

Exploration of nano-composite ceramic materials concept for ballistic application, phase 1: material development and characterization

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Abstract. In order to satisfy future requirements of the Canadian Forces in lightweight ballistic armours for personnel protection, nano-composite ceramic materials concepts is explored. The apparent ability of the nano-particles to deflect cracks represents a major interest for ballistic armour applications. Literature reported that impregnation of nano-particles into various ceramics resulted in either significant improvement or decrease in fracture toughness and hardness. This show the lack of understanding of the relationship between process parameters, microstructure, mechanical properties and the damage mechanisms governing ballistic performance of these ceramics. A Canadian Ceramic Armour Team compose of scientists from several government research organizations in Canada with most of the required technological and testing capabilities has joined their effort to gain this knowledge. This paper presents the 3 years program that began in spring 2010. Preliminary results, mostly from the NRC's Steacie Institute for Molecular Sciences, show the successful impregnation of single walled carbon nanotubes (SWCNT) additives into the ceramic matrix using the proprietary surface modification process.

1. INTRODUCTION

Ceramics, with their high strength to mass density ratio are attractive materials for high performance, low weight armour system. Because of their hardness, they defeat the projectile by erosion or fracture [1]. However, their intrinsic brittleness and low fracture toughness conduct to brittle fracture and shattering, which severely affect their multi-hit capabilities. Also, voids and discontinuities induce during manufacturing provide sources for crack nucleation and propagation which limit their potential performance [2-4].

Reinforcing ceramics with Carbon Nanotubes (CNT) have a great potential to improve the ballistic properties due to the formation of an effective 3D network structure with one dimensional CNT having high aspect ratio. This 3D network is expected to reside within the boundaries of the ceramic grains restraining crack propagation. Also, the very low loadings of CNT can have a significant enhancement of hardness and fracture toughness [5, 6]. To maximize the mechanical performance of the ceramic composite and minimize the mass loading of CNT, the Canadian Ceramic Armour Team believe that single wall carbon nanotubes (SWCNT) would be a better choice than multi wall carbon nanotubes (MWCNT) due to their higher aspect ratios, smaller diameters (1-2 nm as individual SWCNT and 1-100 nm as bundles) and effective mass usage/contribution to the mechanical performance. Therefore, the focus of this work is on SWCNT as filler for the fabrication of lightweight and multifunctional ceramic composites.

This research program is a collaborative effort between DRDC¹ Valcartier who leads the project and evaluates the ballistic performances of the novel ceramic composite materials, the NRC²-IAR³ who is the main technical coordinator and, with NRCAN⁴, develops the new ceramics concept and NRC-SIMS⁵ who develops the impregnated single walled carbon nanotubes additives. NRC-SIMS has extensive expertise and facilities for both, bench mark production for laboratories concept as well as large quantity production for industrial demand.

¹ DRDC: Defence Research and Development Canada

² NRC: National Research Council of Canada

³ IAR: Institute for Aerospace Research

⁴ NRCAN: Natural Resources Canada

⁵ SIMS: Steacie Institute for Molecular Sciences

2. PROGRAM OVERVIEW

Considering ceramic armour is one of the main burdens carried by soldiers, developing solutions to decrease their weight without compromising their ballistic performance, or conversely improve the protective properties without increasing the weight is crucial. To reach that goal, it is of prime importance to understand the correlation between the process parameters, the properties of the developed ceramics and their influence on the failure mechanisms. Therefore, our program on nano-composite ceramic materials concepts began in spring 2010 and is planned to span three years.

During the first year, the main focus is on the development of basic competencies with respect to processing and testing of both monolithic and nano-composite Al_2O_3 -, SiC-, and B_4C -based ceramics. The various processing approaches considered are hot pressing (HP), hot isostatic pressing (HIP), cold isostatic pressing (CIP) and pressureless sintering (PS). The low cost reaction bonding (RB) technology could also be used, but considering the low performance ceramic typically produced, this technology is not included in this research. The nano-reinforcement investigate include nanopowders such as TiN, TiC, TiO_2 and single walled carbon nanotubes and their derivatives such as modified carbon and boron nitride nanotubes (m-CNT and BNNT). The synthesis, purification and functionalization of SWCNT and their integrations into alumina matrix are completed and will be described in the next section. Laboratory ceramic specimen (50mm diameter) made with various nano-reinforcements and using the different manufacturing process are mechanically tested and compared for their density, hardness, flexural strength, and fracture toughness using appropriate ASTM standard [7-10].

