UNIVERSITI PUTRA MALAYSIA

MECHANICAL AND THERMAL PROPERTIES OF CNT-REINFORCED QUARTZITE NANO-COMPOSITE FOR FURNACE LINING

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FK 2018 179

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By

YUSUF TIJJANI

Thesis Submitted To the School of Graduate Studies, Universiti Putra Malaysia, in Fulfilment of the Requirements for the Degree of Doctor of Philosophy

June 2018

DEDICATION

This thesis is dedicated to the following important personalities:
My beloved parents;
My loved wife and son
My dear brothers and sisters;
And my fallen heroes/friends: Engr. Bashir Yahya and Engr. Sani Dahiru who passed away in the course of study in Malaysia (May your souls rest in perfect peace (Aljannatul Firdaus), Ameen).

Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfilment of the requirement for the degree of Doctor of Philosophy

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Chairman: Faizah Bint MD Yasin, PhD
Faculty: Engineering

Nigerian local foundries employ the used-imported bricks from iron and steel industries to line the foundry cupola furnace. On the event of the closure of these industries, the alternative left was to identify the locally available refractory material that would be used for most effective lining of the cupola. Thus, after several trials of different locally available clays, natural crystalline quartzite refractory ceramic material; crystalline silica (SiO_2) was eventually resorted to. The main problem associated with ceramic lining material is lack of good thermal shock resistance. This is more pronounced with silica/silica-based refractory linings; for instance, lining material used in a typical local foundry furnace in Kano State of Nigeria and vicinity that is prepared from local quartzite/crystalline silica, which develops unduly cracks on its surface during cooling after melting. This has been attributed to poor thermal conductivity by the previous researchers. To substantially obviate this setback, the present research aimed to incorporate carbon nanotubes (CNTs) within the matrix of quartzite brick. This entails manufacturing and characterization of CNT reinforced quartzite nano-composite. To obtain a stable suspension of CNTs that would be used in the preparation of homogeneously dispersed nanotubes in the quartzite matrix, the as-received pristine CNTs were functionalized with 6M H_2SO_4 acid by ultrasonication using a water bath sonicator. The pristine/functionalized CNTs were then comprehensively characterized by Fourier-transform infrared spectroscopy (FT-IR), Raman spectroscopy, Thermogravimetric analysis (TGA), Differential scanning calorimetry (DSC), and Scanning electron microscopy coupled with Energy dispersive X-ray spectroscopy (SEM/EDX). Also, the as-mined quartzite and clay were purified by soaking/washing with water and subsequently dried. The as-dried raw material were then subjected to crushing, grinding, pulverizing and grading using Jaw and Cone crushers, ball mill, pulverizer, and size distribution particle analyzer respectively. This is followed by structural and morphological characterization of the powdered quartzite and clay by Energy dispersive X-ray fluorescence spectrometer (ED-XRF), X-ray diffraction (XRD), and SEM/EDX. The durable refractory material for lining foundry cupola is developed by modified conventional powder processing (wet mixing method); involving preparation, cold compaction (by manual hydraulic press) and pressureless sintering (using tube furnace) at 1450°C, for 2 hours dwelling time, under argon atmosphere. Physical, mechanical, morphological and thermal characterizations were determined for the sintered 0, 0.01, 1, and 4 wt. % CNT-quartzite nano-composite blends. The comparable bulk density range obtained for the blends; 1.722 – 1.760 g/cm^3 is an evidence of a complete densification and good homogeneous dispersion of CNTs in the green matrices, thus, good thermo-mechanical properties of the nano-composites. High proportion of tridymite, low percentage of residual quartz (1.1 %), low reversible thermal expansion, modest tensile (2.49 MPa) and compressive strengths (17.39 MPa), moderate Young's modulus; tensile (190 MPa), high fracture strain in tension (0.013027), high (on-duty) thermal shock cycles (7 cycles), high thermal diffusivity $(> 8 \times 10^{-7}\, m^2/s\ at\ 117°\mathrm{C})$ and remarkable thermal conductivity $(> 0.2\, k/Wm^{-1}K^{-1} at\ 117°\mathrm{C})$ of 1 wt. % CNT-quartzite nano-composite signifies the possibility of potential application of CNTs as sintering aid (stabilizer, mineralizer, etc.) and toughening filler in a conventional quartzite/silica refractory mixture for high durability. Finally, the Levenberg Marquardt Back Propagation Artificial Neural Network (LMBP ANN) models were developed to predict the physical, mechanical and thermal properties of the CNT-quartzite nano-composites having formulations within the range of those employed in the experimental process through dataset training, validation and testing. Additionally, the Graphical User Interface (GUI) was created in the study in order to have a user friendly interface for easy characterization of the CNT-quartzite nano-composites.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Doktor Falsafah

