



Synthesis and study of the effect of Shock waves on nano composites

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Abstract

Vanadium Carbide-Carbon Shells nanomaterial was synthesized using hydrothermal method with the help of several steps and also, nano alpha-aluminium oxide was synthesized using solid combustion synthesis process. Preparation of aluminium oxide/vanadium carbide-carbon shells composite was done through as aluminium oxide reinforced with 5 wt. % vanadium carbide-carbon shells. A platelet like structure was observed for vanadium carbide-carbon shell from the SEM images. The XRD graph of α -aluminium oxide was shown the sharp peak which indicates that the nano material was crystalline and graph of vanadium carbide-carbon shells showed a semi crystalline nature. The densities of the sintered samples were calculated and the percentage of densification achieved as compared to the theoretical value was obtained. The compressive strength of pure alumina sample achieved was 7.28 MPa. As observed from the graph, it can infer that the sample has very low compressive strength at the outer layer. The compressive strength of vanadium carbide reinforced alumina sample achieved was 12.57 Mpa. This shows a considerable increase in the compressive strength of the alumina matrix composite because of addition of 5 wt% vanadium carbide. It can be concluded that with regard to thermal stability, this material has the capability to maintain the largest possible temperature difference across its faces over a shorter duration without any deformations.

Keywords: Ceramics; Nano materials; Composites; Shockwaves.

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1. Introduction

Ceramics are one of the areas of interest to a materials scientist and are the oldest branch of materials science. A ceramic is a non-metallic solid made up of either metal or non-metal based compounds that have been heated and cooled. In general, they are hard, corrosion-resistant and brittle. Nowadays, ceramic materials that are not necessarily clay-based have been developed. These advanced ceramics are tough and hard-wearing and are being increasingly used in high-performance applications in engineering and medicine. Such advanced ceramics are referred to as 'Structural Ceramics' in technological terms.

As the name suggests, ceramics which serve as a structural member of devices, often being under mechanical loading, are classified under structural ceramics. These ceramics demonstrate excellent mechanical properties with good thermodynamic stability under demanding conditions such as erosive, corrosive or high-temperature environments.

The strong bond strength in these ceramics allows them to be employed in several applications such as tiles for aerospace vehicles, thermal barrier coatings for gas turbines, tribological applications such as mineral processing units, cutting tools for machining, abrasives for polishing and grinding and others. The fundamental research in this area involves understanding the effect of processing parameters on the microstructure and mechanical properties of ceramic materials, testing the performance of ceramics under working environment such as high thermal and mechanical impact environments created during the impact of Shockwaves onto the materials. Ceramics reinforced with other ceramics possessing contrasting properties, can give significantly improved thermal properties along with the mechanical properties [1].

A shockwave is a type of propagating disturbance that moves faster than the local speed of sound in the medium. Like an ordinary wave, a shockwave carries energy and can propagate through a medium but is characterized by an abrupt, nearly discontinuous, change in pressure, temperature and density of the medium. Generally, shockwaves are generated by heavy explosions, breaking of sound barrier by supersonic aircrafts and due to variation in pressure in hydraulic turbines. As a result of the effect of shockwaves, components in a system experience high thermal and mechanical shocks leading to material failure [2].

Ceramics are a group of materials which possess high thermal stability and significantly high compressive strength.

Taking the characteristics of shockwaves to the advantage of testing the selected ceramic specimen, the behavior of the ceramic is observed at such extreme conditions of thermal shocks and pressure variations [2].

2. Experimental Details

2.1. Synthesis of Nano Vanadium Carbide-Carbon Shells

Nano Vanadium Carbide-Carbon tubes are carbon tubes which are reinforced with Nano-vanadium carbide particles. Generally, synthesis of carbides require very high temperature as their melting points are very high. It is not always feasible to synthesize large quantities of nano-carbides through running high temperature reduction reactions. The synthesis involved three steps, the first two being hydrothermal and the final being a carbo-thermal reduction stage. The materials used in this synthesis were Vanadium Pentoxide (V_2O_5), ethanol (C_2H_5OH) and D-(+)-Glucose ($C_6H_{12}O_6$).