The second year concentrates on the search of optimum powder mixture compositions and process parameters. The nanotubes integration investigation is pursuing for SiC and B_4C -based ceramics. The ultimate goal of these efforts is to improve mechanical properties of the synthesized monolithic and nano-ceramics (i.e., hardness, flexure strength, and fracture toughness). It is expected to initiate ballistic depth of penetration (DoP) tests with larger specimen (100mm diameter) to confirm the screening and optimization from mechanical properties evaluation.

The third year is aimed at evaluating ballistic performance of the best pre-selected candidates under different ballistic loads, analysis of fracture mechanisms, establishing correlation between mechanical and ballistic properties of fabricated ceramics and implementing process adjustments for further improvement of the achieved ballistic performance.

3. SWCNT INTEGRATION IN Al_2O_3

The challenges in making a ceramic composite are to have a uniform dispersion, distribution and a good interface between SWCNT and the ceramic matrix. Due to the inherent inert surface and strong van der Waals interactions, SWCNT have little affinity for, or compatibility with, any matrices including organic, polymeric and inorganic matrix and tend to stay in ropes or bundles. Therefore, surface modifications of SWCNT, either through covalent or non-covalent functionalization, become essential to de-bundle (exfoliate) the SWCNT and to have a favorable dispersion throughout the ceramic matrix. Moreover, coating a layer of in-situ Al_2O_3 onto these functionalized SWCNT facilitate the interface with the industrial Al_2O_3 matrix through the favorable -O-Al(in-situ)-O-Al(industrial)-O-bonding . Various approaches of functionalizing, coating and integrating of SWCNT into the industrial Al_2O_3 matrix, as well as the processing conditions and the preliminary mechanical testing results are briefly described in the following section.

4. STARTING MATERIALS AND COMPOSITE MATERIAL PREPARATIONS:

Alumina (Al_2O_3) ceramic powder was purchased from Accumet Materials Inc., with medium grain size of 160~200 μm . Aluminum chloride and ammonia hydroxide were purchased from Aldrich and used as received. Polyvinyl alcohol was purchase from DuPont.

4.1. SWCNT synthesis, purification and surface modifications:

Single walled carbon nanotubes (SWCNT) used in this research was synthesized using double lasers ablation process developed in-house by NRC-SIMS described in previous papers [11, 12]. The typical scanning electron microscope (SEM) morphology and Raman spectrum of the raw SWCNT material are shown in Figure 1a and b. The wet chemical purification procedure (WCPP) has been applied for purifying SWCNT raw materials. The SEM image of purified SWCNT is shown in Fig. 1c. The WCPP purification protocol has been developed in-house by NRC-SIMS and is briefly described in previous papers [13, 14]. Raman spectroscopy allows quick and reliable characterization of the quality and purity of SWCNT. The radial breathing mode (RBM) observed in the low wavenumber region of the spectrum is quite specific to SWCNT as its wavenumber is inversely proportional to the diameter of SWCNT. The disorder band (D-band) lying near 1350 cm^{-1} is indicative of sp^3 hybridization which occurs as a result of defects on graphitic structures. When a covalent chemistry takes place, the intensity of the D-band increases. The graphitic band (G-band) lying near 1550 cm^{-1} is indicative of graphitic structures (sp^2 hybridization). For SWCNT, because of the curvature, the double degeneracy is lifted and the band appears a doublet structure. The G/D intensity ratio is often used as a quality factor. The band lying near 2600 cm^{-1} is labelled G' and is the overtone of the D-band. The wavenumber of this band is very sensitive to stress and pressure and hence is often used to gauge stress transfer in SWCNT-composite materials.

