents the CNTs dispersed using DNA taken from the literature [89].

The ssDNA-assisted dispersion is advanced and much-accepted technique because there is no requirement for the solvents in this technique. This technique has been verified with the help of optical and fluorescence tests to observe the dispersed molecules [89,90]. Creating stable suspensions of nanoparticles or other tiny particles in liquid is facilitated by the DNA-assisted dispersion approach [89]. This technology exploits the capacity of DNA molecules to self-assemble and form stable complexes with nanoparticles or other tiny particles, which are subsequently disseminated in a liquid. The DNA molecules may be designed to precisely attach to the particle surface, enabling precise control over the dispersion process.

The DNA-assisted dispersion approach may be beneficial in the following instances.

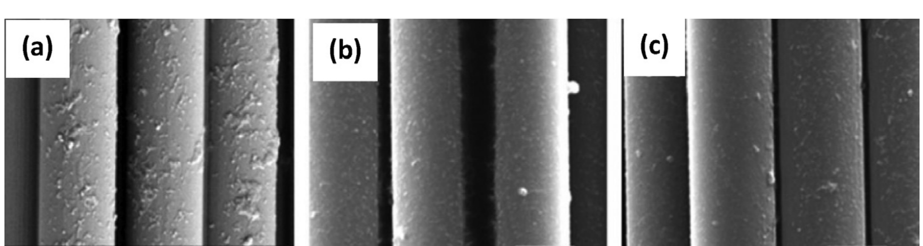

Fig. 10 – Sem image of fibers deposited CNTs with spraying technique [47].

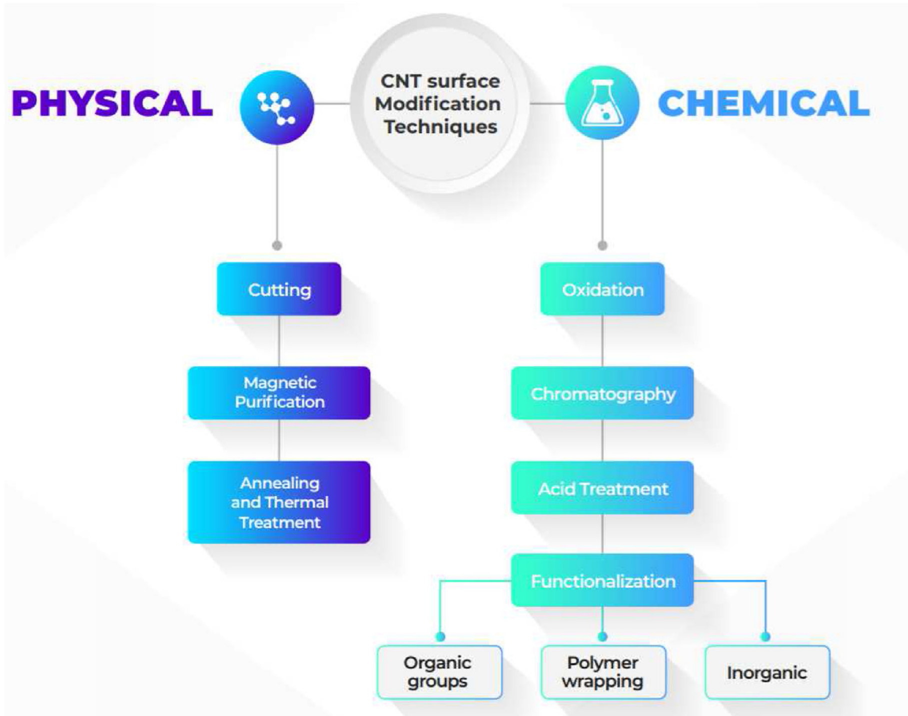

Fig. 11 – Physical & chemical methods utilized for the surface modification of CNTs.

- Developing nanoparticle suspensions for use in biomedical applications, such as medication administration and imaging.
- Using nanoparticles to create uniform coatings on surfaces for application in catalysis or electronics.
- Developing composite materials with increased nanoparticle dispersion and enhanced mechanical characteristics.
- By manipulating the arrangement of nanoparticles, new materials with unique optical, electrical, or magnetic characteristics may be created.
- The DNA-assisted dispersion technology has the potential to revolutionize the manufacture of nanoparticle suspensions, providing a formidable and flexible instrument for materials science and engineering.

3.2. *CNTs dispersion effectiveness control*

Coleman et al. define four main factors affecting the reinforcement effect of CNTs on polymer composites: large aspect ratio, good dispersion, alignment, and interfacial stress transfer [91]. Fig. 15 is showing a gap creation in nanotube embedded in a composite film and, as per literature, it happened due to sword and sheath mechanism [91]. The first factor of large aspect ratio is defined at the stage of CNTs synthesis, and the second and third factors of good dispersion

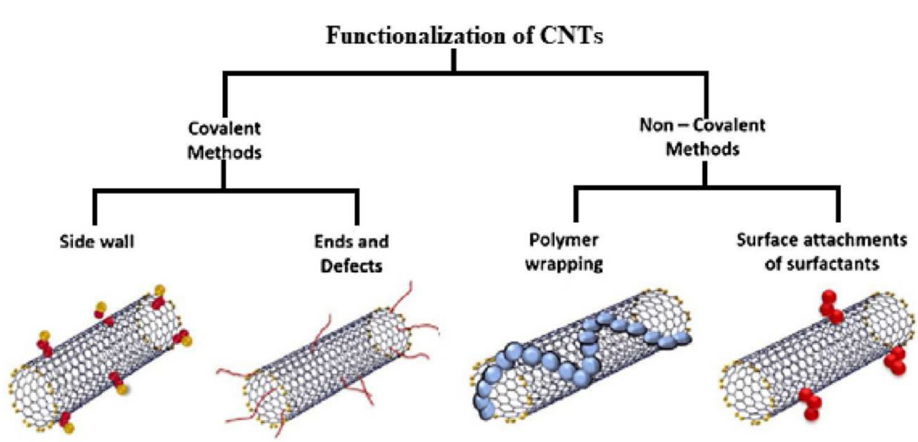

Fig. 12 – Chemical functionalization of CNTs [86].

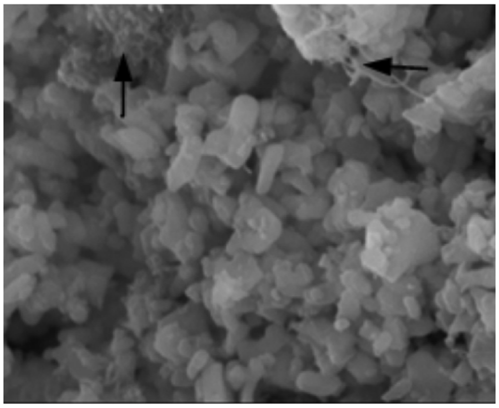

Fig. 13 – Sem images of CNT/Al_2O_3 composite powder after spark plasma sintering [85].

