



# **Ceramic-Carbon Nanotube Composites and Their Potential Applications**

Submitted by **Hamed Parham**

to the University of Exeter as a thesis for the degree of  
Doctor of Philosophy in Engineering  
In December 2012

This thesis is available for Library use on the understanding that it is copyright material  
and that no quotation from the thesis may be published without proper  
acknowledgement.

I certify that all material in this thesis which is not my own work has been identified and  
that no material has previously been submitted and approved for the award of a degree  
by this or any other University.

Signature: .....  .....

## Abstract

Carbon nanotubes (CNTs) have been the subject of intensive research for nearly two decades, and this is due to their exceptional lightness, large aspect ratio, extraordinary mechanical, electrical, thermal properties and additional multi-functional characteristics. Ceramics have high stiffness and good thermal stability with a relatively low density, and they are an important constituent in the fabrication of advanced composites where high thermal and chemical stability are important. However, brittleness has limited their application in many structural applications. The combination of ceramic (alumina in particular) and CNTs, endeavouring to develop functional composites, offers a very attractive system for research and development. The fabrication of such alumina-CNT composites at bulk scale is therefore highly desirable for industrial applications. However, the synthesis of such composites possesses many technical challenges which need to be addressed. Poor synergy between the matrix and CNTs, potential damage to CNTs, obtaining a uniform and agglomeration-free distribution of CNTs within the matrix, and high cost of CNTs and processes involved in their composite fabrication have proved to be the significant challenges.

In this thesis, the focuses are laid on addressing these issues and on the fabrication of specially engineered composites for particular applications such as filter and composites with improved mechanical properties. In this regard, it has been tried to directly fabricate CNTs in different ceramic matrices based on the application requirements. After that, the critical issues and challenges in the fabrication of these functional materials have been clearly investigated and by introducing novel methods and approaches, it has been tried to solve these problems.

Also, a new polymer-ceramic-CNT composite has been fabricated by using two different thermoset (epoxy resin) and thermoplastic (polyamide 12) matrices. In this regard, good interfacial bonding between the composite elements along with good wettability of ceramic and CNTs with polymer had to be addressed as critical issues and challenges in the fabrication process. If the adherence at the interface is not strong enough, the material will tear and fail easier. In contrary, a tailored functionalization of CNTs can lead to an improved wettability and as the results, strong interfacial adhesion and bonding between the composite elements. These dominating factors will improve the degree of filling which results in existence of fewer voids inside the composite. These voids will act later as stress points and reduce the composite strength. At the end, the mechanical properties of the fabricated samples have been assessed.

The CNT filters have been tested in the removal of bioorganic (yeast cells) and inorganic (heavy metal ions) contaminants from water, and of particulates from air, and they all showed very promising results. More than 99.6% of the air particles (size ranges from 0.3 to 10  $\mu\text{m}$ ) were filtered using 300 mm long CNT filter. A complete removal of heavy metal ions from water was reported particularly for single ion. 98% of the yeast cells were filtered. Different factors involved in the filtration efficiency such as ceramic pore size, length of filters, CNT loading and injection rates have also been discussed.

Furthermore, the mechanical properties (compression test, hardness and impact test) of the composite materials (including ceramic-CNT, epoxy resin-ceramic-CNT and polyamide-ceramic-CNT composites) have been assessed. During impact test, the epoxy resin-ceramic-CNT composite absorbed 117.2% and 32.7% more energy compared to the pure epoxy resin and epoxy resin-ceramic composite, respectively. The epoxy resin-ceramic-CNT composite sustained 40% more elastic deformation before breakage compared to the epoxy resin-ceramic composite as a result of the CNT reinforcement. The addition of CNTs to the polyamide12-ceramic composite increased its yield stress by 41%.

All of these results represent a big leap towards practical applications for the composite reported in the thesis, which may open up new opportunities for CNT engineering at industrial scales, due to the easy fabrication methods introduced and the promising performance they have exhibited.

## **Presentations and Publications**

- Parham, H., Bates, S., Xia, Y. & Zhu, Y. A highly efficient and versatile carbon nanotube/ceramic composite filter. *Carbon* 54, 215-223, (2013).
- Parham H, Kennedy A, Zhu Y. Preparation of porous alumina–carbon nanotube composites via direct growth of carbon nanotubes. *Composites Science and Technology*. 2011;71(15):1739-45.
- Parham H, Bates S, Xia Y, Zhu Y. Carbon nanotube composite filter. 3rd International Conference on Nanotechnology: Fundamentals and Applications. Montreal, Quebec, Canada, 7-9 August 2012.
- Carbon nanotube-reinforced porous Al<sub>2</sub>O<sub>3</sub> nanocomposites filters. Tenth international conference on materials chemistry (MC10). Manchester, UK. 4-7 July 2012.