CIRI-CIRI MEKANIKAL DAN TERMA NANO KOMPOSIT KUARZIT BERPENGUAT CNT UNTUK LAPISAN RELAU

Oleh

YUSUF TIJJANI

Jun 2018

Pengerusi: Faizah Bint MD Yasin, PhD
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Kilang-kilang tempatan Nigeria menggunakan batu bata terpakai yang diimport dari industri besi dan keluli untuk menyambung relau leburan kupola. Sekiranya berlaku penutupan industri ini, alternatif yang masih ada adalah dengan mengenal pasti bahan refraktori yang ada di dalamnya yang boleh digunakan untuk lapisan kupola yang lebih berkesan. Oleh itu, selepas beberapa percubaan mengunakan tanah liat tempatan berbeza yang sedia ada, bahan kuarzit kristal seramik refraktori semulajadi; silika kristal (SiO_2) dapat digunapakai. Masalah utama berkaitan dengan bahan lapisan seramik adalah kekurangan rintangan kejutan haba yang baik. Hal ini lebih ketara dengan lapisan refraktori yang menggunakan bahan berasaskan silika. Sebagai contoh, bahan lapisan yang digunakan di sebuah kilang relau tempatan di daerah Kano dan kawasan berdekatannya di Nigeria telah disediakan dari bahan kuarzit tempatan berhablur, telah menyebabkan keretakan yang terlalu ketara pada permukaan semasa penyejukan selepas peleburan. Hal ini telah dikenalpasti sebagai kesan daripada konduksi haba yang lemah oleh penyelidik-penyelidik terdahulu. Bagi mengatasi masalah ini secara mendalam, penyelidikan yang kini dijalankan adalah bertujuan untuk menggabungkan tiub nano karbon (CNTs) dalam matriks kuarzit bata. Ini melibatkan pembuatan dan pencirian nanokomposit kuarzit berpenguat CNT. Untuk mendapatkan penggantungan CNT yang stabil untuk digunakan dalam penyediaan tiub nano yang terserak secara menyeluruh dalam matriks kuarzit, bahan tulen yang digunakan telah difungsikan dengan asid 6M H_2SO_4 melalui kaedah ultrasonikasi menggunakan sonikator rendaman air. CNT yang tulen / difungsikan kemudian dicirikan secara komprehensif oleh Spektroskopi Inframerah Transformasi Fourier (FTIR), Spektroskopi Raman, Terma Analisis Gravimetrik (TGA), Kalorimeter Imbasan Pembeza (DSC), Mikroskop Pengimbas Elektron dan Tenaga Penyerakan Sinar-X (SEM / EDX). Juga, kuarzit seperti yang dilombong dan tanah liat dibersihkan dengan merendam / membasuh dengan air dan kemudiannya dikeringkan. Bahan mentah kering kemudiannya dihancurkan, dikisarkan, dipecahkan dan diperingkatkan dengan menggunakan penghancur Rahang dan Kon, pengisar bebola, pengisar, dan penganalisis partikel pengagihan saiz. Ini diikuti dengan ciri-ciri struktur dan morfologi serbuk kuarzit dan tanah liat oleh analisis spektrometer serakan tenaga sinar X (ED-XRF), X-ray difraksi sinar X dan SEM / EDX. Bahan refraktori yang tahan lama untuk lapisan kupola leburan dihasilkan melalui pemprosesan serbuk konvensional yang diubahsuai (kaedah pencampuran basah); di mana ia melibatkan penyediaan, pemadatan sejuk (dengan mesin hidraulik manual) dan pensinteran tanpa tekanan (menggunakan relau tiub) pada 1450^{o}C di bawah atmosfera argon selama 2 jam. Ciri-ciri fizikal, mekanikal, morfologi dan terma ditentukan untuk 0, 0.01, 1.0, dan 4.0 % berat CNTs, campuran batu bata kuarzit. Julat kepekatan pukal setanding yang diperolehi untuk campuran; 1.722 - 1.760 g / cm^3 adalah bukti penyepadatan yang lengkap dan penyebaran menyeluruh yang baik oleh FMWNTs dalam matriks hijau, oleh itu, sifat-sifat terma mekanikal yang baik dari nano komposit. Peratusan tinggi tridimit, peratusan rendah sisa kuarza (1.1%), pengembangan haba boleh ubah yang rendah, tegangan sederhana (2.49 MPa) dan kekuatan mampatan (17.39 MPa), modulus Young sederhana; tegangan (190 MPa), ketegangan fraktur yang tinggi dalam ketegangan (0.013027), kitaran kejutan haba yang tinggi (atas tugas) (7 kitaran), kelimpahan haba yang tinggi ($> 8 \times 10^{-7}\ m^2/s\ at\ 117$℃) dan kekonduksian haba yang luar biasa ($> 0.2\ k/Wm^{-1}K^{-1}at\ 117$℃) 1 wt. % nano komposit kuarzit CNT menunjukkan kebarangkalian CNT berpotensi untuk digunakan sebagai bantuan sintering (penstabil, pemineral, dan lain-lain) dan menguatkan pengisi dalam campuran konvensional kuarzit / refraktori silika untuk ketahanan yang tinggi. Akhir sekali, model Rangkaian Saraf Tiruan Levenberg Marquardt telah dihasilkan untuk meramalkan ciri-ciri fizikal, mekanikal dan terma nano komposit kuarzit CNT dan berada dalam formulasi mengikut julat yang digunakan dalam proses eksperimen melalui latihan dataset, pengesahan dan ujian. Di samping itu, Antara Muka Grafik Pengguna (GUI) telah dicipta dalam kajian ini untuk mendapatkan antara muka yang mesra pengguna bagi memudahkan pencirian nano komposit kuarzit CNT.