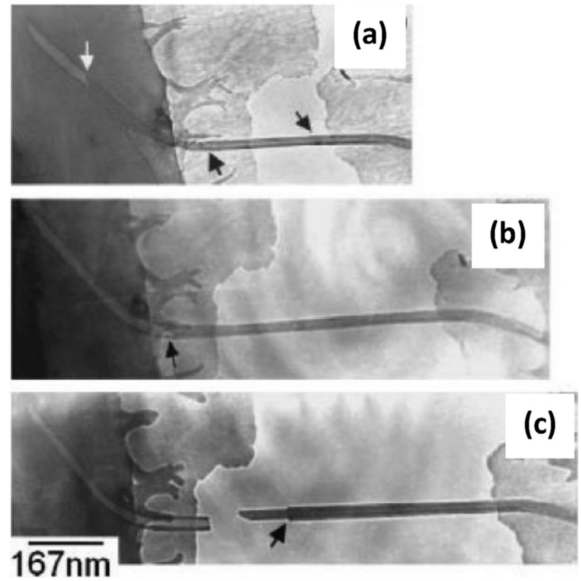

Fig. 15 – Tem images illustrating the failure of MWNT bridging a gap in a film of composite (c) nanotube failed by the mechanism of sword and sheath [91].

and alignment are defined at the time of the composite formation step. The last factor of interfacial stress transfer is defined by the CNT's surface topography, purification, and functionalization as presented in Chen's review [92]. The interface interactions may be characterized by various techniques, as discussed in the review article by Rahmat [93]. Despite the extensive use of microscopy to characterize the composite's uniformity, spectroscopic techniques, such as Raman are more practical for characterization in this case due to the less complicated preparation procedure [94].

In the article of Vigolo et al., it was demonstrated that the coupled use of Raman and Brillouin spectroscopy makes it possible to associate the functionalization degree of SWCNTs and the mechanical properties of bulk composites [95]. Andrews used micro-Raman spectroscopy to identify the strain of CNT in loaded polymer composites under controlled stress [96].

3.3. Comparison of dispersion techniques

There are several techniques mentioned in the literature that can be employed to achieve uniform dispersibility and enhance fracture toughness. In this regard, it is important to mention that almost all the techniques improved fracture toughness but with the constraint of agglomerates formation. It was also observed that the most widely used method is mechanical mixing; however, it is mostly used in manufacturing units for production on a huge scale. Another drawback of mechanical dispersion is that it inevitably leads to the breakage of CNTs during tense mixing. Mechanical mixing is an easily extrapolatable process for industrial applications in practicality and in the ability to produce large volumes in a short time [97,98]. Studies have reported mechanical mixing in masterbatch polymers containing high levels of CNTs, typically 10–50% by weight, to be re-dispersed in selected host matrices [35].

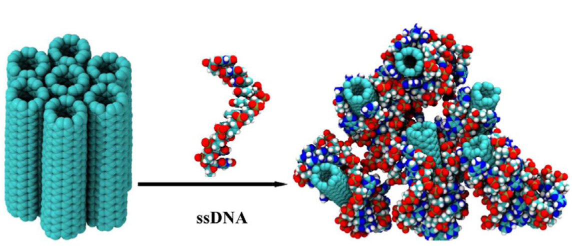

Fig. 14 – DNA-assisted dispersion of CNTs [89].

In comparison with mechanical mixing, the solvent spraying technique is more effective owing to its efficiency. Ultrasonication can also be a viable method, however, cavitation becomes a challenge when bubbles are nucleated (agglomerate formation) which generates considerable local deformation rates during their implosions. The generation of friction forces between the CNTs and moving liquid allows the breaking up of the dense clusters but can also stretch the nanotubes until they rupture [36,99]. Compared to mechanical mixing and solvent spraying technique, chemical functionalization is more efficient in achieving uniform dispersibility and the improved mode-I IFT as it involves chemically modifying the surface of the nanotubes to facilitate their dispersions in the solvents and/or polymers [71,100]. The functionalization method is limited by agglomerates or by a change in the volume of fiber content, owing to the complex procedure of infusion resulting from the high viscosity of the resin.

Lastly, Spark plasma sintering has been regarded as the most effective technique in achieving uniform dispersibility and improved mode-I IFT. This technique is also deliberated as an energy-saving technique owing to its small processing time and limited process stages. The technique also facilitates "grain growth suppression" to offer better grain in the CNT-reinforced composites [71,88]. Compared to all other techniques, the DNA-dispersion technique has its advantages but it suffers from scalability issues and an unusual method to disperse the CNTs. It does not require any organic or inorganic solvents. Only a small ssDNA molecule is required for dispersing these molecules. Striek et al. have reported that ssDNA sorting requires low energy which means that it can sort SWCNTs and MWCNTs by simple sonication method. The high cost of organic or inorganic solvents can also be saved by employing the DNA-assisted dispersion technique [10]. Comparison of all mentioned techniques is given in Table 5.

Table 5 – Comparison of CNTs dispersion techniques.

Dispersion Techniques	Potential outcomes
Mechanical Mixing	• Used a production scale • Breakage of CNTs occur [35]
Solvent spray technique	• Breakup of dense clusters of CNTs • Stretching of CNTs lead to its rupturing [36,99]
Chemical functionalization	• Better than mechanical mixing and solvent spray techniques • Limited by agglomerates or by changes volume fraction of fibers [71,100].
Spark Plasma Sintering	• Small processing time • Limited processing steps • Facilitates grain growth suppression [71,88,99]
DNA-Dispersion technique	• For small scale only • Low energy required • No organic or inorganic solvent required [10]

4. CNT composites mechanical properties

The use of CNT for enhancing the properties of polymer matrixes is extensively studied during the last decades. In this regard, epoxy resin composites are one of the most used, as is evident from the SLR study shown in Fig. 16.

Multiple studies reported that CNT-based composites, including carbon fibre/epoxy composites, exhibit high strength and stiffness characteristics [73]. Such characteristics depend on the SSA of CNTs with an upper range of 1300 m^2/g [101]. CNTs are reported to have a width-to-height ratio in the range of a few thousand. CNTs yield a better electrical and thermal conductivity that may be comparable to semiconductors or metals, as they are a graphite-based structure [102].

4.1. *CNT-integrated composites*

Various studies have indicated enhanced tensile attributes of CNT-integrated composites in comparison to composites without CNT reinforcement [38,103,104]. The CNT may be integrated into the CF composites as a separate ingredient to enhance tensile properties but as a functionalization element of carbon fibres. These tensile properties include, but are not limited to, tensile strain, tensile modulus, tensile strength, and lengthening at break or yield. Tensile tests determine the capability of composites to deform under tensile stresses and to endure tensile loads without failing [102]. Yokozeki et al. have reported that adding a certain proportion of CNTs (0, 5, and 10 wt%) in a matrix can increase tensile strength as well as stiffness of composite up to 25 and 42%, respectively [105]. The authors also observed moderate or no enhancement deprivation of compression after-impact (CAI) strength. Similarly, Kostopoulos et al. have indicated a two-third escalation in stiffness effectiveness of CF/ECs with 0.5 wt% of CNTs [106]. The authors further evaluated an improvement of CAI for both compression strength and modulus effectiveness. The stress–strain curve of CF/ECs with CNTs has been presented by Singh et al. for 1 and 4 wt% mode-I and mode-II interlaminar fracture toughness of composites (also shown in Fig. 17) [107]. The authors reported an increase of up to 70% in the elastic moduli for these weight fractions. Similarly, Hossain et al. have revealed that Young's modulus and tensile strength increased considerably with the increase in the concentration of CNTs [74]. The authors observed an enhancement of 10% in Young's modulus and 19% in tensile strength for 0.3 wt% XD-grade-CNT loading in comparison to the traditional one.