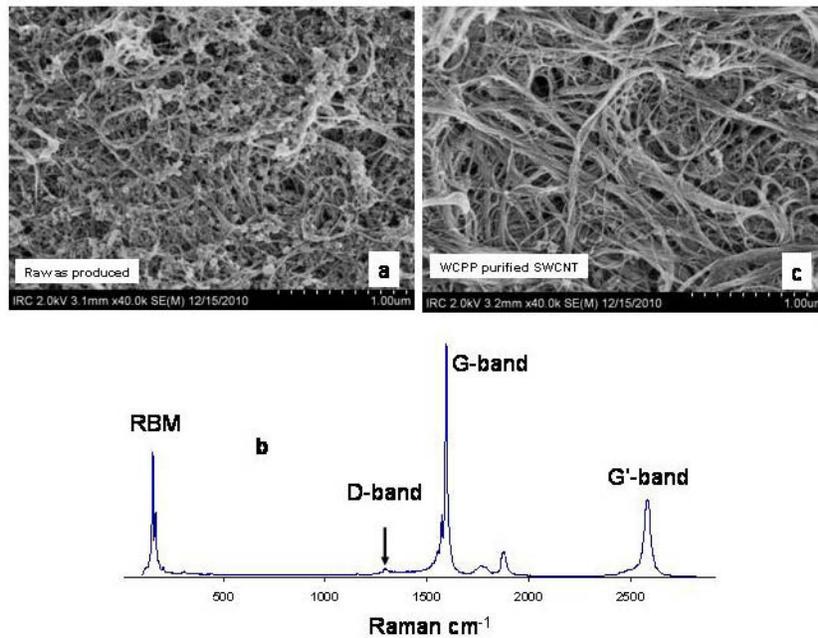


Figure 1. SEM image of SWCNT raw (a) and purified (c), and typical Raman spectrum of SWCNT raw (b)

4.1.1 Covalent functionalization of SWCNT and coating with a layer of in-situ Al_2O_3 :

In order to have a good dispersion and favorable interface between carbon nanotubes and alumina ceramic matrix, SWCNT have been covalently functionalized with hydroxyl (OH) groups by Ozonolysis, followed by coating with a layer of in-situ Al_2O_3 (Figure 2).

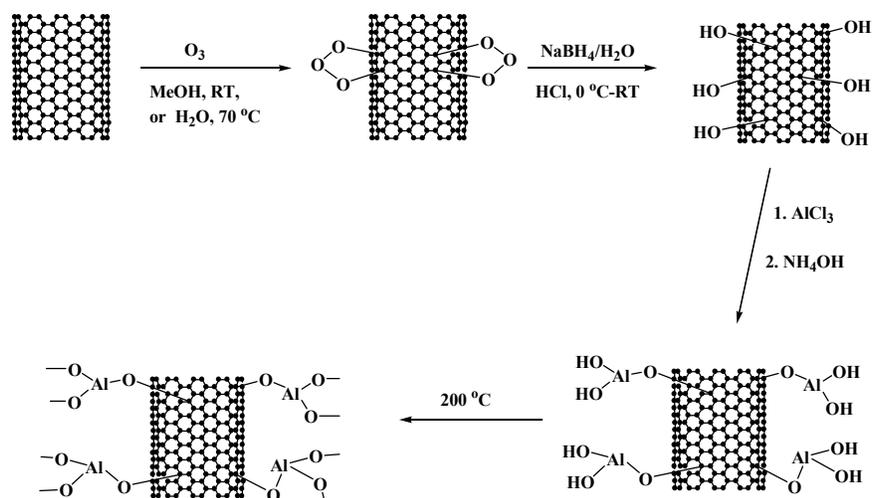


Figure 2. Scheme for the ozonolysis of SWCNTs and the coating of a layer of in-situ Al_2O_3

A typical synthetic procedure for covalent functionalization of SWCNT with OH groups: the Ozonolysis was conducted by two methods and both produced good results: 1) using MeOH as a solvent and the reaction was done at room temperature (RT); 2) using distilled water as a solvent and heated the reaction to 70°C. Typically, SWCNT were suspended in MeOH and bath-sonicated for 1.5 h. The resulted suspension underwent ozonolysis at RT for 8 h with sufficient bath sonication for each 1 hour run. The reaction mixture was then purged with oxygen for 1 h at RT. NaBH_4 was added portionwise at 0°C within 15 min, and the mixture was stirred at room temperature overnight. 3N HCl was then added dropwise at 0°C within 30 min, and the reaction mixture was warmed to RT and stirred for an additional hour. The crude product was diluted in distilled water (DW), centrifuged and decanted. The collected product was dried in the oven at 110°C overnight and characterized.