ACKNOWLEDGEMENTS

All praise is due to Allah, may Allah's Peace and Blessings be upon His Final Messenger, his pure family, his noble Companions, and all those who follow them with righteousness until the Day of Judgment. We thank Him for the gift of life, guidance, protection, blessings, favours, insight, and wisdom.

Firstly, I would like to express my sincere gratitude to my supervisor Dr Faizah Bint Mohd Yasin for her continuous support of my PhD study, high level of patience, tolerances, motivation, and immense knowledge. Her guidance helped me in all the time of research and writing of this thesis.

My heartfelt thanks go to my supervisory committee: Ass. Prof. Dr Mohd Halim Shah bin Ismail and Ass. Prof. Dr Azmah Hanim bt Mohamed Ariff, for their insightful comments, encouragement, and hard questions which in-centred me to widen my research from various perspectives. I could not have imagined having a better committee and mentors for my PhD research.

My sincere thanks also go to Ass. Prof. Dr Khamirul Amin Matori, and Dr Shuhazlly both of Physics Department UPM, who gave me access to their laboratories and research facilities. Without their precious support, it would not be possible to conduct this research.

I thank all laboratory technicians in engineering and science especially Mr Ismail Abdul Ghani, Mr Ismail Abdullah, Mr Termizi Zakaria, etc, my lab-mates such as Fidaus, Shahera, Afifah, and my friends for the stimulating discussions, fun, timely and tireless assistance.

Last but not the least; I would like to thank my family: my parents, relatives, my wife, and my son for their prayers throughout my stay in Malaysia and my life in general. I also thank Prof. Fakharul-Razi Ahmadun for introducing me to the basic of nanotechnology. Thus, “Every test is a blessing from Allah and every blessing is a test from Allah.” Thank you all…

This thesis was submitted to the Senate of University Putra Malaysia and has been accepted as fulfilment of the requirement for the degree of Doctor of Philosophy. The members of the Supervisory Committee were as follows:

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LIST OF ABBREVIATIONS

CNT(s)	Carbon Nanotube(s)
MWNT(s)	Multi-Walled Carbon Nanotube(s)
PMWNT(s)	Pristine Multi-Walled Carbon Nanotube(s)
FMWNT(s)	Functionalized Multi-Walled Carbon Nanotube(s)
SWNT(s)	Single-Walled Carbon Nanotube(s)
FTIR	Fourier Transform Infrared Spectroscopy
ED-XRF	Energy Dispersive X-Ray Fluorescence Spectrometer
SEM	Scanning Electron Microscopy
FESEM	Field Emission Scanning Electron Microscopy
EDX	Energy Dispersive X-ray Spectroscopy
TGA	Thermogravimetric Analysis
DTG	Derivative Thermogravimetric
DSC	Differential Scanning Calorimetry
LFA	Laser Flash Analysis
UTM	Universal Testing Machine
CCS	Cold Crushing Strengths
Q^I	Quartzite + Sintering aids
ITMA	Institute of Advanced Technology
MCL	Material Characterisation Laboratory
NMDC	National Metallurgical Development Centre

CHAPTER 1

INTRODUCTION

1.1 Background to research

Foundry process or casting involves melting metals, pouring into the mould, and extraction of the product after the metal solidified as it cools. The place where these operations are carried out and the product outcomes are referred to as foundry and a cast respectively. The history of casting process was documented circa 1500 by Vannoccio Biringuccio who is popularly known as 'father of the foundry'(Candeo, 2012), whereas evidence from Nigerian foundry historic relics was dated approximately 900 AD (Childs and Killick, 1993). Depending on the type of end users, foundry process can be either commercial foundry; production activity on a large scale and casting achieved by sand casting, die casting, investment casting and lost foam casting, or hobby foundry; small but fully equipped setup inside a house or in the backyard. Also, commercial foundries could be captive foundry; part of a manufacturing company which utilizes the cast items to meet the company production demand, independent foundry; casting produced to suit customer's specifications, jobbing foundry; carry out contractual casting for other companies, production foundry; particular types of casting are made in bulk quantity, and semi-production foundry; this is the combination of jobbing and production foundry.

Captive, jobbing and semi-production, are the common foundries in Nigeria, examples are Nigerian Foundries Limited (NFL), Grand Foundry and Engineering Works Limited, Castmaster Metallurgical Company Limited, Integrated Foundry which is the captive foundry of Nigeria Machine Tools Limited, etc. Foundry being one of the cheapest manufacturing processes is a primary basis for all technological take-off and industrialization. Components ranging from that of machine tools, power plants, ship-building, railways, agriculture and food processing, cement and textile industries to household utensils such as metal bowls, pots, spoons, etc. are realized through casting (Ibitoye and Ilori, 1998). In fact, it is impossible to think of any machinery or equipment which has no components from the foundry.

Sand casting is by far the oldest and the most commonly used casting process. It comprises putting a pattern (possessing the shape of the desired casting) in sand to obtain an imprint, integrating a gating system, isolating the pattern and pouring the molten metal into the prepared mould cavity, allowing the metal to cool as it solidifies, breaking away the sand mould, and removing the casting (Iqbal *et al.*, 2014; Kalpakjian and Steven, 2014; Sulaiman and Hamouda, 2001, 2004). Problems associated with foundries in Nigeria can be broadly categorised as: lack of training of foundry personals such as foundry craftsmen, technicians, and engineers; In adequate investment in casting technology development to ensure profit at least on the long run; Lack of sustainable government policies towards foundry such as strategies setup to better the foundry industries; Lack of developing casting raw materials, mismanagement, and over/under utilization of iron scraps, pig-iron, bentonite, silica sand, binder, refractories, alternatives, etc.; and lack of research and development, foundry being one of the heavy basic industries, an outstanding studies are required in air pollution and industrial waste control, safe working environment, raw materials, maintenance, and finished product (Jimoh *et al.*, 2013).

1.2 Refractory materials

Refractories are materials that withstand high temperatures (usually above 1100°C), resist the action of corrosive liquids, and dust-laden currents of hot gases. They are used in lining furnaces, kilns, incinerators, reactors, and to make crucibles. The refractory materials (depending on whether it is for insulation or conduction) must possess high refractoriness, high resistance to abrasion and erosion, high resistance to thermal fatigue, low/moderate porosity and permeability, low/high thermal conductivity, low coefficient of thermal expansion, and low cost per unit weight of the material treated (Chesti, 1986).