Singh and Gaurav [107] also examined the mechanical properties of epoxy/CNT composites. The researchers observed almost 21% and 34% improvement in flexural and tensile strengths in CNT-modified composites compared to control samples (without CNTs). The authors also observed that composites comprising CNTs demonstrated higher fatigue life whereas MWCNTs with a small aspect ratio are effective in hindering the growth of fatigue cracks.

4.2. *Characteristics of stress–strain relationship, toughness and low velocity impact*

In addition to the conventional stress–strain relationship and toughness characteristics [108], the stability of the composites under the low-velocity impact (LVI) is also essential. The LVI can be considered a quasi-static, out-of-plane impact event ranging from 1 to 10 m/s, and is often responsible for damage or failure in brittle composites, such as epoxy materials. Failure under low-velocity impact can be a result of delamination, matrix cracking, and fibre breakage [109,110]. In this regard, the integration of CNT in polymers has demonstrated a significant improvement in polymer strength [111]. For example, CF composites reinforced with MWCNT have demonstrated about a 50% increment in penetration energy under LVI conditions [112]. Such improvement can be further

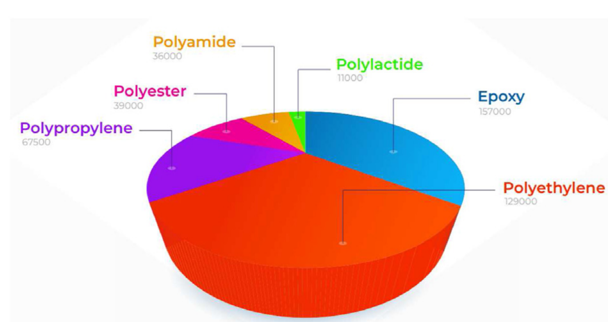

Fig. 16 – Quantity of articles with keywords "carbon nanotubes" + "polymer type".

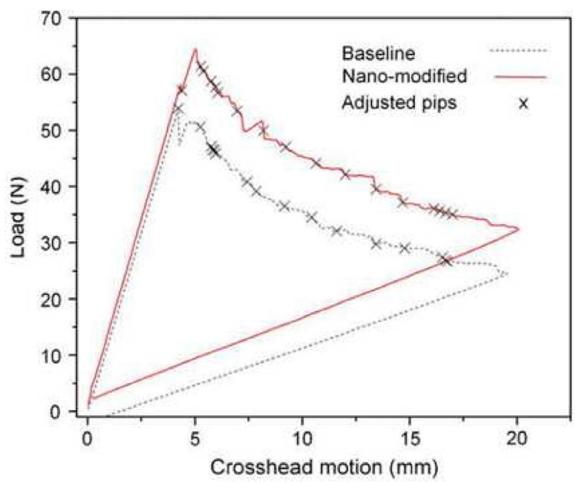

Fig. 17 – load–displacement curves of neat and SWCNT-Modified DCB samples [30].

enhanced by functionalizing MWCNT surfaces to provide chemical compatibility of CNTs with matrix materials. The improved properties can be classified into the improved load-carrying capability and better dispersibility of CNTs [68,69,113,114].

Identical dispersion of CNT establishes mechanical interlocking within the matrix and fibre by "bridging" [37,54,55]. This bridging, in sequence, during tensile loading improves the load-carrying capability of the composites. However, being the lowermost modulus constituent, the matrix begins cracking first during the loading. Afterward, the load is shifted by the CNT to the fibre having a high modulus via the "bridging effect" [68,69,113]. Therefore, the competency of nano-phased composites to absorb energy improves the toughness of the composite [24].

Increasing the ratio of CNT added to the matrix increases the mechanical strength. However, studies have also found that adding CNT beyond 0.4 wt% does not offer proportional enhancement in tensile properties, possibly due to CNTs

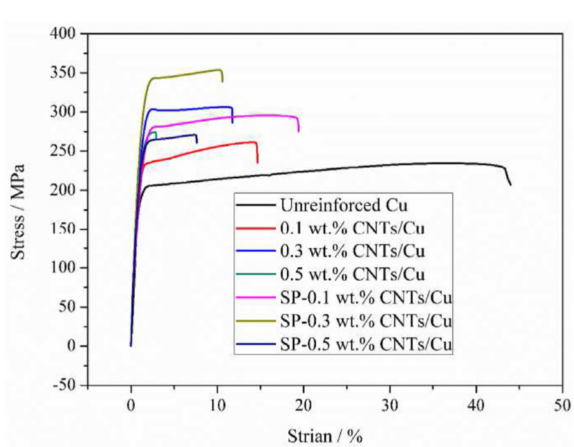

Fig. 18 – Stress–Strain Relationship of Unreinforced Cu, CNTs Reinforced Cu and SP & CNTs Reinforced Cu with Variation in CNTs wt%, Respectively [119].

aggregation. Such aggregation of CNTs can be attributed to van der Waals forces and steric interaction throughout the polymer matrix and nanotubes. Moreover, the aspect ratio of the CNTs also influences the extent to which the mechanical strength of a composite would be improved upon CNT integration. Studies have demonstrated that CNTs with a minor aspect ratio are more effective as epoxy reinforcement, likely because of relatively more homogenous dispersion (hence less aggregation) in the polymer matrix [107]. CNT aggregates eventually bring about weaker interfacial linking among the epoxy matrix and carbon fibre [77,101].

Generally, almost all the previous studies have demonstrated that the CNTs introduced in small proportions in matrix composites could significantly enhance the materials' mechanical properties [115]. In different industries, high-performance structural materials [116,117] are composed of these composites, including specific thermoplastic matrices [64,118]. However, the potential of the CNTs is much better realized when the CNTs are used as a significant component for the manufacturing of fibres. For example, Jiang [119] obtained CNT fibres by injection of a solution of CNT dispersed in sodium diphenyl sulfate in a bath of polyvinyl alcohol, from which the final composite is recovered after drying. The obtained fibres are oriented in the direction of flow and have a module ranging from 15 to 40 GPa, and approximate break stress of ~150 MPa, along with high flexibility. This same process has been adapted to obtain PVA fibres loaded with ~60% in the mass of CNT. These fibres are very deformable (around 100% elongation) and have a tensile strength of around 1.8 GPa. Spider silk allows these fibres to be used in several applications, such as textile and electronics. As shown in Fig. 18, the strength of these composites is enhanced by incorporating CNTs in the Cu matrix and on the other side % elongation decreased as wt% of CNTs increased. When CNTs content was below 0.5 wt%, the tensile strength of the composite was increased [119].