Figure 3 shows a typical Raman spectrum of OH-functionalized SWCNT, in which the D-band (disordered sp^2 to sp^3) has obvious increased comparing with the pristine SWCNT.

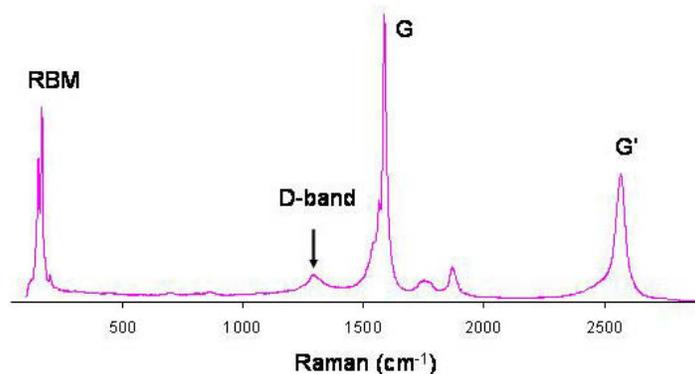


Figure 3. Raman spectrum of SWCNT-OHs, D-band has obvious increased comparing with pristine SWCNT.

A typical synthetic procedure for coating the SWCNT-OHs with an in-situ layer of Al_2O_3 [30 wt% SWCNT-O- Al_2O_3 (in-situ)]: SWCNT-OH was suspended in DW and tip-sonicated for 2 h, and an aqueous solution of AlCl_3 was added. The mixture was bath-sonicated for 3 h and tip-sonicated for 1.5 h. To the above homogeneously dispersed mixture was added ammonium hydroxide solution dropwise with vigorous stirring until pH to 9.5. Tip-sonication with vigorous stirring was applied for an additional 30 min. The resulted mixture was diluted in DW and centrifuged, and the process was repeated twice. The final precipitate was collected and dried at 200°C overnight. 10 wt % and 20 wt% of SWCNT-O- Al_2O_3 (in-situ) samples have also been prepared in the similar fashion.

4.1.2 Non-covalent functionalization SWCNT and coating with a layer of in-situ Al_2O_3 :

Synthesis of 30 wt% SWCNT/PVA/ Al_2O_3 (in-situ): Pristine SWCNT were suspended in DW and PVA was added. The mixture was processed in the same manner as the above procedure with SWCNT-O- Al_2O_3 (in-situ).

As shown in Figure 4 below, both covalently or non-covalently functionalized SWCNT showed a uniform coating of in-situ generated alumina through C-O-Al-O bonding on the side walls of SWCNT.

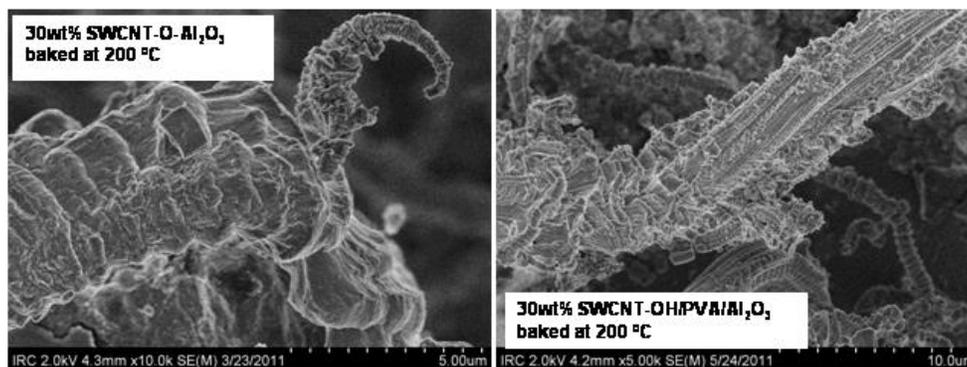


Figure 4. Typical SEM images of in-situ generated alumina coating on the side wall of SWCNT.

4.1.3 Incorporation of functionalized, Al_2O_3 (in-situ)-coated SWCNT into the industrial Al_2O_3 matrix:

During the syntheses of the functionalized, Al_2O_3 coated SWCNT, the in-situ formed $Al(OH)_3$ was converted into alumina Al_2O_3 ceramic by baking at 200-250°C in air, this is to create identical interface on the surface of SWCNT with the industrial alumina ceramic matrix. However, after the above coated SWCNT was integrated into the industrial Al_2O_3 , baking at 200°C has partially decomposed the original binder and decreased the quality of the green compact. Therefore, the following sample preparations have been slightly modified to avoid the loss of the original binder, and the composites were dried at room temperature. The conversion of $Al(OH)_3$ to Al_2O_3 will take place during the first stage of the sintering.