## Table of Contents

Abstract .....	2
Presentations and Publications .....	4
Table of Contents .....	5
Table of Figures.....	9
Acknowledgments.....	25
List of abbreviations.....	26
Chapter 1: Introduction.....	27
Chapter 2: Literature Review.....	32
2. 1. Introduction.....	32
2. 2. History.....	32
2. 3. Morphology and Atomic structure of CNTs.....	32
2. 3. 1. Single-walled CNTs .....	33
2. 3. 2. Multi-walled CNTs .....	35
2. 4. Fabrication methods of CNTs.....	37
2. 4. 1. Arc-discharge .....	38
2. 4. 2. Laser ablation.....	38
2. 4. 3. CVD technique.....	39
2. 5. Properties of CNTs .....	42
2. 5. 1. Mechanical Strength.....	43
2. 5. 2. Thermal stability .....	44
2. 5. 3. Chemical reactivity.....	45
2. 6. Applications of CNTs .....	45
2. 7. CNT-reinforced composites .....	47
2. 7. 1. Polymer/CNT composites .....	48
2. 7. 2. Metal/CNT composites .....	50
2. 7. 3. Dense ceramic/CNT composites .....	51
2. 7. 4. Porous ceramic/CNT composite.....	59
2. 7. 5. Polymer/ceramic composite.....	60

2. 8.	CNT nanofiltration properties .....	64
2. 8. 1.	Air particulate filtration.....	65
2. 8. 2.	Biological contamination filtration .....	69
2. 8. 3.	Heavy metal ions adsorption .....	72
2. 8. 4.	Other CNT filtration applications .....	75
2. 9.	Toxicity of CNTs .....	77
2. 10.	Summary .....	79
Chapter 3: Experimental methodology.....		80
3. 1.	Introduction .....	80
3. 2.	In-situ fabrication of high density ceramic-CNT composite ...	80
3.2.1.	Materials .....	80
3. 2. 2.	Sample preparation.....	82
3. 3.	In-situ fabrication of low density ceramic-CNT composite.....	83
3. 3. 1.	Materials.....	83
3. 3. 2.	Sample preparation.....	84
3. 4.	Filter tests preparation .....	86
3. 5.	Polymer-ceramic composite reinforced with CNT .....	88
3. 5. 1.	Epoxy resin-ceramic-CNT composite.....	88
3. 5. 2.	Polyamide-ceramic-CNT composite .....	91
3.6.	Structural characterization.....	92
3. 6. 1.	Scanning electron microscopy.....	92
3. 6. 2.	Transmission Electron Microscopy .....	93
3. 6. 3.	EDX .....	94
3. 6. 4.	XRD .....	95
3. 6. 5.	$\mu$ CT scan .....	95
3. 6. 6.	Density.....	96
3. 6. 7.	Thermal investigations .....	96
3. 7.	Mechanical property evaluation .....	96
3. 8.	Summary .....	99

Chapter 4: Ceramic/CNT composite .....	100
4.1. In-situ fabrication of high density ceramic/CNT composite ..	100
4. 1. 1. Introduction .....	100
4. 1. 2. Effect of carbon source and catalyst .....	101
4. 1. 3. Yield and quality of nanocomposite .....	109
4. 1. 4. Growth mechanism .....	113
4. 2. Low density Ceramic/CNT composite .....	117
4. 2. 1. Introduction .....	117
4. 2. 2. Ceramic/CNT composite synthesis parameters.....	118
4. 2. 3. Yield of CNTs.....	127
4. 2. 4. CNT characterization .....	133
4. 3. Conclusion .....	136
Chapter 5: Ceramic-Polymer composite reinforced with CNTs .....	138
5. 1. Introduction .....	138
5. 2. Epoxy resin-ceramic-CNT composite .....	139
5. 2. 1. Sample preparation.....	140
5. 3. Polyamide 12-ceramic-CNT composite .....	146
5. 3. 1. Sample preparation.....	147
5. 4. Conclusion .....	152
Chapter 6: Filtration properties of low density ceramic/CNT composite ...	153
6. 1. Introduction .....	153
6. 2. Wettability and functionalization of filter .....	154
6. 3. Yeast filtration.....	155
6. 4. Air filtration .....	162
6. 5. Heavy metal ions filtration .....	170
6. 6. Conclusion .....	177
Chapter 7: Compression and impact behaviour of polymer-ceramic-CNT composites .....	179
7. 1. Introduction .....	179