Hydrothermal synthesis can be defined as the method of synthesis of single crystals that depend on the solubility of minerals in hot water under high-pressure. The crystal growth occurs in a steel pressure vessel called autoclave, in which a nutrient is supplied along with the water and the reactant [3].

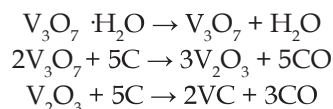
When kept inside an oven, a temperature gradient occurs inside the autoclave. The nutrients dissolve in the hotter side of the autoclave and the crystal formation begins on the cooler side as the seed crystal forms there. The major advantage of hydrothermal method is its ability to grow certain crystalline phases, which are not stable at their melting point. Carbothermal reduction is the process of reducing a compound in the presence of large quantities of carbon and high temperature under inert atmosphere. The inert atmosphere prevents carbon from reacting with atmospheric oxygen. When high temperatures are reached, the compound liberates oxygen which then reacts with carbon present at high temperature. Due to the presence of excess carbon, the broken oxygen bonds are then replaced by carbon. This is one of the easiest methods to synthesize metallic carbides.

The first step involved the synthesis of $V_3O_7 \cdot H_2O$ nano belts. Here, 2.25 g of V_2O_5 was dispersed into 7.5 ml of ethanol and then 100 ml of distilled water was added into a beaker. The solution was placed on a magnetic stirrer and stirred at 600 rpm for 20 minutes. This solution was then placed in a 150 ml Teflon lined autoclave. This was placed in an oven, at 180°C , for 48 hours. The products were then filtered off multiple times with ethanol and distilled water to remove all possible residues. It was then dried in vacuum at 75°C for 1 hour.

In the next step, the $V_3O_7 \cdot H_2O@C$ core-shell composite was prepared. 0.40 g of the obtained product was added into 50 ml of a 0.44 M glucose solution. This solution was then stirred in on a magnetic stirrer at 600 rpm for 1 hour. It was then transferred to Teflon lined stainless steel autoclave, sealed and placed in an oven which was maintained at 180°C for 4 hours. After cooling to room temperature, the products were washed with distilled

water and ethanol several times to remove any residues. It was then dried at 75°C for 1 hour.

To obtain the Vanadium Carbide-Carbon shell, the above obtained product was placed in a tubular furnace with a heating rate of 40°C/min. The furnace was then raised to 1000°C for 2 hours under the flow of argon gas. It was then cooled to room temperature under the continuous flow of argon to prevent oxidation of carbon particles. The reactions which occur in this step are listed below as follows:



2.2. Synthesis of Nano alpha-Aluminium Oxide

Alpha-aluminium oxide or Corundum compounds and their composites have a wide range of applications in various industrial areas as they have high biocompatibility, can form high density ceramics, show very good abrasive and polishing properties, and can be used as a thermal barrier due to their low conductivity. Nano alumina was synthesized using solid combustion synthesis process.

Solid Combustion synthesis is a fast and effective method for synthesizing nanoscale particles. It is a self-propagating high temperature synthesis and sustains itself from highly exothermic reactions. It has been recognized as an alternative to conventional methods for preparing advanced materials including, carbides, borides, nitrides, etc. This high temperature synthesis can also be conducted for gas-gas phases. In addition, integration of combustion synthesis with thermite reactions leads to a method of producing materials with uniformly distributed phases.

The chemicals used were Aluminium Nitrate ($\text{Al}(\text{NO}_3)_3 \cdot 9 \text{H}_2\text{O}$) and Urea ($(\text{NH}_2)_2\text{CO}$). The urea acted as a fuel source and the aluminium nitrate acted as the source of aluminium for the formation of alumina. The reaction was carried out in quartz crucibles in an open muffle furnace at 450°C. Once the crucible was placed inside the furnace, the reaction time was seen to vary between 6-10 minutes. After approximately six minutes of boiling, the liquid was seen to froth and then spontaneously ignite forming a very bright flame [4].

This bright flame continued to burn for 2-3 minutes leaving white solid foam like substance. This foam was then grinded in a pestle and mortar to form a dense powder like substance.

2.3. Preparation of Aluminium Oxide/Vanadium Carbide-Carbon Shells Composite

The final composition of the composite was decided as Aluminium Oxide reinforced with 5 wt. % Vanadium Carbide-Carbon Shells. To see the changes in properties, a pure Alumina sample was also prepared.