A typical procedure for the integration of the above functionalized, Al_2O_3 (in-situ)-coated SWCNT into industrial Al_2O_3 ceramic matrix: The above wet precipitate of 30% SWCNT-O- $Al(OH)_3$ (in-situ) was dispersed in DW and mixed with a suspension of pre-sonicated aqueous industrial Al_2O_3 . The mixture was tip-sonicated with vigorous stirring until a homogeneous dispersion was achieved. The grey mixture was centrifuged and washed with DW, repeated twice, and dried at room temperature.

The SEM images in Figure 5 demonstrated that the as synthesized SWCNT- $Al(OH)_3$ have good dispersion/distribution in the industrial alumina matrix for both covalently and non-covalently surface modified SWCNT samples.

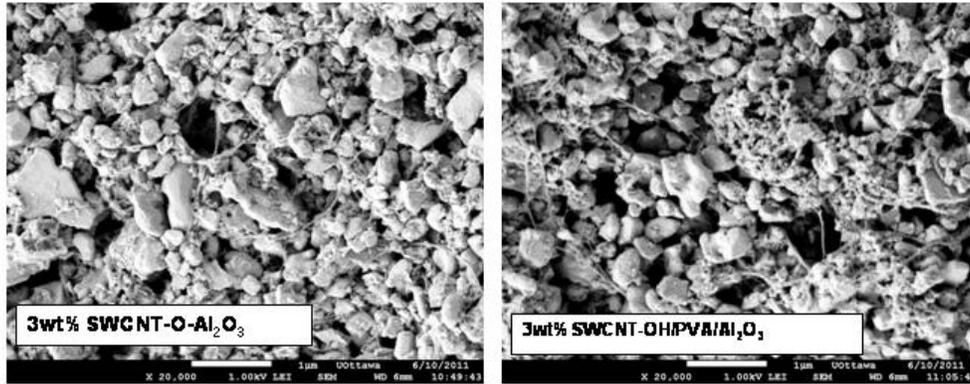


Figure 5. SEM images of surface modified SWCNT diluted into commercial alumina matrix and dried at room temperature.

Table 1 describes the SWCNT/Al₂O₃ and baseline materials that have been synthesized and characterized, as well as the mechanical testing methods.

Table 1. The SWCNT/Al₂O₃ and baseline materials and the mechanical testing methods

Products	Comments	Processing method	Sintering method
36g, 1.5wt% SWCNT-O-Al ₂ O ₃ (in-situ+industrial)	Covalent functionalization with coating.	Solution processing	PS
36g, 1.5wt% SWCNT(Pristine)/PVA/Al ₂ O ₃ (in-situ+industrial)	Non-covalent functionalization with coating.	Solution processing	PS
40g, 3wt% SWCNT-O-Al ₂ O ₃ (in-situ+industrial)	Covalent functionalization with coating.	Solution processing	PS
40g, 3wt% SWCNT(Pristine)/PVA/Al ₂ O ₃ (in-situ+industrial)	Non-covalent functionalization with coating.	Solution processing	PS
36g, 3wt% SWCNT(Pristine)/Al ₂ O ₃ (industrial).	No functionalization and no coating.	Solution processing	PS
40g, 0wt%, Al ₂ O ₃ (in-situ)/Al ₂ O ₃ (industrial)	Baseline sample, no nanotubes.	Solution processing	PS
72g, Processed industrial Al ₂ O ₃ (mortar/pestle)	Baseline sample, no nanotubes.	Solution processing	PS
20g, 3wt% SWCNT(Pristine)/Al ₂ O ₃ (industrial)	No functionalization and no coating.	Processed by coffee grinder	PS and HP (to be done)
500g, 1wt% SWCNT-O-Al ₂ O ₃ (in-situ+industrial)	Covalent functionalization with coating.	Solution processing	PS and HP (to be done)
500g, 1wt% SWCNT-O-Al ₂ O ₃ (in-situ+industrial)	Covalent functionalization with coating.	Ball milling	PS and HP (to be done)