7. 2. Compression strength .....	179
7. 2. 1. Epoxy resin-ceramic-CNT composite.....	179
7. 2. 2. Polyamide-ceramic-CNT composite .....	182
7. 3. Hardness .....	187
7. 4. Impact and toughness.....	188
7. 4. 1. Epoxy resin based composite .....	188
7. 4. 2. Polyamide based composite .....	196
7. 4. Conclusion .....	199
Chapter 8. Summary and conclusions.....	200
Chapter 9. Future work .....	205
References.....	207

### Table of Figures

Fig. 2. 1. Different hybridization states and forms of carbon-based nanomaterials [45] .....	33
Fig. 2. 2. (a) Schematic of the fabrication of SWCNT which is a strip cut from an infinite graphene sheet rolled up to form a tube [46]. (b) Sketch of the vector of helicity $C_h$ and angle of helicity $\theta$ [47]. .....	34
Fig. 2. 3. Sketches of three different SWCNT structures: (a) zig-zag $(n, 0)$ , (b) armchair $(n, n)$ and (c) helical $(n, m)$ nanotube [37]. .....	35
Fig. 2. 4. (a) Transmission Electron Microscopy (TEM) image of a concentric multi-walled CNT made by the electric arc-discharge method. The inset illustrates a sketch of the Russian doll like arrangement of graphene. (b) TEM images of herringbone and bamboo multi-walled CNTs (bh-MWCNT) prepared by CO disproportionation [48]. .....	36
Fig. 2. 5. (a) TEM image of a bh-MWCNT which shows nearly the periodic nature of texture that happens frequently [50], (b) TEM image of a bc-MWCNT [51] .....	37
Fig. 2. 6. Schematic of an electric arc-discharge reactor [19] .....	38
Fig. 2. 7. Schematic of the laser ablation technique with a continuous $CO_2$ laser device [19] .....	39
Fig. 2. 8. Schematic of the CVD process. ....	40



Fig. 2. 9. Growth mechanism of CNTs over metal catalyst suggesting two possible methods known as (a) "tip growth" and (b) "root growth" [73].	41
Fig. 2. 10. Well-aligned mats of CNTs grown on glass by the PECVD process [78].	42
Fig. 2. 11. The effect of catalyst size on the diameters of CNTs in a CVD process [78]	42
Fig. 2. 12. Tensile loading and fracture behaviour of CNTs during tests [11, 80]	43
Fig. 2. 13. (a) Schematic simulation of a deformed CNT when they are loaded [37], and (b) in-situ bending sequence on a single CNT [82].	44
Fig. 2. 14. Schematic of four different categories of nanocomposite from the concept of new material designs based on (a) Niihara [113], and (b) Mukherjee [114] classification.	48
Fig. 2. 15. (a) TEM image of a Polystyrene/CNT composite (arrows show regions of polymer shrinkage and inset illustrates the length distribution of CNTs [120]. (b) Epoxy/CNT composite which shows the alignment of the nanotubes in the direction of cutting with microtome [121].	49
Fig. 2. 16. SEM micrograph showing two regional structures of composite consisting of CNT-free (i) and Cu-CNT (ii) parts [127].	51
Fig. 2. 17. Schematic of the reinforcement toughening: (1) crack deflection, (2) crack bridging and (3) fibre pull-out [135].	52
Fig. 2. 18. Schematic and actual SEM images of crack propagation (a) and the bridging mechanism (b) [24, 137, 138].	52
Fig. 2. 19. Schematic and SEM images of the pull-out mechanism in carbon fibres (a), and CNTs (b) reinforced composites [137, 139].	53
Fig. 2. 20. SEM images of nanocomposites fabricated from powder processing. (a) by SPS process [26]; (b) by hot-pressed method [2]	55
Fig. 2. 21. Schematic of a hot pressing furnace [142].	56
Fig. 2. 22. SEM images of the production of amorphous carbons at grain boundary junctions of a composite as the result of high temperature involved in hot-pressing process [116].	56