Fig. 19 (a) illustrates the images of a densely packed carbon nanotube film [120] that is aligned with MWCNT film. These films are synthesised in a two-step experiment and observed through an SEM image. Carbon nanotubes are useful in replacing the traditional carbon black without deterioration in functionality. Moreover, CNTs are capable of being stretched through plastic matrix of brush, thereby improving electrical and thermal conductivity. There are different combinations of CNT films with materials that are beneficial for improving the overall material characteristics. Amongst these combinations, Fig. 19 (b) and (c) [121] show SEM images of CNTs/shape memory composite (SMP) nanocomposites. Similarly, depending on synthesis conditions, CNTs forests show different structural morphology such as relatively aligned (Fig. 19(d)), or wavy and entangled morphology, as shown in Fig. 19(e) [122].

5. Experiments for damage analysis of CNT/polymer nanocomposites

5.1. *Fracture toughness test*

The mode-I and mode-II interlaminar fracture toughness of composites were quantified by using two main tests. One of the most commonly used methods for measuring the

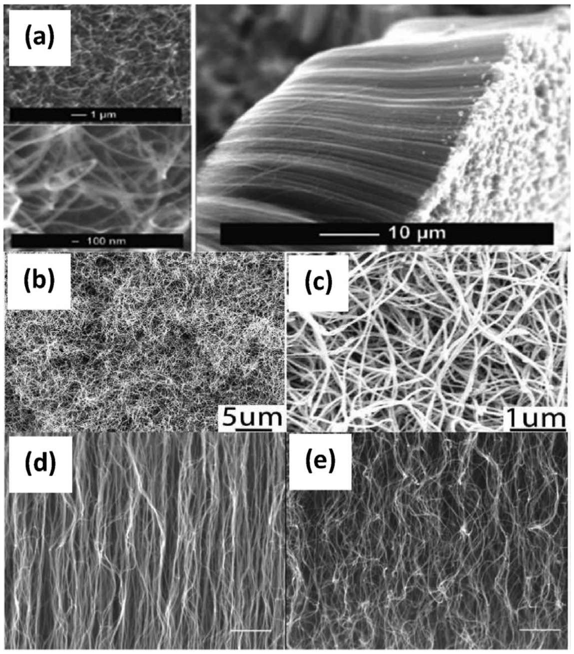

Fig. 19 – (a) SEM Image of a Densely Packed Carbon Nanotube Film (b–c) CNTs/Shape Memory Composite (SMP) Nanocomposites at 5 and 1 μm, respectively (d–e) CNT Forests Aligned and Wavy, respectively [120–122].

beginning and propagation values of mode-I fracture energy under static and cyclic loading conditions is the DCB test [123]. Besides the Double Cantilever Beam (DCB) test, the ENF test has emerged as the most used method for quantifying mode-II (IFT) metallic and laminated polymer composite adherents [124]. The End Notched Flexure (ENF) is essentially a DCB specimen loaded in a three-point or four-point flexure. Nevertheless, several difficulties arise in the DCB test of multidirectional laminates frequently, which avert precise measurements of the mode-I release rate of critical strain energy G_{Ic} [125]. Fig. 20 % the fractographic image of the Al_2O_3/CNTs composite with different percentage of CNTs [85].

Arai et al. have examined IFT for mode-I, and mode-II deformation for CF/ECs reinforced by CNT employing DCB and ENF tests, correspondingly [126]. The experimental outcome confirmed that the mode-I interlaminar fracture and mode-II fracture toughness were 50% greater and 2 to 3 times higher than that of base carbon fiber reinforced thermoplastic (CFRP) laminates, respectively. Romhany and Szebenyi have also employed DCB tests assisted by acoustic emission to examine the interlaminar properties of CF/ECs reinforced with 0.1, 0.3, 0.5, and 1 wt% of MWCNTs and reported a 33% increase in the IFT [39]. Similarly, Bilge et al. have examined the mechanical performance through the ENF test by incorporating MWCNTs with P(St-co-GMA) and reported that the nano-fibrous interlayers enhance the mechanical behavior of the laminates for epoxy that is chemically cross-linking [127].

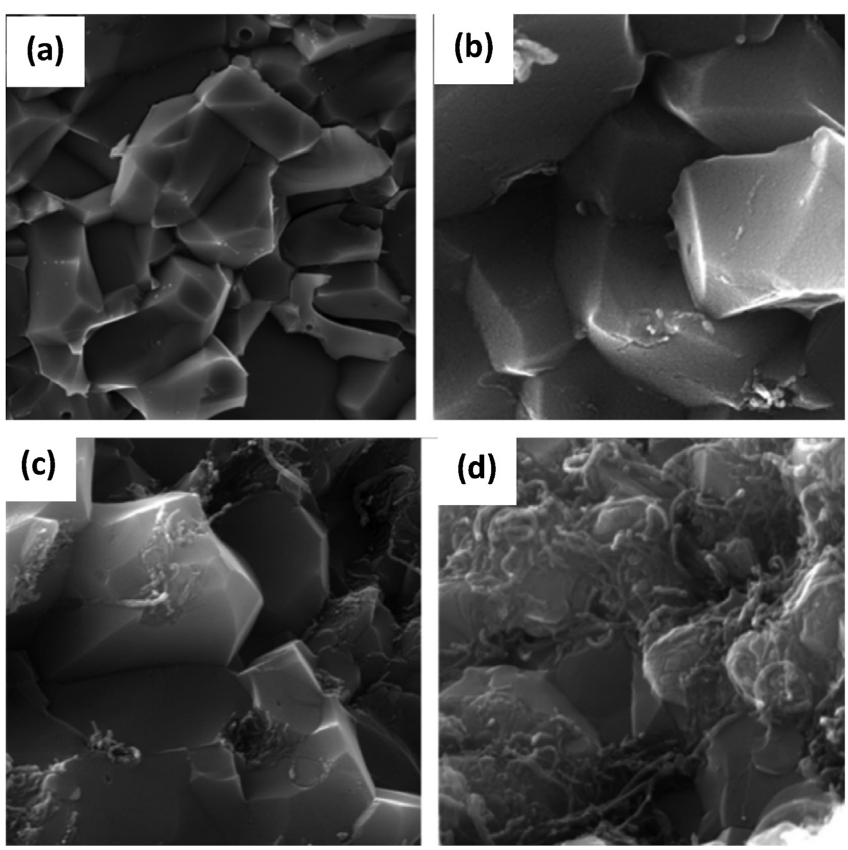

Fig. 20 – FESEM Fractographic Image of (a) Monolithic Al_2O_3, (b) 1 wt% CNT/Al_2O_3, (c) 2 wt% CNT/Al_2O_3, (d) 3 wt% CNT/Al_2O_3 Nanocomposites [85].