Based on the composition of oxides present in the refractory; silica (SiO_2), alumina (Al_2O_3) and silica, magnesia (MgO), chromite ($FeCr_2O_4$), and magnesia and chromite, it may be chemically classified as acidic, basic or neutral refractory. Acid refractories; are based on silica and consist of SiO_2, sillimanite, fireclays with 30-42% Al_2O_3, and andalusite containing approximately 60% Al_2O_3. Acid refractories are neutral to acid slag but react readily with basic slags. Basic refractories; are based on magnesia (MgO) and comprise magnesite and dolomite, chrome magnesite, magnesite, chrome, alumina, mullite, and many special refractories such as ThO_2 and BeO. The special refractories are mostly new, very expensive, reserved for research purposes, atomic energy and gas turbine technologies, etc. They include materials like ZrO_2 and BeO and can be classified as acid, basic or neutral. Special refractories with a melting point above approximately 1900°C are referred to as super refractories. Basic refractories are inert to basic slags but are readily attacked by acid slags. Neutral refractory; these are relatively inert to both siliceous and limy slags. They include carbon, chromite ($FeO.Cr_2O_3$) and forsterite ($2MgO.SiO_2$) bricks (Arthur *et al.*, 1977). As far as refractory is concerned, there is no such things as best properties, rather, a compromise is necessary when it comes to the selection of optimum performance criteria based on rigidity, size and shape stability, strength at operating temperature, ability to withstand thermal shock, resistance to chemical attack; by gas, slag or metal.

The silica employed for the fabrication of silica refractories exists in the form of quartz; quartz is the only type of silica that is stable at atmospheric temperature, therefore, all the un-combined silica in the earth's crust exists as quartz and is present in quartzite, canister, sand, flints, etc. Quartzite which contents the low-quartz modification (α-quartz) is the most used form of quartz employed for the production of silica refractories in most countries. This is achieved via conventional coarse ceramic manufacturing that involves preparation, shaping, drying and firing (Brunk, 2000). The silica brick could be dense (≥93% SiO_2); used in the construction of coke oven, glass furnace and hot blast stoves, lightweight (≥91% SiO_2, true porosity >45% and up to 72%); employed primarily in thermal insulation of glass melting tank crowns, shaped fused or vitreous; used as a material for hot repairs in the glass industry, and unshaped silica product (>90% SiO_2; precisely between 95 to 98%); which find application in induction furnaces for melting copper or steel (Brunk, 2001). Silica refractories are also used in the construction of roofs of open hearth furnace and in the exposed sidewalls due to their high melting point, rigidity, and strength at elevated temperature (Almarahle, 2005; Alnawafleh, 2009; Chesters, 1983). Almarahle (2005) and Alnawafleh (2009) have been reported to have successfully fabricated silica bricks from white quartz sand mixed with lime.

The high-quality pure silica fused at 1715ºC (3119ºF). As much as possible, the silica should be pure with merely about 1-2% alumina, titania and other impurities as their presence in greater percentages largely reduce its refractoriness (Chesti, 1986). The commercially available silica brick was reported to have failed at 1649ºC (3000ºF), and by restricting the number of oxides in a noble silica refractory invented, the refractoriness was increased by 38ºC (100ºF) (Harvey and Birch, 1944). Silica bricks are relatively resistant to attack by iron oxide and alkalis. To avoid iron oxide pick-up (for pure silica brick), Sosman recommended the minimum safe operating temperature to 1650ºC (3002ºF) (Chesters, 1983).

Foundry Cupola Furnace is a melting device used in foundries to melt cast iron, Ni-resist iron, and some bronzes. Most of the conventional cupolas are operated in temperature range between 1482ºC and 1510ºC (2700ºF to 2750ºF) (Carter, 1953), whereas the melting temperatures of commercial cast iron, austenitic nickel iron (low expansion type), copper, and bronzes (with tin content 20%) are 1135ºC to 1150ºC (2075 to 2102°F), 1230ºC (2246°F), 1084ºC (1983ºF), and 910ºC (1670ºF) respectively (Angus, 2013; Dainian and Cohen, 1996).