Fig. 2. 23. (a) Photograph of composite created by extrusion during the early stage (left hand) and final (right hand) stage of the process, and (b) alignment of CNTs along the extrusion direction [143].	57
Fig. 2. 24. Schematic of a SPS apparatus [145].	58
Fig. 2. 25. High resolution TEM micrograph of SWCNT/alumina composite showing (a) aligned SWCNT bundle, (b) disordered graphite and some SWCNTs (arrows), (c) carbon nano-onions (arrows), and (d) diamond nanocrystals [146].	59
Fig. 2. 26. Schematic of the fabrication method for highly ordered CNTs in ceramics [148].	60
Fig. 2. 27. Optical images of a) $\text{SiO}_2\text{.ZrO}_2\text{/polymer}$ , and b) $\text{SiC/polymer}$ composite material [42].	61
Fig. 2. 28. Compression stresses of a) $\text{SiO}_2\text{.ZrO}_2\text{/polymer}$ and b) $\text{SiC/polymer}$ [42].	62
Fig. 2. 29. Variation in weight loss of ball as a function of sliding distance for a) $\text{SiO}_2\text{.ZrO}_2\text{/polymer}$ and b) $\text{SiC/polymer}$ [42].	62
Fig. 2. 30. (a) Microstructure of the ceramic-elastomer composite, (b) crack propagation in composite after compression test, (c) cracked ceramic matrix stuck by the elastomer [41], and (d) stress-strain graph of porous ceramic, elastomer and ceramic-elastomer composite [40].	63
Fig. 2. 31. Collection efficiency of the molecular capture mechanism as a function of particle size. The minimum efficiency is contributed to the MPPS [173].	66
Fig. 2. 32 SEM images of various CNT membranes grown on microstructured and oxidized Si chips: synthesis times of (a) 0, (b) 20, (c) 30, and (d) 40 min. (e) Pressure drop vs flow rate of nanotube membranes. Samples with longer growth time show decreased permeability due to the tortuous CNT films and closed up macroscopic holes [8].	67
Fig. 2. 33. (a and b) SEM images of NaCl particles collected onto the filter [174]. Performance comparison of the raw metal filter and the metal-CNT-filter. (a) Pressure drop is not significantly changed by the direct growth of CNTs. (b) Filtration efficiency increases by the CNT growth [36].	68

Fig. 2. 34. (a) Pressure drops, (b) particle filtration efficiencies for glass fibre-CNT filter [175].....	69
Fig. 2. 35. SEM images of a) the cross-section of SWCNT layer and b) E. coli cells on the base membrane [10].....	70
Fig. 2. 36. Cylindrical membrane filter made by aligned CNTs for removing hydrocarbons from petroleum wastes and bacteria and virus separation from water [9]. .....	71
Fig. 2. 37. SEM images of bundles of SWCNTs wound around <i>S. mutans</i> (a); the bacteria adhered to the meshwork comprising bundles of SWCNTs (b); 30-MWCNTs wound around bacteria (c); 200-MWCNTs adhered but did not wind (d); The white arrows indicate CNTs. The black arrows indicate fibrous substances produced by bacteria [177]. .....	71
Fig. 2. 38. SEM images of E. coli cells exposed to MWCNTs (a) and SWCNTs (b) [178] and also Salmonella cells exposed to acid treated (-COOH) SWCNTs (c) MWCNTS(d) [179].....	72
Fig. 2. 39. (a) A monolithic sponge with a size of 4 cm× 3 cm× 0.8 cm, (b) a cross-sectional SEM image of the sponge, (c) TEM image of CNT shows their thin and good quality, (d) illustration of sponge and its open pores, (e) comparison between polyurethane and CNT sponges in which the CNT sponge floating on the top while the polyurethane sponge has sunk after adsorbing water, (f & g) flexibility of the sponge is shown after bending and twisting three round turns without breaking, and (h) densification of the cubic shape sponge into a small pellet and its full recovery to first shape after adsorbing ethanol [195].....	75
Fig. 2. 40. Large area oil clean-up demonstration. A diesel oil film with an area of 227 cm <sup>2</sup> and 2 grams in total weight spreading on water and the densified sponge (pellet shape) placed in the centre (left panel). The cleaned water surface after complete oil removal process is shown in the middle. The images on right hand side illustrate the change in volume and shape of sponge after adsorption from a 6 mm diameter spherical pellet to a rectangular monolith (2 cm× 1.4 cm× 0.6 cm) [195]. .....	76
Fig. 2. 41. SEM and optical images of a stainless steel mesh before and after the synthesis of nanotubes (a, b and c), and separation of diesel from water using the membrane (d and e) [196]. .....	77