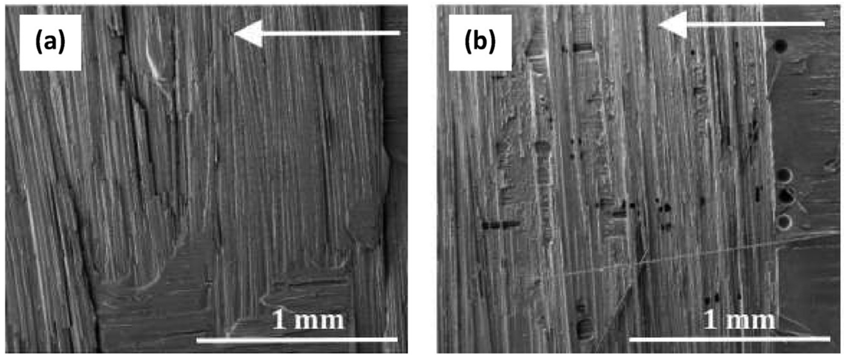

Fig. 21 – Sem micrographs of the fracture surfaces of the crack propagation regions after DCB test for (a) carbon-fiber/epoxy composite, (b) CNTs in cooperated carbon-fiber/epoxy reinforced composite [79].

Fig. 21 represents the fractographic image of CNTs deposited on fibers [79]. 3 wt% of CNTs were incorporated in carbon fiber-epoxy and mode I (double cantilever beam) and mode II (end notch flexure) fracture toughness were evaluated. Mode I fracture toughness was increased by 25% and mode II fracture toughness was improved by 50%.

Likewise, Yokozeki et al. have examined the enhancement of the IFT of CFRP laminates using CNTs subjected to DCB and ENF tests [76]. The results displayed that CNT-dispersed laminates had higher fracture toughness. Inam et al. have prepared DCB specimens for examining mode-I IFT of CF/ECs micro-nano composite laminates and reported the enhancement in IFT of laminates [65]. Silva et al. have performed the ENF test to check mode-II IFT of MWCNT-reinforced CF/ECs and presented an increase of 50% for 3 wt% of CNT and an increase of 30% for 1 wt% of CNT [66]. Similarly, Borowski et al. have conducted a DCB test for investigating the mode-I IFT of CFRP composites and reported 25%, 20%, and 17% upsurge in the maximum IFT by adding 0.50, 1, and 1.50 wt% of MWCNTs, correspondingly [67]. Rodríguez-González et al. have evaluated the mode-I and mode-II fracture toughness of the EC/CFs implemented with sprayed GO, MWCNTs, and GO/MWCNTs and displayed a constructive impact with 17% and 14% improvements on CF/ECs on the mode-I and mode-II fracture toughness after conducting ENF and DCB tests [41].

Rodríguez-González and Rubio-González have sprayed the surface of unidirectional CF/ECs with various concentrations of MWCNTs on composite laminates to assess their influence on IFT under mode-I and mode-II loading as well as effects in mechanisms of fracture [41]. The authors have examined mode-I and mode-II IFT by employing DCB and EFT tests on laminates and reported a 17% improvement in material

Table 6 – Improvement of mechanical properties by incorporating CNTs in FRP composites.

Year	Paper Title	Mechanical Properties, Reference
2014	Dispersion of Carbon Nanotubes in Alumina using a Novel Mixing Technique and Spark Plasma Sintering of the Nanocomposites with Improved Fracture Toughness	14% increase in fracture toughness [85]
2009	Fracture toughness improvement of CFRP laminates by dispersion of cup-stacked carbon nanotubes	Up to 300% increase in interlaminar fracture toughness [76]
2010	Multiscale Hybrid Micro-Nanocomposites Based on Carbon Nanotubes and Carbon Fibers	Increase in flexural modulus by 35%, 5% improvement in flexural strength, a 6% improvement in absorbed impact energy, and a 23% decrease in the mode I interlaminar toughness [65]
2014	Composites Part A: Applied Science and Manufacturing	For Mode I, IFT improvement by the incorporation of nanoparticle fillers, reached 17% for 1 wt% of MWCNT. In Mode II the increase was about 30% for 1 wt% of MWCNT [66]
2015	Interlaminar Fracture Toughness of CFRP Laminates Incorporating Multi-Walled Carbon Nanotubes	25%, 20%, and 17% increase in the maximum interlaminar fracture toughness of the CFRP composites with the addition of 0.5, 1.0, and 1.5 wt% MWCNTs [67]
2018	Influence of the Hybrid Combination of Multiwalled Carbon Nanotubes and Graphene Oxide on Interlaminar Mechanical Properties of Carbon Fiber/Epoxy Laminates	Enhancement in GIC and GIIC by 17% and 14% improvements on CF/E laminates with 0.25 wt.% MWCNTs/GO hybrid content compared to the neat CF/E [41]

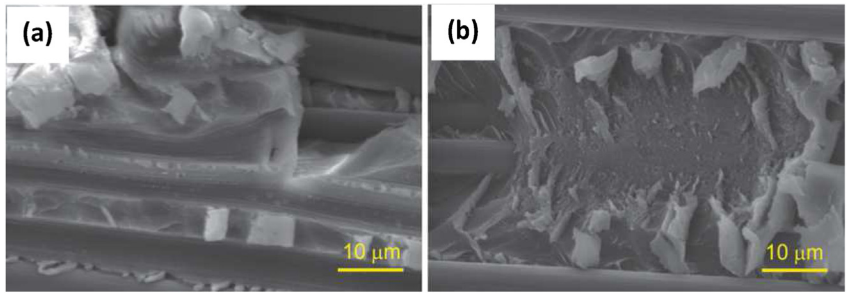

Fig. 22 – Sem images of mode II fracture surfaces of CF/E laminates without and with carbon nanostructures (a) CF/E and (b) MWCNTs-CF/E [65].

properties based on the IFT of CF/ECs laminates. Table 6 represents the effect on the mechanical properties of the composites [128] reported in various papers.

5.2. *Low-velocity impact and compression after impact tests*

Impact tests are employed to examine failure modes and dynamic deformation of material [50]. The risk of impact damage and the difficulties related to holes, restrict the criteria of design for CF/ECs laminates. As per Sjoblom and colleagues, the low-velocity impact is mainly deliberated because the damage might be left unnoticed [129]. In several circumstances, the impact level at which harm is noticeable is much higher than the level at which considerable harm to residual properties takes place [129]. Consequently, Low-velocity impact (LVI) testing is employed to comprehend the damage and deformation mechanisms included in the effect of targets, for the efficient design of CF/ECs laminates [75]. Subsequently, the damage resistance of composites is defined with the employment of the CAI test after an influential event [15].

Fig. 22 is showing SEM image of the mode II fracture surface of CF/E [65]. The LVI response of laminates began in the 1980s by Elber et al., and Abrate has conducted a comprehensive appraisal of all the studies accessible from 1989 to the date and perceived damage imposed by impact-contained delamination [130], matrix cracking [17], and fibre breakage [131]. Shrirao and Naik [132] have considered a phenomenon similar to LVI, which is once the period of impact is bigger than the period needed for a lowermost vibrational mode of the specimen [133,134].