The cupola is mostly lined with fire clay or similar heat resistance material (Klug, 1915). Therefore, in my view, the application of the silica refractory ceramic could be extended to the lining of foundry cupola save for its low thermal shock fracture resistance. Fracture mechanics is the scientific study of the behavior of the progressive crack extension in structures. Using its primary tools; characteristic material values, test procedures, and failure analysis procedures (various fracture criteria) it controls the brittle fracture and fatigue failures in structures. Prior to the advent of the theory of fracture mechanics Kingery (1955), Hasselman (1963a), Hasselman (1963b), and Hasselman (1969) were some researchers that study the adverse effects of fracture by thermal shock in refractory ceramics (Baker *et al.*, 2006). With the development of modern (refined) fracture mechanics various researchers (Bahr *et al.*, 1987; Bahr *et al.*, 1986; Bahr and Weiss, 1986; Bahr *et al.*, 1988; Baker *et al.*, 2006; Dey *et al.*, 1995; Dudczig *et al.*, 2012; Evans and Charles, 1977; Geyer and Nemat-Nasser, 1982; Gruber *et al.*, 2016; Hou *et al.*, 2013; Kašiarová *et al.*, 2014; Lu and Fleck, 1998; Lutz and Swain, 1991; Marshall and Swain, 1988; Mcnaney *et al.*, 1999; Nemat-Nasser *et al.*, 1978; Nemat-Nasser *et al.*, 1980; Ribeiro *et al.*, 2016; Ritchie, 1988; Schneider and Petzow, 2013; Soboyejo *et al.*, 2001; Steinbrech *et al.*, 1983; Swain, 1990) applied its concepts to investigate and/or model and control the behavior of thermal shock in monolithic and/or fibre/nano particle reinforced refractory ceramics.

Thermal shock is a failure of the material, especially a brittle material when subjected/exposed to a sudden transient thermal condition (a rapid change in temperature) (Soboyejo *et al.*, 2001; Tokovyy and Hetnarski, 2014; Wang and Singh, 2013). Also, thermal shock is observed in a material due to the unequal expansion (stress or strain) of its parts as a result of the differential thermal gradient. In some instance, this stress could exceed the strength of the material, which eventually gives rise to a crack formation. Structural failure in the material may then arise if there is no impediment or obstacle to the crack propagation (Wang *et al.*, 2013). The ability of a material to withstand the thermal shock is referred to as thermal shock resistance (Carter and Norton, 2007; Tite *et al.*, 2001). Thermal shock fracture resistance depends on/and increase with high values of tensile strength σ_f, thermal conductivity k, thermal diffusivity K, and fracture toughness K_{IC}; and decrease with low values of coefficient of thermal expansion α, Young's modulus of elasticity E, Poisson's ratio ϑ, and emissivity ε, (Hasselman, 1963a; Lu and Fleck, 1998).

The facilities for periodic usage; blast oxygen furnace, casting ladles, degasser, heat exchangers, gas turbine engines, catalyst support, and solid oxide fuel cell and those of continuous casting; ladle shrouds, slide plates, and submerged nozzles are more prone to thermal shock than installations of continuous process such as blast furnace and electric arc furnace (Atkinson *et al.*, 2004; Colombo, 2002; Soboyejo *et al.*, 2001; Tokovyy and Hetnarski, 2014; Vedula *et al.*, 1998). Unfortunately, to date, there is no simple and unified standard test method for the determination and comparison of thermal shock resistance (TSR) of ceramic materials that would at the same time simulate the actual conditions in service, thermal cycles, and sample geometry. However, sample testing involving transient heating and cooling could be carried out but this is only reliable for comparative analysis of similar materials (Li *et al.*, 2014; Rendtorff and Aglietti, 2010). Semi silica brick with about 88-93% SiO_2 produced from natural or artificial sand-clay mixtures is cheaper, tougher, more volume stable than fire clay brick, possess higher thermal shock resistance than silica brick and is prompt to lesser slag attack than fire and silica bricks (Chesters, 1983).