2.4. Die Manufacturing

The main tests being conducted on the composite were compression, wear and hardness tests. As the samples were being impacted with shockwaves, the dimensions of the shock tube were considered while producing the samples. Special dies were manufactured to prepare samples to accommodate them in the Universal Testing machine and Shock Tube.

To prepare the test samples, two sets of dies were manufactured. One die having a cylindrical cavity measuring 1.5x90 mm was made to prepare a test sample for the compression test. The second die, internal cylindrical cavity measuring 30x20 mm was designed to prepare a test sample for wear, hardness and shock tube tests. The dies were made of hardened steel with HRC 30.

After final turning and honing, the dies were coated with an anti-rust coating [5].

2.5. Preparation of the UTM Sample

The sample for the compression test in the Universal Testing Machine (UTM) was prepared with the help of the 1.5 x 90 mm die. Stearic acid was chosen as a lubricant for the die. The lubricant is essential to prevent the powder from sticking to the wall and for easy removal of the sample. During the preparation, it was also noticed that if only nanoscale materials were used during compaction, the sample would stick to the plunger.

Therefore a few grains of micro grade alumina were sprinkled on the surface of the plunger to facilitate easy removal of the sample. Polyethylene Glycol was selected as the binder. The binder is used to supply temporary strength to the green compact. As the temperature of the compact increases during sintering, the binders evaporate.

Two samples, one of pure alumina and one of aluminium oxide/vanadium carbide-carbon shells composite were prepared. The total volume of the test sample was found to be 3534.3 mm³. The density of pure alumina was taken to be 3.85 g/cc. 80% of the theoretical density was taken as the practical value and the pure alumina mass required was found to be 10.886 g. 1.08 g of polyethylene glycol was dissolved in 1.5 ml of water and heated until the solid powder dissolved. This was then added to the alumina and the mixture was thoroughly ground for 10 minutes.

Stearic acid was coated on the walls of the die and the powder was transferred into the die. It was then compacted with a force of 15 kN in a Universal testing machine. The powder was kept at this pressure for 10 minutes and then the die was released from the UTM. The sample was then carefully removed and preserved until sintering.

To prepare the Vanadium carbide-carbon shell reinforced sample, 10.52 g of pure alumina and 0.55 g of the vanadium carbide-carbon shells were taken. The powders were mixed and grinded for 30 minutes to obtain a homogeneous mixture. 1.01 g of polyethylene glycol was dissolved in 1.5 mL of water and added to the homogeneous mixture. It was then further ground for 10 minutes and the die was loaded, after being coated with stearic acid. The powders were again compacted at 15 kN force in a Universal testing in the machine for 10 min. The obtained green compact was carefully removed and stored [6].

2.6. Preparation of the Shock Test sample

The volume of the shock test sample was the same as UTM test sample. The same procedures mentioned above were followed. The pressure used to compact this sample was 12 kN as it was observed that increasing pressure further caused the sample to crack during removal. One pure alumina sample was prepared and two composite samples were prepared, one to characterize before the shockwave impact, and another for post impact characterization.

All the prepared samples were then pre-sintered at 200°C for 1 hour in an oven to remove the binder and organic impurities. The samples were then sent for sintering.

2.7. Sintering of the Test Samples

The pre-sintered samples were then sintered in a microwave furnace. This method was selected over conventional sintering as it has been found to reduce grain growth time and increase densification.

The hold times and temperature were also found to be significantly lesser for microwave sintering compared to conventional sintering. It was also seen that when alumina consisting of particles greater than 50 micrometer was used, the sample was not sintered even when the prescribed temperatures were reached. Many studies have suggested that the activation energy required for the atomic diffusion in large grains is greater compared to smaller particles. As a result, nano-scale alumina was synthesized and used in its place.

The samples were kept in a microwave furnace and the temperature was raised to 1500°C at the rate of 10°C/min. The samples were held at this temperature for 30 minutes after which it was allowed to cool in air.

While many studies suggest that maximum densification can begin and be achieved nearing 1400°C, the above-mentioned procedure was followed to ensure the complete sintering of the samples.