Soliman, Sheyka, and Taha have examined matrix-dominated characteristics such as LVI resistance and interlaminar shear stress [112]. They also reported an increase in impact strength after the incorporation of CNT into the matrix. It was observed that the incorporation of CNT with cement-based material also provides flexural and tensile strength [135]. Baltopoulos et al. have used non-functionalized MWCNTs in carbon fibre-reinforced polymers and observed no improvement in overall energy absorbed upon impact compared to unmodified CFRP [106]. This confirms reports by others, that significant improvements in the LVI response of FRP can be achieved with the help of functionalized MWCNTs. These

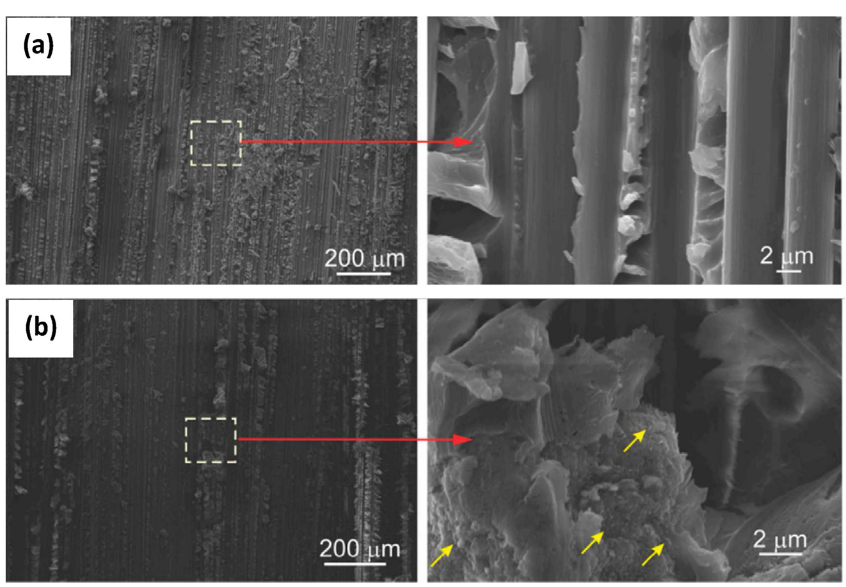

Fig. 23 – Fracture Surfaces of Mode I Composite Laminates. (a) CF/E, (b) 0.05 wt.% of MWCNTs in CF/E [136].

Table 7 – Mechanical properties of CNTs-based composite.

Year, Reference	Research Title	Mechanical Properties
2020 [135],	Effect of carbon nanotubes on compressive, flexural and tensile strengths of Portland cement-based materials: A systematic literature review	The incorporation of CNTs increased compressive strength by up to 30% and flexural and tensile strength by up to 50%
2011 [112],	Low-velocity impact of thin woven carbon fabric composites incorporating multi-walled carbon nanotubes	The addition of 1.5% MWCNTs resulted in 50% increase in energy absorption.
2020 [142],	Experimental assessment of adding carbon nanotubes on the impact properties of Kevlar-ultrahigh molecular weight polyethylene fibers hybrid composites	Adding 0.1 wt.% carbon nanotubes in this configuration was caused to increase the normalized absorbed energy more than 6.5 times.
2022 [143],	Comparison of impact resistance of carbon fibre composites with multiple ultra-thin CNT, aramid pulp, PBO, and graphene interlayers	Compression after impact tests were conducted on these impacted specimens, showing 8.1–37.8% improvements in residual compressive strength from different micro- and nano-filler reinforcements
2012 [29],	Carbon nanotubes, science, and technology part (I) structure, synthesis, and characterization.	The compressive-after-impact strength of CF/ECs laminates has displayed an upsurge of around 8% when 10 wt% CNTs are used to modify CF/Ecs laminates
2000 [141],	Low-Velocity Impact Testing.	CAI performance of CF/Ecs laminates reinforced with CNTs and found a 30% enhancement in CAI in comparison to non-reinforced CF/Ecs.

improvements can be mainly attributed to higher interfacial adhesion between the functionalized matrix and CNT, and subsequent interaction between fibres and the modified matrix [43]. Avila, Soares, and Neto have reported a 48% improvement in absorbed energy by CRFP during LVI testing with an infusion of 5 wt% nano-clay [81].

Fig. 23 shows the fracture surface of the CF/E composite with and without CNTs tested in mode II [136]. When loaded under compression, CF/ECs laminates with impact damage face substantial reductions in strength because of the local uncertainties arising from the widespread damage [137–139]. However, there is a lack of literature on the CAI of CF/ECs laminates [50,63,140]. The compressive-after-impact strength of CF/ECs laminates has displayed an upsurge of around 8% when 10 wt% CNTs are used to modify CF/Ecs laminates [29]. Xu et al. have efficiently achieved the dispersibility of MWCNTs and polyether ketone cardo (PEK-C)/dichloromethane and found that CAI was improved substantially for enclosed composite laminates in comparison to standard composite laminate [74].

Nikfar and Njuguna have examined the CAI performance of CF/Ecs laminates reinforced with CNTs and found a 30% enhancement in CAI in comparison to non-reinforced CF/Ecs [141]. The residual CAI strength of the composites enhanced considerably for the reinforced sample with the glass fibre laminates, displaying a maximum enhancement of 55% in comparison to non-reinforced samples. Erdogan and Bilisik have investigated the CAI properties of the multi-stitched composite and found that the CAI strength of the multi-stitched composite was high compared to the unstitched composites [76]. Ismail et al. have examined the LVI response and CAI of CF/E composites reinforced with montmorillonite (MMT) nano-clay, carboxylic functionalized MWCNT (COOH-MWCNT), and MMT/MWCNT hybrid nanoparticle. The authors reported enhanced impact properties (15.55%) and compressive strength of composites (10.75%) [81]. MB Bigdilou et al. have reported an increase in CAI for Kevlar polyethylene fibers hybrid composites [142]. Similarly, Y Hu et al. have reported an increase of 8.1–37.8% for multiple ultra-thin CNT, aramid pulp, PBO, and graphene interlayer composites [143]. Table 7 summarizes few important researches that highlight the impact of CNTs incorporation in various composites. Tensile and flexural strength is increased by 50% and compressive strength by 30% after adding the CNTs in Portland

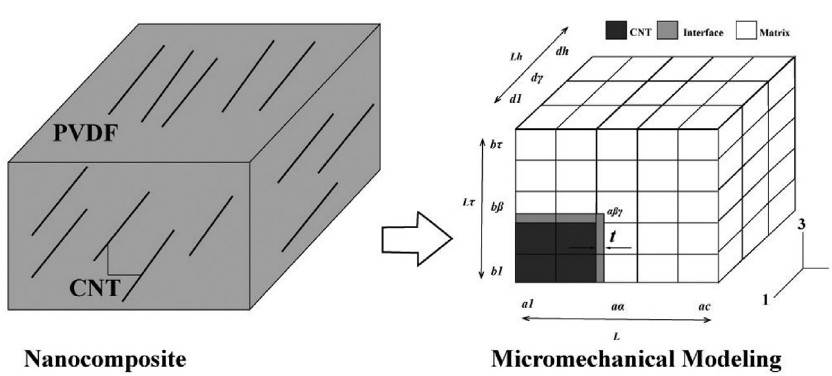

Fig. 24 – Micromechanical model of nanocomposite [144].

Table 8 – Classification of Properties based on the Predictive Model.

