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QUARTZ, GLASS, AND GLASS-CERAMIC MATRIX NANOCOMPOSITES; CONTAINING CARBON NANOTUBES: A REVIEW

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ABSTRACT
Various concepts of techniques for incorporating carbon nanotubes in quartz, glass and glass-ceramic matrices are overviewed. Mechanical; in particular fracture toughness, hardness and strength, physical; density and microstructures, and functional; thermal and electrical conductivities of the fabricated CNT-loaded nanocomposites via different processing route and measuring techniques were compared and reported. Processing challenges such as the homogenous dispersion of the CNTs in the quartz, glass and glass-ceramic matrices and the loss of graphitic nanotubes during the consolidation process are still the major impending issues in CNT-quartz/glass/glass-ceramic matrix nanocomposites. There is need to explore in-situ production techniques, spark plasma sintering consolidation method, and controlled colloidal/sol-gel processes for CNT-quartz/glass/glass-ceramic matrix nanocomposites.
Keywords: Quartz, glass-ceramics, carbon nanotubes, nanocomposites, processing techniques, mechanical and functional properties.

INTRODUCTION

Naturally occurring quartzite (crystalline silica), glass, and glass-ceramic possess fascinating mechanical; specifically, hardness and stiffness, and physical functional properties such as thermal conductivity. This latter property could be due to the high density and low porosity of the unreinforced materials. The most problematic properties of ceramics in general (Cho *et al.*, 2009a; Curtin and Sheldon, 2004) and silica, in particular, are low fracture toughness and thermal shock resistance. There has been a wealth of literature on the enhancement of thermal conductivity of quartz; which is one of the key functional properties that determine the thermal shock. Also, in the available reports, variable production parameters ranging from moulding techniques, grain size/distribution, binder/mineralizer, additives, and micro/nano-phase fiber/particle reinforcement were controlled and optimized in order to address the intrinsic behavior of thermal shock and mechanical fracture in quartz, glass and glass-ceramic matrix composites (Almarahle, 2005; Aveston, 1971; Bansal, 1997; Ben *et al.*, 1964; Boccaccini *et al.*, 1996; Brennan and Prewo, 1982; Cho *et al.*, 2009b; Crivelli-Visconti and Cooper, 1969; Gadkaree, 1994; Mahler *et al.*, 1971; Manivasakan *et al.*, 2010; Prewo and Brennan, 1980; Rafukka *et al.*, 2013; Roether and Boccaccini, 2005; Sambell *et al.*, 1972; Sung, 1999; Sung and Park, 2000; Sung and Sung, 1997; Vaidya *et al.*, 1992).

Due to their exceptional energy dissipation ability; damping, physical and mechanical properties, carbon nanotubes (CNTs) are very attractive for many application and technologies (Baughman *et al.*, 2002; Koratkar *et al.*, 2005; Salvetat *et al.*, 1999a; Salvetat *et al.*, 1999b; Salvetat *et al.*, 1999c; Suhr *et al.*, 2005; Suhr and Koratkar, 2008; Terrones, 2003; Treacy *et al.*, 1996; Walters *et al.*, 1999; Yu *et al.*, 2000a; Yu *et al.*, 2000b).

In a comprehensive report; book chapter on carbon nanotube reinforced glass and glass-ceramic matrix nanocomposites (Subhani *et al.*, 2013), where ten years earlier studies on CNT reinforced glass/glass-ceramic matrix nanocomposites were reviewed, the following findings were observed: Densified glass matrix nanocomposites with homogeneously dispersed CNTs up to 15 wt. % were realized; moderate enhancement in fracture strength, toughness, thermal conductivity, and remarkable increases in electrical conductivity have been conclusively obtained with randomly dispersed CNTs in glass/glass-ceramic matrices, whereas the effects on hardness and stiffness is not yet understood clearly; Aligned CNTs in one or more defined direction, responsible for better structural and functional properties is yet to be obtained; CNT reinforced glass/glass-ceramic