In the guess to provide better toughening material for ceramics, variety of reinforcing fibers have been explored, these comprise; boron, SiC, α-alumina, mullite, and carbon fibres (Bunsell and Berger, 2000; Dicarlo, 1985; Phillips, 1974). However, carbon fibres (CFs) are among the highest performance toughening elements studied, and in the context of matrix reinforcement in composites, carbon nanotubes (CNTs) are usually considered to be the next generation of carbon fibres (Cho *et al.*, 2009a). Due to their unique arrangement of its carbon atoms, carbon nanotubes (CNTs) possess a remarkable electrical, thermal, and mechanical properties (Kaushik and Majumder, 2015; Li *et al.*, 2009). These properties offer the CNTs wide range of applications and studies in the field of nanomaterials that are unique and could not be achieved by either fiber or whisker reinforcement (Calvert, 1992; Castillo-Rodríguez *et al.*, 2016; Castillo-Rodríguez *et al.*, 2015; Estili and Sakka, 2014; Nishida, 2013; Ruoff and Lorents, 1995; Suraya *et al.*, 2009; Treacy *et al.*, 1996; Wang, 2017; Xiong *et al.*, 2015; Xu and Gao, 2015). Apart from mechanical and physical effects, the carbon nanotube as a second phase reinforcement may benefit other functional properties viz. thermal expansion coefficient (Cho *et al.*, 2016; Hamamda *et al.*, 2017; Pal and Sharma, 2014; Van Trinh *et al.*, 2018; Wei *et al.*, 2002), thermal shock resistance (Feng *et al.*, 2014; Fu *et al.*, 2015a; Fu *et al.*, 2015b; Roungos and Aneziris, 2012; Sasthiryar *et al.*, 2014; Sribalaji *et al.*, 2018), etc. To this extent, the present research would apply the functionalized carbon nanotubes within quartzite (silica)/CaO-clay refractory ceramic matrix so as to reduce the high-temperature gradient responsible for the uneven expansion of the silica, cracks initiation, and the low thermal shock resistance.

1.3 Problem statement

Most of the Nigerian local foundries used a prepared sand mixture called "plastic ramming mixture" (containing mainly high percentage of alumina [Al_2O_3] and silica [SiO_2] with the very small percentage of impurities in the form of iron oxides and alkalis) to line the foundry cupola furnace before and after each melt. Consequently, the downtime in production could be up to eight (8) hours; spent for furnace preparation; re-lining the refractory materials before the subsequent melts. This could be attributed to the surface cracks/thermal shock of the ceramic lining material (commonly made locally from quartzite and clay). These cracks have been identified by the previous researchers such as Baker *et al.* (2006) to occur due to the sudden temperature change in pouring metal into a ceramic-lined crucible or rapid cooling of the cupola furnace to ambient temperature. They demonstrated an enhancement of the thermal shock resistance circle in the monolithic aluminosilicate material with the addition of 4 wt. % Na_2O by promoting viscoelastic toughening. Also, in an attempt to address the above-mentioned problem in our local foundries, Rafukka (2009) was able to characterize the various blends used in a typical foundry in Kano State, Nigeria and recommended the blend of used-imported brick plus Gezawa clay as the best out of the three possible blends based on the highest refractoriness and thermal shock resistance. The other two blends were made from as-mined locally available materials of Burji clay and Gezawa stone (quartzite); to which Gezawa clay was added to each. In the event of the closure of local iron and steel industries which are the major consumers of the imported bricks, then the local foundries that depend mostly on the used-bricks from these industries were left with two options; either Burji clay with Gezawa clay or quartzite with Gezawa clay. It has been established that the quartzite blended with Gezawa clay possess higher refractoriness as compared to Burji clay blended with Gezawa clay but the former has lower thermal shock resistance (Rafukka *et al.*, 2013a, 2013b). Thus, quartzite could be employed advantageously for lining foundry cupola furnace as compared to Burji clay save for its low thermal shock cycle.

With the aim to improve the physical, mechanical and functional properties of local refractory materials, to reduce the production downtime, and also to save refractory lining replacement costs, the present study would review the local Gezawa stone (quartz/crystalline silica stone) (Rafukka *et al.*, 2013a) and Gezawa white clay (clay). Precisely, the CNTs would be added to conventional quartzite to improve its thermo-mechanical properties.

1.4 Objectives

The aim of this study is to determine the mechanical and thermal properties of CNT-reinforced quartzite nano-composite for furnace lining; this entails the following objects:

1. To investigate the microstructural properties of as-received/functionalized carbon nanotubes (CNTs), as-mined quartzite and clay.
2. To determine physical, mechanical, and thermal properties of the novel fabricated CNT-quartzite nano-composites.
3. To apply the Levenberg Marquardt Back Propagation Algorithm (LMBP) for training Artificial Neural Network (ANN) models that would be used to characterize/predict the physical, mechanical and thermal properties of the CNT-quartzite nano-composites possessing formulations within the range of those trials used in the experimental datasets.