Fig. 2. 42. TEM images of O-CNTs before (a) and after (b) adsorption of tar in the MS, and (c) the removal efficiency of tar ( $\eta_{tar}$ ) as a function of O-CNTs mass ( $M_o$ ) [197].	77
Fig. 2. 43. Lungs from mice instilled with 0.5 mg of a test material per mouse. (A) Serum control. (B) CNT. The portions of the lung receiving CNT have an abnormal appearance [198].	78
Fig. 2. 44. (a) Penetration of CNTs in bacteria has inhibited their growth [199], and (b) harmful effect of CNTs on <i>Daphnia magna</i> (large numbers of tubes filling the gut track at 45 min and 1 h) [202].	79
Fig. 3.1. (a) Photograph and (b) SEM image of an alumina brick (10×10×15 mm) used as the substrate for CNT growth.	80
Fig. 3. 2. Schematic of the CVD set-up for the synthesis process	83
Fig. 3.3. (a) Photograph (disc o.d. 27× 10 mm) and (b) SEM image of ceramic used as the substrate for CNT growth.	84
Fig. 3. 4. A schematic of the sample preparation and experimental set-up for the in-situ fabrication of CNTs on a fitted ceramic foam inside quartz tube.	85
Fig. 3. 5. A schematic of the acid functionalization process of the ceramic-CNT filters	86
Fig. 3.6. Experimental set-up for yeast and heavy metal filtration.	87
Fig. 3.7. Lighthouse portable airborne laser particle counter (SOLAIR 3100) with filter placed on its inlet.	88
Fig. 3. 8. Silica mould made for infiltration of epoxy resin into the ceramic matrix.	89
Fig. 3. 9. Photograph of the aluminium mould used for the infiltration of polymer inside ceramic matrix (a); and schematic of mould showing three parts of mould: part I: the passage way for air to be sucked out in vacuum chamber, part II: where ceramic located inside the mould, and part III: the bank for required amount of polymer which will be injected after 24 h vacuuming by turning the screw.	90
Fig. 3. 10. Optical images of samples after infiltration with epoxy resin.	90
Fig. 3. 11. Photograph of samples after polishing without (left) and with (right) CNTs.	91

Fig. 3. 12. Photograph of samples after (a) injection of PA 12, and (b) polishing.....	92
Fig. 3. 13. (a) Schematic of SEM. (b) Schematic diagram of SE and BSE generation by primary beam and their collection by the detector to produce the SEM image [205]. .....	93
Fig. 3. 14. Schematic illustration of TEM[205] .....	94
Fig. 3. 15. Photograph of the Instron Dynatup 9250HV test machine used for the impact tests. A special sample holder with the test specimen was fixed between the two plates.....	97
Fig. 3.16.The specially designed mould for increasing the diameter of the composite samples (located in the middle of the holes) by adding an extra epoxy resin ring around the central composites. ....	98
Fig. 3. 17. Optical image of PA12-ceramic-CNT composite after increasing its diameter by the addition of epoxy resin for impact test. ....	98
Fig. 4.1. Schematic fabrication method of producing highly ordered CNTs inside AAO [148] .....	100
Fig. 4.2. SEM images of: (a) surface, and (b) centre of a sample, revealing the non-uniform distribution of the catalyst particle at various sections of a ceramic matrix. ....	102
Fig. 4. 3. SEM images showing the agglomeration of catalyst particles in one place which resulted in a regional growth of CNTs across the sample. ....	103
Fig. 4. 4. SEM images show the uniform distributions of catalyst particles inside the ceramic matrices at high (a) and low (b) magnifications .....	104
Fig. 4. 5. SEM images: (a) The oily surface of the sample owing to the tar pitch, (b) different carbon materials produced, and (c) sphere carbon being the dominant products. ....	106
Fig. 4. 6. A photograph of the acetone solution containing nickel nitrite and camphor.....	108
Fig. 4. 7. SEM images of the surface of a sample synthesised using camphor as the carbon source and nickel nitrate as the catalyst at low (a) and high (b) magnifications.....	109

Fig. 4. 8. (a) The alumina brick dimensions. The broken line illustrates where the sample was sectioned for SEM investigation. (b) Showing the locations (A, B and C) examined after CNT growth. ....	109
Fig. 4. 9. SEM images of CNTs grown inside the ceramic matrix, taken from centre (a, b), ¼ thickness position (c, d), and near the surface of the sample (e, f). ....	110
Fig. 4. 10. SEM images. Agglomerations of the catalyst particles circled in (a), leading to the formation of clumps of CNT in matrix circled in (b). ....	112
Fig. 4. 11. TEM images of CNTs collected from the composite sample. (a) The hollow structural feature of the nanotubes is visible, and the high contrast black dots are the catalyst particles, and (b) showing the atomic inter-layer distance is 0.34 nm and an attached carbon particle, circled. ....	112
Fig. 4. 12. SEM images: (a) evidence of the 'root' growth of CNTs, and (b) CNTs with catalyst-free tips. ....	114
Fig. 4. 13. Thermal decomposition of nickel (II) nitrate hexahydrate [215] ..	115
Fig. 4. 14. TGA result of catalyst particle under Ar atmosphere which indicates that the catalyst decomposition finalised at 325°C with a total weight loss of 75%. ....	115
Fig. 4. 15. Nickel-aluminium-oxygen phase diagram at constant temperatures [216, 217]. ....	116
Fig. 4. 16. Open-cell ceramic foam composed of three parts of strut, cell, and window [222]. ....	118
Fig. 4. 17. SEM image of big catalyst clusters formed on the ceramic. The slow process of water removal gives enough time for the nickel nitrate particles to agglomerate, especially at the bottom of the sample due to gravity. ....	120
Fig. 4. 18. (a) SEM images of sample using SE shows the growth of CNTs near some big particles, (b) SEM BSE image of the same place proves the big particles to be Ni(NO <sub>3</sub> ) <sub>2</sub> . ....	120
Fig. 4. 19. (a) SEM images of the rough surface of a ceramic foam after heat treatment in a furnace at 780°C (b) Tiny catalyst particles are shown in the inset at higher magnification. ....	121