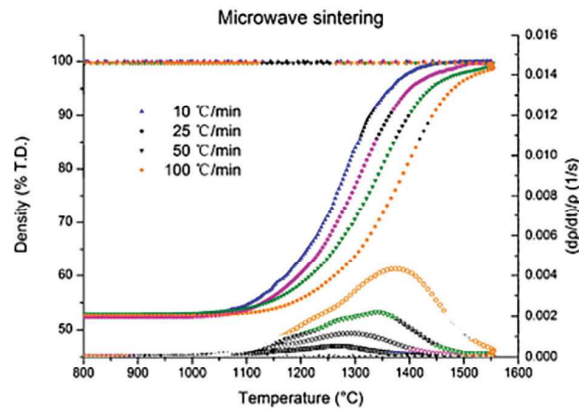


Fig. 1: Temperature profile for alumina sintering

As shown in the Fig. 1, the densification begins to occur above 1200°C. It is seen that a lower heating rate leads to faster densification. This may be due to the slower heating which leads to an even formation of necking around the alumina grains. The slow rate allows for atomic diffusion to occur more evenly leading to a dense sample.

3. Result and Discussion

3.1. Scanning Electron Microscope (SEM) Analysis

A scanning electron microscope (SEM) is a type of electron microscope that produces images of a sample by scanning the surface with a focused beam of electrons. The electrons interact with atoms in the sample, producing various signals that contain information about the surface topography and composition of the sample. The electron beam is scanned in a raster scan pattern, and the position of the beam is combined with the intensity of the detected signal to produce an image.

Thus a SEM microscope is a powerful magnification tool that utilizes focused electron beams to obtain high-resolution, three-dimensional images for topographical, morphological and compositional information. The SEM was done on the ceramic powders produced i.e. aluminium oxide and vanadium carbide as shown in Fig. 2 a-d below.

The SEM of Alumina is as follows:

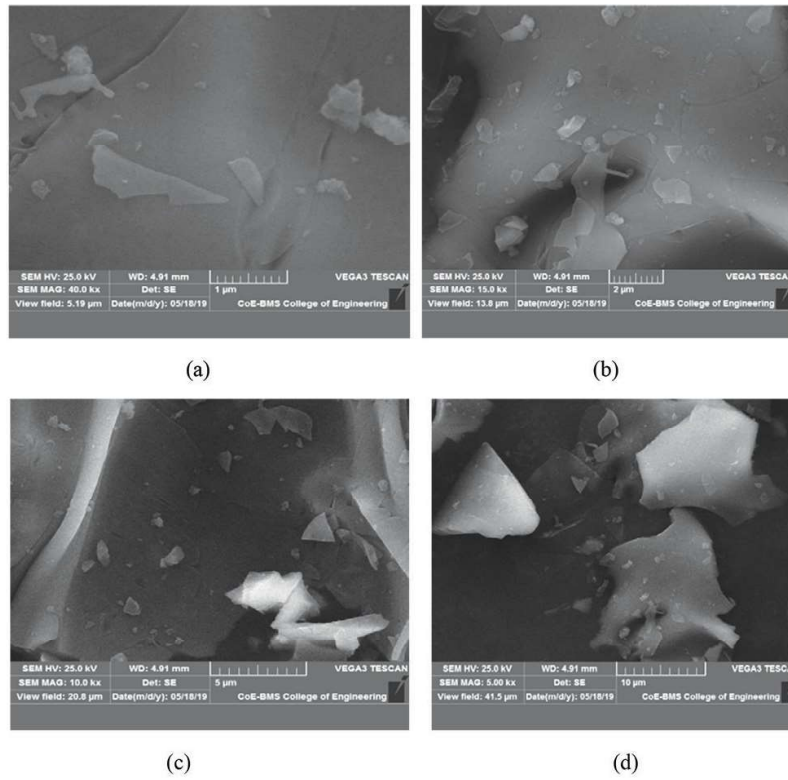
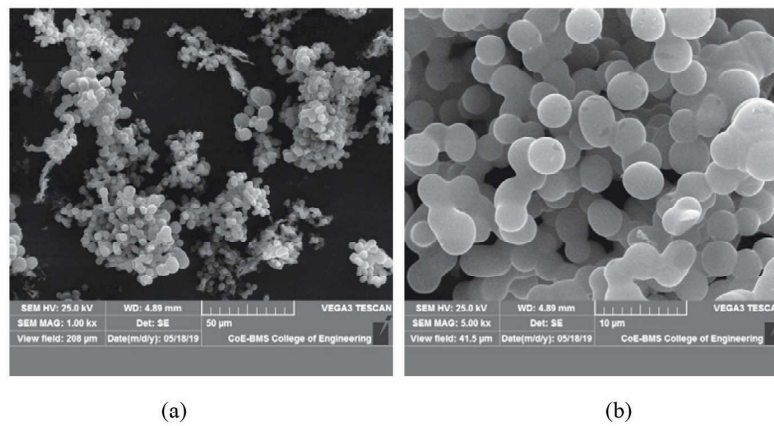
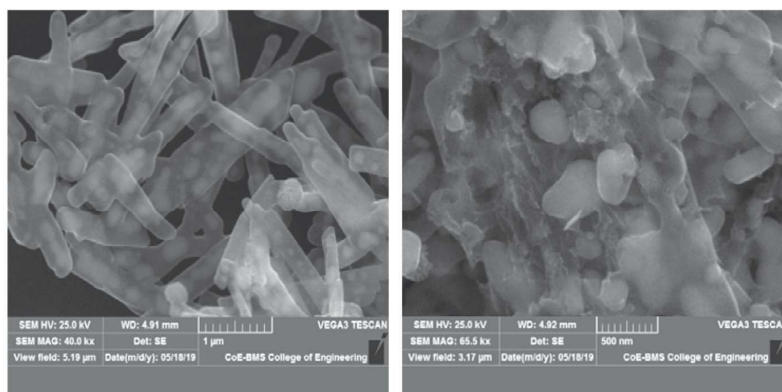