1.5 Scope and limitation of research

The scope of the present research includes the surface activation of as-received pristine carbon nanotubes, characterization of the pristine/activated CNTs, preparation and characterization (chemical, morphological, and determination of particle size distribution) of as-mined quartzite and clay, introduction of functionalized (activated) carbon nanotubes as reinforcement into the natural crystalline silica (quartzite)/CaO-clay matrix, and the fabrication of CNT-quartzite nano-composite test specimen pellets. This is followed by the determination of foundry physical properties of the samples; linear and diametrical expansion, apparent porosity, bulk density, cold crushing strength, thermal shock resistance, microstructure; crystallite size/shapes, and surface morphology. Then mechanical properties specifically elastic modulus (tensile), tensile strength, compressive strengths, fracture strain in tension and compression (strain tolerance) have been obtained. Also, thermal conductivity is determined by an indirect method using the values of bulk density; obtained from Archimedes' principle, specific heat capacity; determined by the equation and using differential scanning calorimetry (DSC) heat flow curves, and thermal diffusivity; evaluated by the laser flash apparatus. Lastly, the Levenberg Marquardt Back Propagation Artificial Neural Network (LMBP ANN) models have been developed based on the limited dataset's range of the experimental work to characterize the nano-composites for physical, mechanical and thermal properties.

The limitations of this study are:

- The specific heat capacity and the thermal conductivity of the CNT-quartzite nano-composites were obtained at low temperatures.
- The LMBP ANN developed models are only applicable to the present study and would be reliable only for the prediction of the thermo-mechanical properties of the CNT-quartzite nano-composites within the lower and upper boundaries of the experimental datasets.

1.6 Justification

There have been rapid declines in the number of industries that manufacture silica/silica-based refractories in United Kingdom (UK) in particular and precisely world in general. In fact, it has been reported that as per back as 1960, the steel industries utilized about three-quarters of all the silica bricks manufactured in the UK, the other main users being glass and carbonizing industries. In 1969 the steel industries consumption reduced to about one-third, and the carbonizing industries to about one-quarter of the 1960 amount. The only high utilization reported is in glass industries. The drop could be explained as a result of change from open-hearth to basic oxygen furnace (BOF) and arc steelmaking (as the open-hearth is one biggest consumer of silica brick), the replacement of silica gas retorts by natural gas, and changeover from silica to high-alumina bricks in the roofs of electric-arc furnace to decrease the downtime in plants that depend for their survival on high operating rates. This led to the closure of many silica brick industries and a significant reduction in research work in the high silica area (Chesters, 1983).

In light of these, the present research would try to address downtime issue, suggest additional areas in which the silica bricks could be used as part of the effort to revive the silica/silica-based industries and further the research works in the field. Another point of pivotal importance is the effort by the present Nigerian government to revolutionize the industrial sector; more especially iron and steel industries. Silica/silica-based material being one of the very important keys in this area, as its application is obvious in coke oven for the production of coke from coal and blast furnace for the smelting of iron ore, this and others justify the present research as timely and along awaited.

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LIST OF PUBLICATIONS

Tijjani, Y., Yasin, F. M., Ismail, M. H. S., and Ariff, A. H. M. (2018). Manufacturing and Mechanical Characterisation of Multiwalled Carbon nanotubes/Quartz Nanocomposite. *Journal of the Ceramic Society of Japan (JCS), 126 (12) 984*-991. *http://doi.org/10.2109/jcersj2.18100.*

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Tijjani, Y., Yasin, F. M., Ismail, M. H. S., and Ariff, A. H. M. (2018). Effect of Carbon nanotubes addition on the foundry physical properties of Silica refractory nanocomposite: PART A, **Presented in** *1st International Postgraduate Conference on Mechanical Engineering (IPCM),* 31st October 2018, Universiti Malaysia, Pahang. (To be published in Scopus indexed proceedings and journals).

Tijjani, Y., Yasin, F. M., Ismail, M. H. S., and Ariff, A. H. M. (2018). Characterization of Carbon nanotube reinforced Silica refractory nanocomposite using Artificial Intelligence Modeling: PART B **Presented in** *1st International Postgraduate Conference on Mechanical Engineering (IPCM),* 31st October 2018, Universiti Malaysia, Pahang. (To be published in Scopus indexed proceedings and journals).

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