Fig. 4. 20. Poor quality of samples were observed using styrene as carbon source.(a) Thick CNTs and CNFs grown on the surface of a ceramic foam; and (b) massive amounts of amorphous carbons were detected at inner layers of ceramic foam. ....	122
Fig. 4. 21. Illustration of quality of sample at two surfaces faced to inlet (left) and outlet (right) of furnace revealing dominating effect of carrier gas speed on quality of sample. ....	123
Fig. 4. 22. Fabrication of amorphous carbon (a) and CNFs (arrowed in b) beside CNTs dropping quality of composite as result of high injection rate of carbon source into the furnace. ....	123
Fig. 4. 23. SEM images showing the quality of CNTs on surfaces facing the inlet (left) and outlet (right) stream of furnace. Sample was produced at 725° .....	125
Fig. 4. 24. SEM image of a sample prepared at 850°C showing the CNTs covered by a large amount of amorphous carbon, taken from the surface facing the front gas inlet side of the sample. ....	125
Fig. 4. 25. a, Low magnification SEM images of a porous ceramic matrix before the CNT growth; b, Low magnification SEM image of the ceramic/CNTs composite; c, a $\mu$ -CT scan 3D image of a ceramic substrate showing the interconnectivity feature of the foam; and d, SEM image of CNTs grown on ceramic foam produced at 780°C. ....	126
Fig. 4. 26. A thick layer of CNT collected from the quartz working tube when using ferrocene mixed with carbon source at low (a) and high (b) magnifications. ....	128
Fig. 4. 27. Iron nanoparticles (bright particles) stuck among CNTs as the result of addition of ferrocene. ....	129
Fig. 4. 28. SEM images show addition of nickel nitrate to the camphor-acetone solution result in deposition of nickel particles inside the sample affecting its quality at high (a, c) and low (b, d) magnifications. ....	130
Fig. 4. 29. TGA results show the thermal stability (up to 630°C in argon) of CNTs grown on ceramic matrix (a), and decomposition temperature of nickel nitrate in argon, (b). ....	132
Fig. 4. 30. TGA of ceramic/CNT composite conducted in air that shows yield of 3.1 wt. % for CNT growth. ....	132

Fig. 4. 31. SEM images of samples produced during the multi-stage process for increasing the yield of CNTs. It is clear that the sample surface is covered with amorphous carbon and the pre-produced CNTs have been destroyed. ....	133
Fig. 4. 32. SEM images of the sample after synthesising large amount of CNTs at low (a) and high (b) magnifications.....	134
Fig. 4. 33. (a) TEM image of CNTs showing the structure feature of the produced nanotubes; and (b), a higher magnification TEM image of the zoomed part in (a) which shows one catalyst particle attached to the tip of a CNT. ....	134
Fig. 4. 34. TEM images of CNT (a) free of catalyst at tip, and (b) catalyst particles encapsulated at both ends. ....	135
Fig. 4. 35. TEM image shows the length of a CNT.....	135
Fig. 5.1. Schematic illustration of the molecular structure of DGEBA, DDM and PEO [240, 241] .....	140
Fig. 5. 2. Photograph of a plain ceramic matrix (left) before and after polymer infiltration (middle), and the ceramic-CNT-epoxy resin composite (right). ....	142
Fig. 5. 3. Optical images of the cross-section of the epoxy resin-ceramic composite (a), and epoxy resin-ceramic-CNT composite (b). Low magnification SEM image of the composite containing CNTs (c). ....	142
Fig. 5. 4. TGA result for pure epoxy resin in air which suggests its stability up to 350°C in air.....	143
Fig. 5. 5. SEM images of CNTs embedded inside the epoxy resin at different magnifications.....	145
Fig. 5. 6. Densities of pure epoxy resin, polymer-ceramic and polymer-ceramic CNT composites obtained by Archimedes principal. ....	146
Fig. 5. 7. $\mu$ -CT scan image of a broken ceramic substrate as the result of high injection pressure. ....	148
Fig. 5. 8. $\mu$ -CT scan proved the successful infiltration of PA12 into the porous ceramic substrate with more than 99% degree of filling. ....	148
Fig. 5. 9. Photograph of the polished surface of a polyamide-ceramic-CNT composite. Black curves, grey parts and white bright parts are contributed	