Fig. 2. a-d SEM images of α -aluminium oxide

From the SEM images of aluminium oxide, a platelet like structure was observed. Many particles were observed to be of less than 1 micrometer.

The SEM image of Vanadium Carbide is as follows:





(c)

(d)

Fig. 3. a-d SEM images of vanadium carbide

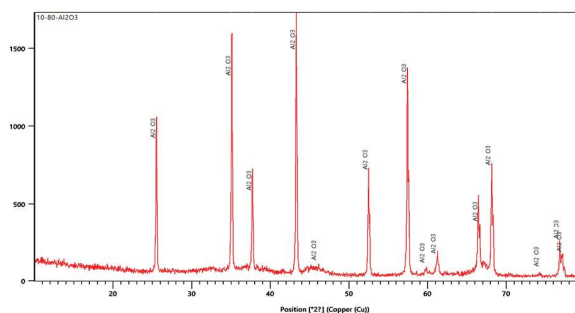
From Fig. 3. a-d, the SEM images of Vanadium carbide-carbon shell, we see that there appears to a shell-like structure encapsulating small nanoparticles.

As the XRD confirms that the major crystalline phase in the compound is Vanadium carbide, and the fact that a carbon shell was grown around the Vanadium oxide nano belts during synthesis, we can conclude that the image shows a carbon shell encapsulating vanadium carbide nanoparticles [7-8].

3.2 X-ray Diffraction (XRD) Analysis

X-ray diffraction (XRD) was a rapid analytical technique primarily used for phase identification of a crystalline material and can provide information on unit cell dimensions. The analysed material was finely ground, homogenized, and average bulk composition is determined.

The following graphs shown in Fig. 5.a and Fig. 5.b were the XRD patterns obtained when the two compounds were analysed.

**Fig. 4.a** XRD pattern of α -aluminium oxide

From the above graph we can see that all major peaks match that of alpha aluminium oxide or corundum. The lack of other major peaks indicates that the sample did not contain any other crystalline impurities. The sharp peaks indicate that the compound was crystalline and did not contain any amorphous phases.