Year, Reference	Predictive Model	Classification of properties	Accuracy
2015 [145],	Coarse-grain Model	Elastic and fracture behavior, Mechanical behavior, Mass density	Highly accurate with the increase in computational speed
2015 [146],	Representative volume element (RVE), Mori–Tanaka micromechanics scheme, and atomistic-based continuum (ABC) multiscale modeling technique	Interfacial and mechanical properties	High
2016 [147],	Finite element (FE) method	The role of the interphase on mechanical performance, stress analysis	High
2017 [144],	Simplified Unit Cell (SUC) micromechanical model	Electrical, Thermal, and Elastic	Moderate
2018 [148],	Realistic representative volume elements (RVE)	Conductivity, elastic response, and strain sensing capability of conductive composites	Moderate
2019 [149],	Halpin–Tsai (H–T) micromechanical model	Elastic modulus and tensile strength	High
2019 [150],	3D finite element multi-scale modeling	Mechanical properties	High
2020 [151],	Electrical analytical the model with the Monte-Carlo method	Electrical conductivity	High
2020 [151],	Volume element model	Mechanical response of the nanocomposite under tension	High
2020 [152],	Square representative volume element (RVE)	Storage modulus, loss modulus, and strain energy	High
2021 [122],	CNTNet regression module (image-based deep learning classifier module)	CNTs forest stiffness and buckling load properties	>91%
2021 [153],	Molecular dynamics (MD) simulations with INTERFACE and reactive INTERFACE force fields (IFF and IFF-R)	Mechanical Properties	Moderate
2022 [154],	Multilayer perception	Stress–strain	>99.8%
2022 [154],	A one-dimensional convolutional neural network	Stress–strain	>99.8%
2022 [154],	Residual neural network	Stress–strain	>99%

cement-based material as given in Table 7 [135]. In another study, an addition of 1.5% MWCNTs in woven carbon fabric composite has increased toughness by 50% [112]. Rahimian-Koloor et al. conducted a study that the defects are induced in the CNTs while processing. Defective SWCNTs produced lower mechanical properties as compared to pristine SWCNTs [12].

5.3. *Prediction of the mechanical behavior*

A Micromechanical model of nanocomposite is given in Fig. 24 [144].

Apart from testing, there are also some techniques that can be employed to predict the behavior of polymers reinforced with CNTs. Some of these predictive techniques are mentioned in Table 8.

Most of the methods presented in Table 8, such as the representative volume element (RVE), Finite element (FE), and Neural Networks, make good predictions for the properties they intend to classify. In this regard, machine learning and deep learning methods are more preferred, as they can provide extremely high accuracy for predictions.

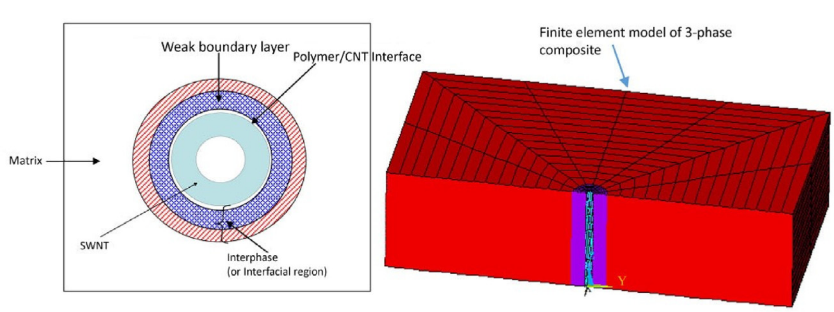

Fig. 25 – Schematic representation of Polymer/SWCNTs [129].

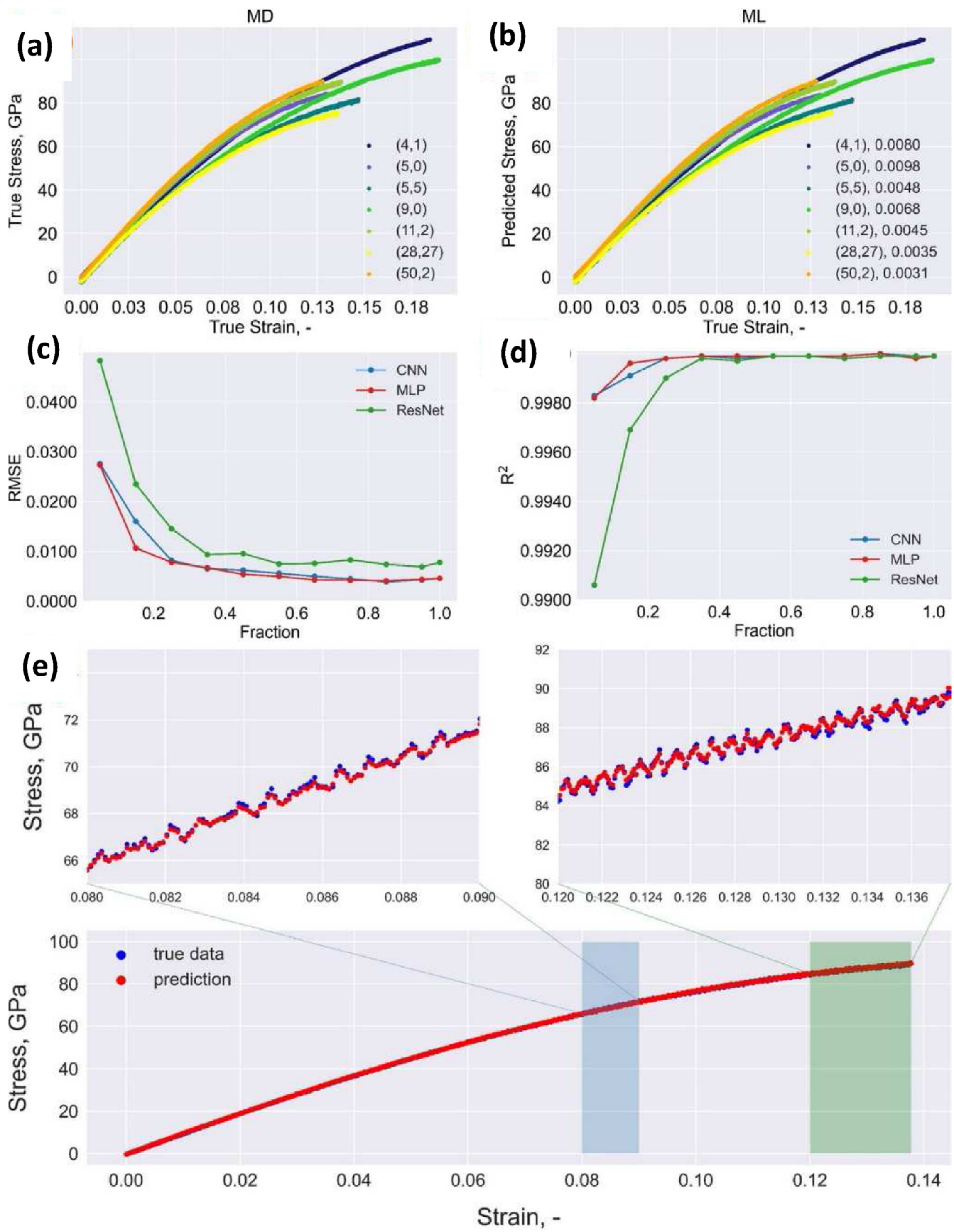

Fig. 26 – Comparison between molecular dynamics (a) and machine learning (b) models for prediction of stress curves, (c) comparison of root-mean-square-Error (RMSE) between different deep learning prediction techniques, (d) R2 for different techniques, (e) thermal fluctuations of true stress-true strain showing true data curve and predicted curve from deep learning technique [154].