matrices synthesised by In-situ have not yet been produce; and conclusively, improved functional, mechanical, and technological properties; wear and friction resistance, thermal shock, cycling and ageing resistance signified the potential application of CNT-glass/glass-ceramic matrix nanocomposites in many industries for low and moderate temperature environments. To the best of my knowledge, up to the early 2018, there has been no study that incorporates the CNTs in the matrix of powdered quartzite (natural or synthetic) and fabricated by either cold processing or high-temperature sintering process, and attempted to determine its low, moderate or high-temperature characteristics. Until late 2018 and early 2019 when the novel studies were made to manufacture and determined the mechanical and physical characteristics of multiwalled carbon nanotubes reinforced quartz (natural crystalline silica) nanocomposite (Tijjani *et al.*, 2019a, 2019b; Tijjani *et al.*, 2018). Hence, the present review provides an up to date literature on quartz, glass, and glass-ceramic matrix nanocomposites; containing carbon nanotubes

Production and Characterization of CNT-Ceramic, Glass/Glass-Ceramic Matrix Nanocomposites

For the fabrication of CNT-based ceramic, glass, and glass-ceramic nanocomposites of better structural, mechanical and functional properties, six main processing issues are essential: 1) Good tube quality and less impurity; high crystallinity, high aspect ratios, little structural defects, 2) Achieving a homogeneously dispersed nanotubes within the ceramic and hindering of CNTs' natural affinity to agglomerate with highly detrimental effects on material integrity/performance 3) Obtaining appropriate interfacial bonding between the inorganic matrix and the nano-filler to promote toughening via development of energy dissipation mechanisms 4) Establishment of high consolidation levels during the sintering stage 5) Restriction of the destruction of CNT during densification by pressureless sintering, hot-pressing (HPS) and hot-isostatic pressing (HIP) and finally, 6) Consideration of the type of ceramic manufacturing and processing routes (Cho *et al.*, 2009a; Cho *et al.*, 2011; Curtin and Sheldon, 2004; Dassios, 2014; Dassios *et al.*, 2015; Thostenson *et al.*, 2001).

The main fabrication techniques for CNT-ceramic, glass/glass-ceramic nanocomposites are:

1) Conventional powder processing (CNT-ceramic nanocomposite): Powder processing involves the dispersion of CNT in a polar liquid e.g. distilled water/ethanol (Ahmad *et al.*, 2010; Mo *et al.*, 2005; Ning *et al.*, 2003) to obtain CNT/water or CNT/ethanol suspension, the "wet" mixing with ceramic powder, followed by ultra-sonication and/ or ball milling, then drying, crushing and sieving, and finally hot/cold compaction, and consolidation/densification to obtain sintered nanocomposite pellet (Arsecularatne and Zhang, 2007; Boccaccini *et al.*, 2005; Cho *et al.*, 2009a; Subhani *et al.*, 2013).

2) In-situ growth of CNT by chemical vapour deposition (CNT-ceramic-metal nanocomposite): This entails the synthesis of CNT in a CVD reactor over the dispersion of transition metal catalyst nanoparticles; for nucleation and CNTs growth, supported on the ceramic powders destined to form the matrix (Arsecularatne and Zhang, 2007; Cho *et al.*, 2009a; Subhani *et al.*, 2013).

3) Colloidal processing (ceramic coated/reinforced CNT): Colloidal processing is heterocoagulation of the colloidal suspensions of CNTs and similar size ceramic nanoparticles to encourage the ceramic particles to coat the CNTs or vice versa (depending on their respective sizes) to address the issue of CNTs agglomeration (Arsecularatne and Zhang, 2007; Cho *et al.*, 2009a; Subhani *et al.*, 2013). Another colloidal processing techniques applied for the production of coatings and films from CNTs and nanoparticles are Electrophoretic and Electrolytic depositions (EDP/ELD) (Boccaccini and Zhitomirsky, 2002). Surface activation or functionalization of CNTs usually with mineral acids is one of the important factors prior to EPD (Boccaccini *et al.*, 2010).

4) Sol-gel processing: In Sol-gel technique, the CNTs are dispersed in a molecular precursor solution which undergoes a condensation reaction to generate a green body for subsequent consolidation (Cho *et al.*, 2009a).

Whereas, consolidation or densification of the nanocomposites via high-temperature sintering is achieved by hot pressing (HP) (Boccaccini *et al.*, 2005; Braley and Belk, 2018), hot-isostatic pressing (HIP), pressureless sintering (PS) (Bocanegra-Bernal *et al.*, 2017), and spark plasma sintering (SPS) (Guo *et al.*, 2007; Shin *et al.*, 2018).