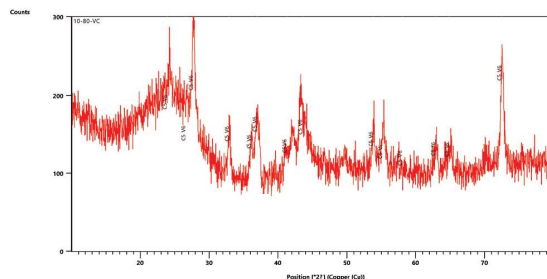


Fig. 4.b XRD pattern of vanadium carbide-carbon shells

From the above graph it can see that the major peaks correspond to the compound vanadium carbide and the peak broadening may indicate that the size of the crystallites was very small. It was confirmed the pattern that, it has semi crystalline nature [7-8].

3.3. Impact with Shockwaves

The sintered specimen then underwent finishing operations in order to obtain a smooth surface finish, on which the shockwaves would be impacted. After the finishing operation, the specimen was mounted on the driven end of the Reddy Tube and properly tightened onto a rigid cover plate.

The shock tube was then fired, the diaphragm inside the tube bursted and subsequently shockwaves of the magnitude of 8 bar and 700 K was produced and impacted on the surface of the specimen held at the driven end. The pressure sensors on shock tube were connected to a computer by a Cathode Ray Oscilloscope (CRO) and the graph of pressure rise with time was obtained as shown in fig 3.12 for calculating the speed of the impacted shockwave [9].

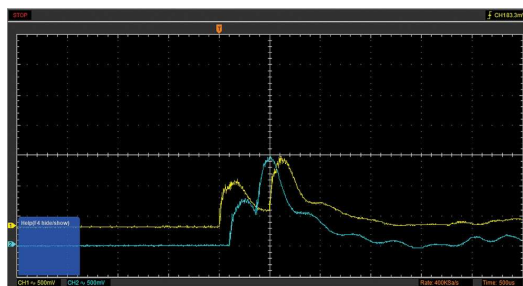


Fig. 5. Graph of Pressure of shockwave v/s Time

3.4. Densification

The term densification refers to ratio of density obtained from the sample to theoretical density of the compound. As ceramics are produced using sintering, it is impossible to achieve 100% densification due to the atomic transport mechanism followed during sintering. There are always pores left in between the channels formed for atomic transport, leading to a decrease in achieved density. Generally, higher sintering temperature leads to higher densification percentage. It is also dependent on the holding times [10].

The densities of the sintered samples were calculated and the percentage of densification achieved as compared to the theoretical value was obtained, as shown in Table 1 and Table 2 below.

Table 1 Densification of sintered samples in cylindrical shape

Sl. No.	SAMPLE	MASS OF SAMPLE (g)	VOLUME OF SAMPLE (cm ³)	DENSITY (g/cm ³)	DENSIFICATION ACHIEVED (%)
1	Alumina	4.6109	2.802	1.645	52.56
2	Alumina +VC	4.9958	2.889	1.729	54.71

Table 2 Densification of sintered samples in disc shape

Sl. No.	SAMPLE	MASS OF SAMPLE (g)	VOLUME OF SAMPLE (cm ³)	DENSITY (g/cm ³)	DENSIFICATION ACHIEVED (%)
1	Alumina	6.2470	4.101	1.52	48.1
2	Alumina +VC	5.9487	3.448	1.725	54.54

As inferred from the above values, the geometry of the sample also affects the densification achieved of the sintered sample. In case of cylindrical sample, pure alumina has achieved higher densification whereas in case of the disc shape sample, VC reinforced alumina has achieved higher densification. This could be due to the interaction of microwaves with different geometries and the presence of carbide particles in the matrix [10].

The theoretical density of alumina is 3.95 g/cm³. A densification of 80% was taken as the maximum densification value required i.e. density of the sintered sample was required to be 3.16 g/cm³. But the maximum densification obtained was 54.71% which had a density of 1.729 g/cm³.

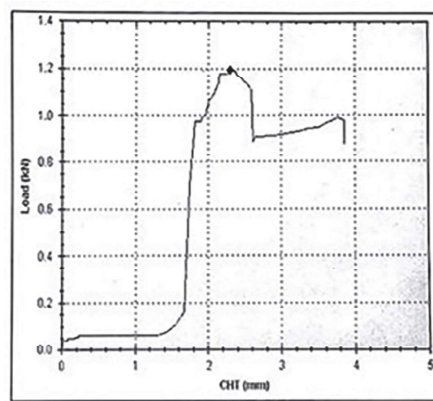
3.5. Compressive Strength Test

Compression testing is a very common testing method that is used to establish the compressive force or crush resistance of a material and the ability of the material to recover after a specified compressive force is applied and even held over a defined period of time.