to the CNT, PA12 and ceramic, respectively. The interconnectivity feature of the pores is also visible in this photo.....	149
Fig. 5. 10. SEM images of CNTs embedded inside PA12 at different magnifications.....	150
Fig. 5. 11. Densities of PA12, PA12-ceramic and PA12-ceramic CNT composites obtained by Archimedes principal.....	152
Fig. 6. 1. An illustration of the surface hydrophobic behaviour of filter with as-CNTs .....	155
Fig. 6. 2. SEM images of fresh yeast cells placed on CNT nanofilter at different magnifications.....	156
Fig. 6. 3. SEM images of the filtered yeast cells by CNTs at different magnifications. The tangled CNTs are closely connected with the yeast cells and have immobilised them. ....	157
Fig. 6. 4. Composite filter efficiency for yeast cells and the role of CNTs in the filtration. a, Composite filter efficiency for yeast as a function of filter length and injection rate; b, Composite filter efficiency for yeast as a function of injection rate and filter pore sizes; and c, SEM image of two ceramic substrates with 300 $\mu\text{m}$ (on top) and 500 $\mu\text{m}$ (on bottom) pore sizes. ....	159
Fig. 6. 5. Hollow ceramic foam produced by the supplier.....	160
Fig. 6. 6. Several filters burning after filtration of yeasts by adding acetone, representing a method for the filter regeneration.....	160
Fig. 6. 7. TGA results show the high thermal stability (up to 530 $^{\circ}\text{C}$ in air) of CNTs grown on ceramic matrix (a), showing an unchanged onset weigh loss temperature after burning, and the total weight loss owing to CNT content, (b).....	161
Fig. 6. 8. SEM (a) and TEM (b) images of CNTs after burning the filter, showing their morphologies identical to pristine CNTs.....	161
Fig. 6. 9. Filters maintained their high removal efficiency after three burning cycles.....	162
Fig. 6. 10. Lighthouse portable airborne laser particle counter (SOLAIR 3100) with filter placed on its inlet.....	163

Fig. 6. 11. Air particulate filter efficiency as a function of filter length for 0.3 $\mu\text{m}$ (a), 0.5 $\mu\text{m}$ (b), 1 $\mu\text{m}$ (c), 5 $\mu\text{m}$ (d), and 10 $\mu\text{m}$ particles.....	165
Fig. 6. 12. Filtration efficiency as a function of particle size for CNT-300 filter.....	166
Fig. 6. 13. Pristine CNT and air oxidized CNT filters show similar efficiencies for particulate filtration in air. Figures represent filter efficiency of a pristine filter, pristine CNT-300, and air oxidize CNT-300 for different filter lengths of 50 mm (a), 100 mm (b), 150 mm (c), and 200 mm (d) as a function of particle sizes. ....	167
Fig. 6. 14. a, $\mu$ -CT scan 3D image of a ceramic substrate showing the interconnectivity; b, a schematic illustration of set-up for measuring the pressure drop; and c, the pressure drop of a 10 mm disc as a function of air flow rates. ....	168
Fig. 6. 15. Comparison between pressure drop of 500 $\mu\text{m}$ pore sized filter with and without CNTs. ....	169
Fig. 6. 16. Diesel particulate filter with parallel channels which reduce the pressure drop in diesel engines [254].....	170
Fig. 6. 17. Single metal ( $\text{Cu}^{2+}$ ) adsorption as a function of injection rates using 20 mm long, 300 $\mu\text{m}$ pore-sized filter. ....	170
Fig. 6. 18. (a) Adsorption efficiency of 10 mm long functionalized filters for 30 ml of heavy metal solution containing 5 mg/l of each of $\text{Fe}^{2+}$ , $\text{Cu}^{2+}$ , $\text{Mn}^{2+}$ & $\text{Zn}^{2+}$ . One filter was oxidized in air and the other was treated in acid. (b) The adsorbed amount of metal ions in mg shows different tendencies for various ions when the total adsorbed amount is almost similar. ....	172
Fig. 6. 19. (a) Adsorption efficiency of filters as a function of their lengths, tested using heavy metal solution containing 5 mg/l of each of $\text{Fe}^{2+}$ , $\text{Cu}^{2+}$ , $\text{Mn}^{2+}$ & $\text{Zn}^{2+}$ ; and (b) The saturation point of a 50 mm long filter tested using the same solution. ....	174
Fig. 6. 20. The saturation point of 50 mm long filters tested using heavy metal ion solution containing 5 mg/l of each of $\text{Co}^{2+}$ , $\text{Mn}^{2+}$ and $\text{Zn}^{2+}$ . ....	175
Fig. 6. 21. Filter efficiency for individual metal ions under a competitive condition at different injection rates. ....	175