Thus, the present review would be based on the processing, mechanical, and functional properties of CNT-quartz, glass and glass-ceramic matrix nanocomposites (excluding the CNT reinforced biocompatible and polymer-derived glass/glass-ceramics) for the last nineteen years to date. Tables 1, 2 & 3 summarized respectively, all the related matrix/CNTs, comparison of various processing techniques, and mechanical and functional properties of the nanocomposites (based on Table 1).

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Table 1: Quartz, glass and glass-ceramics containing CNTs

Matrix	**CNTs: Types/loading**	**Processing route**	**Characterizations**	**References**
SiO_2-glass	MWNTs-6 wt.%	Sol-gel/pressureless sintering	Hardness enhanced (~100%) using Vicker's indentation	(Hwang and Hwang, 2001)
SiO_2-glass	MWNTs	Colloidal processing/Sol-gel	Microstructural	(Seeger *et al.*, 2001)
SiO_2-glass	MWNTs	Sol-gel/hot pressing	Microstructural	(Seeger *et al.*, 2002)
SiO_2-glass	SWCNTs-0.1 – 1 wt.%	Sol-gel	Optical	(Dimaio *et al.*, 2003)
SiO_2-glass	MWNTs	Sol-gel	Microstructural	(Grobert *et al.*, 2003)
SiO_2-glass	MWNTs-5 Vol.%	Powder/hot pressing	Bending strength 85 MPa (65%), fracture toughness 2 $MPa.m^{1/2}$ (100%) (Vickers)	(Ning *et al.*, 2003)
SiO_2-glass	MWNTs	Sol-gel/laser treatment	Microstructural	(Seeger *et al.*, 2003)
SiO_2-glass	MWNTs-5 Vol.%	Sol-gel/hot pressing	Bending strength 97 MPa (88%), fracture toughness 2.46 $MPa.m^{1/2}$ (146%) (Vickers)	(Ning *et al.*, 2004)
SiO_2-glass	MWNTs-10 wt.%	Powder/pressureless sintering.Powder/hot pressing	Density up to 1.98g/cm^3 and porosity as low as 11%	(Boccaccini *et al.*, 2005)
SiO_2-glass	MWNTs	Sol-gel/dip coating	Microstructural	(Berguiga *et al.*, 2006)
Barium aluminosilicate glass-ceramic (BAS)	MWNTs-10 wt.%	Powder/hot pressing	Flexural strength 245 MPa (192%), fracture toughness 2.97 $MPa.m^{1/2}$ (143%) (SENB)	(Ye *et al.*, 2006)
SiO_2-glass	SWNTs	Sol-gel	Compressive failure stress 6 MPa (33%), toughness 0.32 $MPa.m^{1/2}$ (53%)	(Zhang *et al.*, 2006)
Borosilicate glass	MWNTs-10 wt.%	Powder/hot pressing	Hardness 5.8 GPa (-22%), elastic modulus 54 MPa (-14%), fracture strength 63 MPa (-41%), fracture toughness 0.72 MPa.m1/2 (-10%), electric resistivity 13 ohms-cm (monolithic glass- 10^5 ohms-cm).	(Boccaccini *et al.*, 2007)
SiO_2-glass	MWNTs	Electrophoretic deposition	Microstructural	(Chicatun *et al.*, 2007)
SiO_2-glass	SWNTs-4 wt.%	Sol-gel/dip coating	Vicker's hardness 600 HV_{100} (28%), microstructural	(Jung De Andrade *et al.*, 2007)
Mullite	MWNTs-5 wt.%	Powder/hot pressing	Bending strength 512 MPa (10%), fracture toughness 3.60 $MPa.m^{1/2}$ (78%) (SENB)	(Wang *et al.*, 2007)
SiO_2-glass	SWNTs-0.025, 0.050 & 0.075 wt.%	Sol-gel/high pressure technique	Toughness 1.05 $MPa.m^{1/2}$ (69%) for 0.050 wt. % SWNT, Vickers indentation 532 HV (44%) for 0.075 wt.% SWNT	(De Andrade *et al.*, 2008)
SiO_2-glass	MWNTs	Colloidal	Microstructural	(Kim *et al.*, 2008)
SiO_2-glass	MWNTs	Sol-gel	Microstructural	(Chan *et al.*, 2008)
Borosilicate glass	MWNTs-0 - 3 wt.%	Sol-gel	Compressive strength 445 MPa (77%), thermal	(Thomas *et al.*, 2009)