The goal of a compression test is to determine the behavior or response of a material while it experiences a compressive load by measuring fundamental variables such as strain, stress, and deformation.

Compressive test was conducted on universal testing machine. The test was carried out as per ASTM C773-88 standard. Both the sample i.e. one before the impact of shockwave and one after the impact of shockwave were tested for strength.

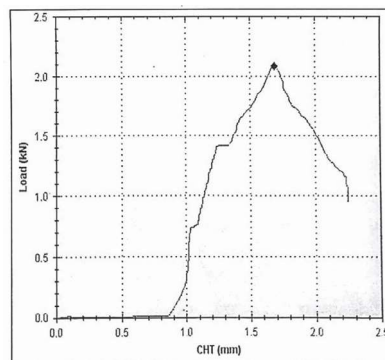
The compressive strength test was done in a computerized UTM and the load v/s deformation curve was plotted as shown in Fig. 6.1 and Fig. 6.2 below.



(a)

Fig. 6.1: Compressive strength of specimen 1 (alumina)

The compressive strength of pure alumina sample achieved was 7.28 MPa. As observed from the graph, it can infer that the sample has very less compressive strength at the outer layer. As the outer layer was compressed to its full extent (a to b), the load applied further to the bulk densified portion tends to show higher compressive strength (b to c), as shown in the above graph.



(b)

Fig. 6.2: Compressive strength of sample 2 (alumina + vanadium carbide)

The compressive strength of VC reinforced alumina sample achieved was 12.57 Mpa. This shows a considerable increase in the compressive strength of the alumina matrix composite because of addition of 5-wt% vanadium carbide [10-11].

This increase in the compressive strength can be attributed to the presence of VC- Carbon shells in the alumina matrix.

3.6. Thermal Stability

Thermal stability is defined as the ability of the material to withstand thermal shocks without any change in its properties. To test the thermal stability of the sintered sample, thermocouples were connected to both sides of the sample and the shock waves were impacted on one side of the sample. The temperatures recorded by the thermocouples were observed and noted.

The Hand driven shock tube was used to generate the shockwave of pressure up to 8 bar and Mach 1 speed. Thus the generated shockwave was impacted onto the disc specimen mounted on the open end of the shock tube [12-13].

A thermocouple setup was made to measure the temperatures at the two faces during the impact of shockwave onto the specimen and the obtained results are tabulated as shown in table 6.3 below.

Table 3 Thermocouple readings

Sl. No.	Thermocouple 1 reading (T_1) in K	Thermocouple 2 reading (T_2) in K
1	602	297
2	597	297
3	613	297
4	605	298
5	609	299

From the above table it can be concluded that this material has the capability to maintain the largest possible temperature difference across its faces over a shorter duration without any deformations.

Conclusion

The objective of the work was to perform a comparative study between pure alumina and VC-reinforced alumina composite and observe the effect of shockwaves on the ceramic composite.

Micro alumina (<10 microns) was synthesized using solution combustion synthesis and to reinforce this, Vanadium Carbide - carbon shell was synthesized using hydrothermal and carbo-thermal reduction method.

The synthesized compounds were ground along with the binding agent and sintered in Microwave furnace at 1500°C. The sintered samples were polished and tested for densification, compressive strength and thermal stability.

As the commercially available refractory materials have densification percentage between 50%-80%, 80% was selected as the upper limit for the sintering purpose. The maximum obtained densification was 54.72% and it was able to withstand the impact of shockwaves. Thus, we can conclude that the material produced can be used as an effective thermal barrier.

The compression test shows the inhomogeneity in the densification from the surface to the center of the specimen. It is also observed that the addition of VC has improved the compressive strength significantly along with the surface hardness.

The thermal stability test shows that the sample is able to withstand thermal shocks under high pressure. It is also observed that the material is capable of maintaining the largest possible temperature difference across its two faces.

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Conflict of Interest

The authors hereby declare no potential conflicts of interest with respect to the research, funding, authorship, and/or publication of this article

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