Fig. 7.1. Compression test results of the plain ceramic, pure epoxy resin, epoxy resin-ceramic composite and epoxy resin-ceramic-CNT composite samples. The plain ceramic graph has been slightly shifted to the right for better observation. ....	181
Fig. 7.2. Optical image of the compressed samples where the left one is the epoxy resin-ceramic-CNT composite and the right one is the epoxy resin-ceramic one. ....	182
Fig. 7.3. The compression test profiles of PA12-ceramic and PA12-ceramic-CNT composites.....	182
Fig. 7.4. The compression test results of the plain ceramic, pure PA12, PA12-ceramic and PA12-ceramic-CNT composite samples. The plain ceramic graph has been slightly shifted to the right for a clearer view. ....	185
Fig. 7.5. SEM images of PA12-ceramic-CNT composite after the compression test at different magnifications. ....	187
Fig. 7.6. Schematic of hardness test and how its results can be affected by different portions of CNT, polymer and ceramic placed under the indenter. ..	188
Fig. 7.7. Impact test profiles of pure epoxy resin (a), epoxy resin-ceramic (b), and epoxy resin-ceramic-CNT (c) composites obtained under constant impact energy of 5 J. ....	190
Fig. 7.8. Optical images of different post impact samples. (a) and (b) represent the pure epoxy resin and epoxy resin-ceramic samples, respectively. (c) and (d) represent the epoxy resin-ceramic CNT composites. ....	191
Fig. 7.9. SEM images of a fracture surface of the epoxy resin-ceramic-CNT composite reveal the bridging mode and pull-out mode of CNTs within the composite acted as the toughening mechanism. ....	195
Fig. 7.10. Impact test results of the pure PA12 (a), PA12-ceramic (b), and PA12-ceramic-CNT (c) composites obtained under constant energy of 5 J....	197
Fig. 7. 11. Optical images of different post impact samples. Pure PA12, PA12-ceramic composite and PA12-ceramic-CNT composites are presented in (a), (b) and (c), respectively. The red circles clearly show the cracks at the surface of samples. ....	197
Table. 2. 1. Unique properties of carbon nanotubes [6] .....	43

Table. 2. 2. SPS conditions and the resultant properties on alumina/SWNT composite [24].	58
Table. 2. 3. Physical properties of carbonaceous nanomaterials and their related environmental applications [45].	65
Table. 2. 4. Maximum sorption capacities of divalent metal ions with CNTs [185]	73
Table. 3. 1. List of catalysts and carbon sources tested for the in-situ growth of CNTs inside porous alumina in order to find out the most effective matches for nanocomposite fabrication.	81
Table. 3. 2. Physical properties of ferrocene, styrene and camphor [203]	82
Table. 3. 3. Number of air particles before filtration	88
Table. 4. 1. A summary of catalysts and carbon sources utilised in this study	101
Table. 4. 2. Physical properties of ferrocene and styrene [203]	105
Table. 4. 3. Physical property of camphor [203]	107
Table. 4. 4. Key differences between the fabrication processes of high and low density ceramic/CNT composites.	119
Table. 4. 5. Weight of samples at different stages of experiment suggesting the yield of CNTs	130
Table. 5. 1. Mechanical properties and fracture toughness of DGEBA/DDM/PEO blends for all PEO contents and cure temperatures [238]	140
Table. 6. 1. Number of air particles before filtration	163
Table. 7. 1. Peak load, energy to maximum load and total energy absorbed during impact test are summarized for the pure epoxy resin, epoxy resin-ceramic and epoxy resin-ceramic-CNT composites.	192
Table. 7. 2. Peak load, energy to maximum load and total energy absorbed during impact test are summarized for pure PA12, PA12-ceramic and PA12-ceramic-CNT composites.	198