			conductivity 1.55 Wmk^{-1}, hardness 7.9 GPa (13%) (Vickers), Young modulus 42 GPa (2.4%), electrical conductivity (2.2 x 10^{-4} S/m).	
Glass-ceramic	MWNTs	Powder/pressureless sintering	Electrical resistivity at 1000°C; reduce to 29.61 ohms-cm from 6.39 x 10^{12} ohms-cm, and to 7.46 ohms-cm from 4.86 x 10^{12} ohms-cm for monolithic V2.0 & B-glass-ceramics.	(Giovanardi *et al.*, 2010)
SiO_2-glass	MWNTs-0.1 wt.%	Sol-gel/mechanical mixing	Vickers hardness 0.31 GPa (11%), Young modulus 72 GPa (4%), toughness 0.78 $MPa.m^{1/2}$ (24%).	(López *et al.*, 2010a)
SiO_2-glass	MWNTs	Sol-gel/dip coating	Fracture toughness (Vickers) enhanced for the coating on grounded substrates	(López *et al.*, 2010b)
Silica substrate	MWNTs	Sol-gel/dip coating	Resistivity 0.03 ohms-cm, two order < previous reported for CNT/Sol-Gel composite.	(Erismis *et al.*, 2011)
SiO_2-glass	MWNTs-0.1 wt.%	Sol-gel/dip coating & mechanical mixing	Wear resistance reduces with the addition of CNTs	(López *et al.*, 2011)
Lead silicate-glass	SWNTs-1.5 wt.%	Melt-quench	Hardness (Vickers) 31% enhancement at 1N load, thermal conductivity increased by 42 % at 683 K, toughness by Vickers 0.62 $MPa.m^{1/2}$ (27 %) & SEVNB 0.91 $MPa.m^{1/2}$ (27 %).	(Ghosh *et al.*, 2012)
SiO_2 xerogel films	MWNTs	Sol-gel	Elastic modulus 2.127 GPa (>800%), hardness 0.035 GPa (250%)	(Jung *et al.*, 2012)
SiO_2-glass	MWNTs-1 wt.%	Colloidal/pressureless sintering	Microstructural	(Arvanitelis *et al.*, 2013)
Lead silicate-glass	SWNTs-0.68 vol.%	Melt-quench	Electric conductivity enhanced by 10^5 and increase with temperature between 308 & 438 °K	(Ghosh *et al.*, 2014a)
Lead silicate-glass	SWNTs-0.5, 1.2 &1.5 wt.%	Melt-quench	True hardness using Vickers indentation and applying M-PSR model 4.28 GPa (22 %)	(Ghosh *et al.*, 2014b)
Borosilicate glass	MWNTs-0 – 1.5 wt.%	High shear compaction/spark plasma sintering	Young and shear modulus for 0.5 wt.% sintered at 650°C are 65.7 (5 %) and 28.5 (10 %) GPa.	(Dassios *et al.*, 2015)
Alumino borosilicate glass	MWNTs-20 vol.%	Sol-gel/hot pressing	Elastic modulus 102 GPa (32 %), bending strength 1.4 GPa (8 %)	(Otieno *et al.*, 2015)
SiC/zinz aluminium silicate	MWNTs-3 wt.%	Electrophoretic deposition	Shear strength 33.7 MPa (94.8 %) for EPD time 40 s. Then after thermal shock process at 800°C & 1000°C to RT for 40 times, the shear strength were 20.5 MPa (24.2 %) & 19.4 MPa (21.3 %).	(Feng *et al.*, 2016)