5. MATERIAL PROCESSING AND CHARACTERIZATIONS

Monolithic Al_2O_3 , similar to other ceramic armour materials, can be produced by a variety of manufacturing processes including hot pressing (HP), pressureless sintering (PS), hot isostatic pressing (HIP), and reaction bonding (RB) [15-18]. Each of these methods has its advantages and shortcomings with respect to the manufacturing cost, the shape of the parts, and the degree of structural densification that strongly affect the ballistic performance. For example, HIP typically results in fully dense ceramic parts with complex shapes; however, this is one of the most expensive processes. HP is less expensive and the most commonly used; however, it is limited to simple nearly plane body armour shapes. PS and most RB processes are very economical, however, most sintered ceramics are not as well consolidated as hot-pressed ones and this may affect ballistic performance. RB can result in fully densified ceramics through the use of reactive additives; however, these additives may negatively affect hardness and fracture toughness of the ceramic material.

At the present stage of the project, hot pressing (HP) and pressureless sintering (PS) were used to obtain ceramic coupons for initial evaluation. The processing and characterization was performed in two steps; first from the as received ceramic powder (without nanotubes) and then for the SWCNT-reinforced ceramics.

5.1 Green disc preparation:

To form a green compact out of alumina powder, the apply pressure needed is in the range of 140 MPa. Two processing methods were considered to do that: a cold isostatic pressing (CIP) and a hydraulic arbour press with a capacity of $\sim 27,000$ kg. Since both methods produced similar results, the latter one was adopted for further processing. The use of the arbour press restricts the size of the specimen to 50 mm (2") diameter in order to achieve the desired load. A two part mould was made of stainless steel along with a backing plate and an extraction screw, shown in Figure 6a and 6b.



Figure 6a. Bottom portion of the mould.



Figure 6b. Top part of the mould.

The powder was placed in the bottom part of the mould on top of the backing plate, the top part of the mould slides within the bottom part of the mould and contacts the powder. After compaction, the green compact was removed from the mould using the extraction screw pushing on the backing plate. The thickness of the green compact was ~ 4 mm.

5.2 Pressureless sintering and hot pressing:

Green disks were sintered at temperatures up to 1750°C using different furnaces and different sintering profiles. The sintering cycles were done either in vacuum of 7×10^{-5} millibar or helium environment to protect CNT from oxidation.

Hot Pressing was carried out using 50 ton press with temperature capability up to 1600°C and coupon size up to 4 inch (100 mm). A typical processing time was 30 minutes.

After processing, all coupons were evaluated for dimensional changes, density, hardness and fracture toughness. Preliminary evaluations of the physical properties of the coupons were carried out

to assess effectiveness of the manufacturing process and to establish optimum processing technique for required mechanical properties.

5.3 Density test:

To determine the relative density of the coupons sintered in house, the buoyancy method was used. Before the measurements, samples were submerged in distilled water for 24 hours to help reduce water absorption in the samples. However, it was found that buoyancy method might be not appropriate for some materials, as water could seep into surface connected voids and give an artificially higher density measurement. Thus, alternative method, based on coupon dimension measurement to determine volume was also used. Example of test results for pure alumina is shown in Table 2 and 3.

Table 2. Typical physical and mechanical properties of Hot Pressed Al₂O₃ samples

	Density, g/cm ³	Density, %	HV _{10kgf} , kg/mm ²	HV, GPa	Fracture Toughness, MPa·√m
1	3.776	95.4	1319	13.51	4.4
2	3.917	98.9	1564	15.3	3.2

Table 3. Typical physical and mechanical properties of Pressureless Sintered Al₂O₃ samples

	Density, g/cm ³	Density, %	HV _{10kgf} , kg/mm ²	HV, GPa	Fracture Toughness, MPa·√m
1	3.8	95.9	1271	12.5	3.4

Table 2 and 3 show that, in general HP produce harder and more fracture toughness resistant ceramics. Also, Table 2 shows that higher hardness results in lower fracture toughness that may affect ballistic performance of the materials. Initial test results on SWCNT-reinforced alumina produced density values strongly dependent on CNT loads, with lower densities for higher CNT loads (up to 3 wt%). The reason for this behavior is explained in Section 5.5.