A schematic cross-section view of a polymer/SWCNT composite is shown in Fig. 25. The image illustrated the interphase, interface, weak boundary layer, and a corresponding finite element model [147].

Fig. 26 (a, b) shows the comparison between conventional molecular dynamic (MD) and machine learning (ML) models for predicting the properties of polymers reinforced with CNTs. The machine learning models are shown to be more

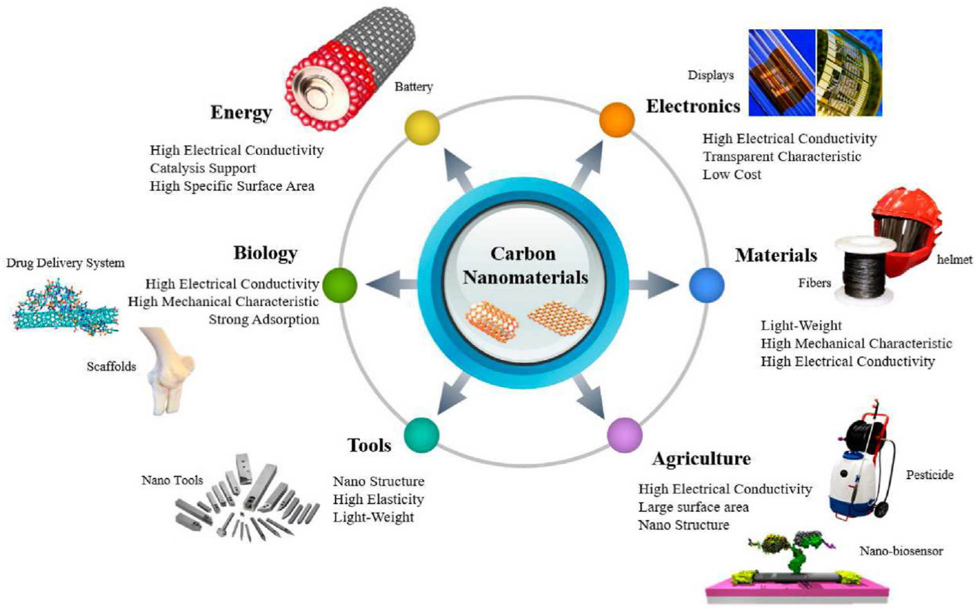

Fig. 27 – Applications of carbon nano tubes (CNTs).

accurate in comparison to conventional predicting schemes. Amongst different machine learning and deep learning models, the convolutional neural network (CNN) model was found to be the most accurate, as shown in Fig. 26(c) and (d) respectively [154]. The model gives an extremely accurate prediction for stress, as depicted in Fig. 26(e).

Fig. 26 represents the data achieved by performing the mechanical test. The data represented in (a) and (b) show that molecular dynamics and machine learning show almost the same results. The strain is directly proportional to stress, and it increases with the increase in stress value. The data shows that (4,1) and (9,0) show the maximum strain at a higher stress value. Fig. 26 (c) shows that ResNet has a higher RMSE value while CNN and MLP and has almost the same results. Fig. 26(d) represents that ResNet has a lower R^2 at the start, but from almost 3.8 all the techniques have the same R^2 value. It is shown in Fig. 26 that the thermal fluctuations of true stress-true strain, both true and predicted graphs, have the same values and therefore their graphs overlap.

6. Study implications and future research

Nowadays, the mechanical properties of CNT reinforced polymer composite are highly addressed by the dispersion of CNTs and its adhesion with a polymer matrix. Consequently, the interfacial characteristics associated with load transfer between polymer matrix and nanotubes are not studied generally because of complex spectral shifts, linked to two dimensional CNTs. These subject properties may be analyzed through complex methods, i.e. Raman-Brillouin Scattering for micro-level and simple methods, i.e. notch flexure and double cantilever beam testing for macro-level physical testing of the CNTs specialized composite.

Conclusively, CNTs have worldwide acceptability for number of applications in many fields because of highly attractive physiochemical structural abilities. The abilities and properties are highly linked with graphene sheets and number of graphene layers making CNTs. The sensors based on CNTs, because of their properties i.e. fast response time, sensitivity, stability and luminesce characteristics are considered to be utilized under applications of smart electronics and related fields. Furthermore, the CNTs are also used under applications related to the field of nanotechnology for the fabrication of smart fuel cells, batteries and electrodes for storage of energy and water treatment. Some of the applications of CNTs are parented in Fig. 27. Subsequently, precise and efficient separation or extraction is highly observed utilizing the CNTs for the removal of contaminants from sample materials. Generally, CNTs presents greater electrochemical properties with higher control. CNTs can store hydrogen energy in them and that is the reason they are considered as extraordinary multipurpose efficient smart material.

However, the aforementioned properties and respective methods are being covered in number of researches separately but there is a big research gap of utilizing these properties simultaneously for quality valuation and characterization of composites as per required properties or end-use applications. Moreover, this gap may be uplifted from laboratory scale to industrial ones with progressive results and ultimately lead to commercialization of these innovative solutions.

7. Conclusion

The study has reviewed aspects of mechanical properties, formation, and characterization of carbon nanotubes reinforced epoxy polymer composites. The systematic literature research method helped to establish that the field of such materials is extensively studied and is currently at a growing stage. The key factors determining the mechanical properties of CNT composites include the type of nanomaterial, which is in turn determined by the synthesis method, subsequent surface treatment, functionalization, and the uniformity of CNTs distribution inside the epoxy matrix.

The strategy of monitoring properties of CNT polymer composites on different levels includes the control of the material's structure and nanotube particle distribution by microscopy (TEM, SEM), and control of the material's aggregation by coupled Raman and Brillouin scattering. A depolarized DLS may also be used for determining the mean diameter and length of individually dispersed single-walled CNTs by modeling CNTs as rigid rods non-destructively with the help of calculated translational and rotational diffusion coefficients. Tensile strength and modulus form the next level of composite properties, which may be measured with conventional methods. Furthermore, more specific properties such as end-notched flexure and double cantilever beam testing, compression-after-impact, and low-velocity impact testing may be employed to characterize the deformation of the CNT-reinforced matrix in different conditions and their respective fracture behavior.

Different dispersion techniques were also reviewed, such as mechanical mixing, solvent spraying technique, chemical functionalization, and spark plasma sintering. Compared to other techniques, spark plasma sintering was considered the most effective technique, as it enables grain growth suppression, thus helping to realize uniform dispersibility.

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Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper

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