Glass fiber textures (GTs)	MWNTs-0.1, 0.25 & 0.5 g/L	Electrophoretic deposition	Volume conductivity improved by 10^8 x, interlaminar shear strength (ILSS) enhanced by 42 % of simple (GTs) specimens in 0.25 g/l CNT concentration for 3 minutes.	(Haghbin *et al.*, 2017)
Quartz (natural crystalline silica)	MWNTs-0.01 – 4 wt.%	Powder/pressureless sintering	For 0.01 wt.% MWNTs/quartz nanocomposite; bulk density 1.760 g/cm^3 (1.3 %), porosity 30.37 (-7 %), tensile strength 4.27 MPa (44 %), Young's modulus 579 MPa (88 %) compressive strength 63.70 MPa (63 %) strain tolerance (tensile) 0.0076 (-17 %) and for 1 wt.% MWNTs/quartz; moderate mechanical strengths and strain tolerance (tensile) 0.013027 (41 %).	(Tijjani *et al.*, 2018)
Quartz	MWNTs- 0.01–4 wt.%	Powder/pressureless sintering	Foundry physical properties; diametric expansion 1.57 (-2 %), linear expansion 1.37 (-40 %), bulk density 1.735 (-0.2 %), apparent porosity (27 %), cold crushing strength 177 kg/cm^2 (-56 %), and thermal shock resistance 7 cycles (75 %) for 1 wt.% MWNTs/quartz nanocomposite.	(Tijjani *et al.*, 2019b)

The percentage given in the curly bracket stands for the percentage of the property enhancement of the CNT-loaded nanocomposite as compared to the unreinforced plain material.

Table 2: Comparison of the various processing techniques (based on Table 1)

Technique	**Extent of CNT dispersion**	**Common consolidation technique used**	**Possible damage to CNTs**	**Density after sintering**	**Frequency**
Powder	low	Hot pressing	high	low	6
In-situ	-	-	-	-	0
Colloidal/Electrophoretic deposition (EPD)	high	-	low	-	6
Sol-gel	high	Hot pressing	low	high	18
Melt-quench	high	-	low	high	3
High compaction sintering (HCS)	high	Spark plasma sintering	low	high	1

Properties not determined (-)

Table 3: Comparison of the various mechanical and functional properties (based on Table 1)

Technique	Extent of mechanical properties enhancement						Extent of functional properties enhancement			
	Hd	**Rel**	**Frt**	**Rel**	**Strength**	**Rel**	**Therm**	**Rel**	**Elect**	**Rel**
Powder	-	-	high	√	high	√	-	-	high	√
In-situ	-	-	-	-	-	-	-	-	-	-
Colloidal/EPD	-	-	-	-	high	√	-	-	high	√
Sol-gel	low	×	high	×	high	√	-	-	high	√
Melt-quench	low	×	low	×	-	-	low	√	high	√
HCS	-	-	-	-	-	-	-	-	-	-

Low stands for the percentage of improvement < 50 %, whereas, high signifies > 50 % enhancement. √- Reliable, ×- Unreliable methods & (-) properties not determined

Key: Hd = hardness, Rel = Reliability, Frt = Fracture toughness, St = Strength, Therm = Thermal conductivity, Elect = Electrical conductivity

An Overview of the Processing, Mechanical, and Functional Properties

Reference to Tables 1, 2 and 3, the following conclusions could be made on the various processing routes, mechanical, and functional properties of the CNT-loaded quartz, glass/glass-ceramic nanocomposites considered:

1) Nanocomposites processed by powder processing possess the least dispersion and are more susceptible to CNT-damage than those produced by colloidal/EPD, sol-gel, low-temperature melt-quench and high compaction sintering techniques.

2) Notwithstanding, there is more enhancement in mechanical properties in powder processed nanocomposites via more reliable methods, for instance, single edge V-notch beam (SEVNB) than those reported in sol-gelled and melt-quenched nanocomposites.

3) There is no study on fabrication and determination of physical, mechanical and functional properties of CNT-loaded quartz, glass and glass-ceramic nanocomposites by In-situ growth of CNT by chemical vapour deposition on the quartz, glass and glass-ceramic powders.

4) There may be some future prospects in the fabrication and determination of physical, mechanical and functional properties of quartz, glass and glass-ceramic containing carbon tubes via the novel high compaction sintering technique.

5) Also, hardness and thermal conductivity should be determined for powder, sol-gel and colloidal processed nanocomposites pellets and coatings.

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