5.4 Hardness and Fracture toughness:

Rockwell superficial (HR 15N) hardness tests were carried out to compare the effects of processing temperature and time on the samples. These values were for comparison purposes only, while the actual hardness of these samples was obtained by Vickers indentation method as per the ASTM C1327 – 08 Standard Test Method for Vickers Indentation Hardness of Advanced Ceramics [8]. Preliminary tests have shown that for indentation loads above 15 N (1.5 kgf) results were independent on load value.

Cracks generated by Vickers indentation, as detected by SEM examination, were used to determine the fracture toughness of the ceramic materials [19]. Examples of range of hardness and fracture toughness results for pure alumina coupons can be found in Table 2 and 3. Table 4 presents comparison of hardness and fracture toughness results for pure and CNT-reinforced alumina (1 wt% SWCNT/Al₂O₃) produced by hot pressing at 1500°C and pressure of 4000 psi (28 MPa).

Table 4. Mechanical properties of Hot Pressed Al₂O₃ and 1 wt% SWCNT/Al₂O₃ samples

	Sample	HV _{1kgf} , GPa	HV _{10kgf} , GPa	Fracture Toughness, MPa·√m
1	Pure alumina	15.57	15.04	3.41
2	CNT-reinforced	-	15.67	3.42

The results in Table 4 show 4% improvement in hardness, with practically unchanged fracture toughness for the CNT-reinforced alumina. The process optimization and further testing will continue.

5.5 SEM examination:

SEM examination of SWCNT-reinforced alumina was performed to confirm whether CNT remained in the microstructure after high temperature processing. A 1.5 wt% SWCNT/Al₂O₃ coupon sintered at 1650°C was sectioned, polished and examined under high magnification (up to 40,000). The SEM micrograph of this coupon is shown in Figure 7.

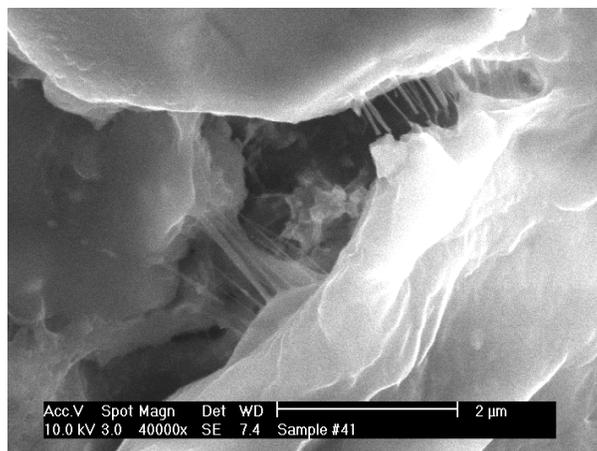


Figure 7. SEM image of 1.5 wt% SWCNT/Al₂O₃ after sintering at 1650°C.

The picture confirms very clearly the presence of CNT in the microstructure and bridging effect of carbon fibers, thus validating the functionalization process developed at SIMS. At the same time, the image in Figure 7 shows that coating is not fully dense (voids) and it is suspected that high CNT contents prevented full densification (by spring-like action) that also resulted in low hardness. Work is in progress to optimize PS and HP processes to produce SWCNT-reinforced alumina ceramics and evaluate role of manufacturing method on ceramic properties.

6. CONCLUSION/REMARK

At the mid term of a 3 years research program to explore the potential of nano additives to ceramic armour, integration of SWCNT in Al₂O₃ ceramic is successful, but work remain to be done to optimized the densification, increase hardness and obtain the full potential of the ceramic nanotube composite.

Other process such as HIP will be investigated and properties compare to find the best technique to make SWCNT-reinforced ceramic.

During the current year, SWCNT will be integrated in other ceramic base (SiC, B₄C) material to evaluate their potential. We expect also to initiate ballistic test.

At this point of the program, work progress as plan and results are encouraging.

Acknowledgements

The great thanks are to the SWCNT production team in SIMS-MaNA group Dr. Chris Kingston, Dr. KuenSu Kim and Mr. Dean Ruth, without their dedication to produce the SWCNT, it is not possible to conduct this research. Thanks are also to Mr. Gordon Chan at NRC-IRC for taking SEM images of the powders and Dr. Qi Yang from NRC-IAR for SEM images of sintered SWCNT- reinforced alumina.

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