EFFECT OF SINTERING TEMPERATURE ON THE MICROSTRUCTURE OF ADVANCED ALUMINA CERAMICS

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DEDICATION

This project is dedicated to God Almighty who has been faithful thus far in my academic pursuit and to my parents Mr. & Mrs. Martins OMOZOJIE for their love, guidance, moral and financial support.

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I pray that the Almighty God continually guide and shower his unending grace upon our lives Amen

ABSTRACT

The formulation of an advanced ceramics using alumina is for the purpose of light weight. Ceramics have been considered one of the most important materials for lightweight high impact applications due to their low density, high compressive strength, and high hardness. This work is on the effect of sintering temperature on the microstructure of an advanced alumina ceramics (Al₂O₃) which was studied and samples were prepared and evaluated with temperature 1300°C - 1600°C. The process to prepare this samples are mixing the alumina which is the main ceramic material Al₂O₃ 96.2%, addictive (MgO 0.5% and CaO 2.3%) and the binder (Polyvinyl alcohol) 1% and then pressed into a mould using uniaxial pressing method and finally sintered at different temperatures to know the effect of temperature on the ceramics. The sintered samples where characterized using a scanning electron microscope test. From observation their results which were evaluated was discovered that the increase in temperature reduces the grain size of the alumina ceramic. Alumina having small grain size and high density yield good mechanical properties which are improved strength, hardness and wear resistance.

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CHAPTER ONE:

1.0 INTRODUTION

1.1 PREAMBLE

Ceramics have been considered one of the most important materials for lightweight armour applications due to their low density, high compressive strength, and high hardness (Silva, Al-Qureshi, Montedo, & Hotza, 2014). Ceramic materials used as ballistic armour must be sufficiently rigid to fragment the bullet and reduce its speed, transforming it into small fragments that should be stopped by the layer of flexible material that supports the ceramic. Thus, it is necessary that the ceramic material presents high elastic modulus and high hardness (Silva, Al-Qureshi, Montedo, & Hotza, 2014). Fracture toughness is also a very important requirement for this application.

The main ceramic materials used commercially in the development of ballistic armours are Al₂O₃, B₄C, SiC, and ceramic matrix composites (CMCs) such as Al₂O₃/ZrO₂. Alumina provides the best cost-benefit ratio among advanced ceramics, featuring high modulus of elasticity, high refractoriness, high hardness, and relatively lower cost. However, because of its low fracture toughness and low flexural strength, ballistic performance of alumina is lower when compared to SiC and B₄C (Avila *et al.*, 2011).

The properties of the alumina may be improved, either by introducing zirconia or by the manufacturing CMCs, which increase fracture toughness and flexural strength by introducing tetragonal zirconia particles or ceramic fibers, respectively (Bhatnagar, 2006). Thus, ceramic materials are usually a part of a ballistic personal or vehicle protection system. Even in this case, only with a rigorous control of the microstructure assured a reliable ballistic performance can be. For example, a system consisting of a composite of B₄C and glass fibers or aramid fibers coated with a protective fabric is often used. The replacement of metallic materials by ceramic materials in armoured vehicles may lead to weight reduction, autonomy increase, and higher level of protection (Callister, 2003).

A ballistic armour system consists of several layers. The first layer is usually formed of ceramic materials whose function is to cushion the initial impact of the projectile. This layer must fracture the tip of the projectile dissipating much of the kinetic energy of the projectile fragment mass and improve the distribution of impact pressure on the second layer. The second layer is also called backing and is formed of ductile materials. Its function is to absorb the kinetic energy of the fragments derived from the residual projectile and ceramics by plastic deformation (Silva, Al-Qureshi, Montedo, & Hotza, 2014). The most important requirement of the backing is no fail during the initial stages of the penetration process of the projectile, that is, the backing must withstand compressive stresses transferred to

the ceramic after impact. Thus, it would prevent the penetration of the shrapnel containing high kinetic energy and would not deform excessively, as this would jeopardize the lives of persons protected by the armour or the integrity of the equipment.

It is important to mention that impacts at high speeds (high kinetic energies) are high complexity phenomena that present limited reproduction, because parameters such as the incidence of the projectile on the armour, for example, are extremely difficult to be adjusted or prevented (Ernst, 2001).

The design of armour using ceramic materials should consider that the fracture is associated with instantaneous loads in ballistic impacts, which are quite different from those associated with static loads. In static load condition, stresses and strains are distributed throughout the body subjected to the impact and all points are involved in the start of fracture. In the instantaneous loads, stresses and strains are very well localized, so that fractures may occur in an isolated part of the body. This kind of change can dramatically affect the mechanical properties of the material due to the high pressures and loading rates. The impact of high kinetic energy projectiles on the ceramic composite armour usually gives rise to a cone of fractures with radial and circumferential cracks (Callister, 2003).

Projectiles fired over alumina targets, whose tips were flattened, presented higher residual speeds after drilling the targets and therefore greater power of penetration in relation to projectiles with sharp geometry. In this case, immediately after the impact on the first plate occurs the formation of an axial crack in the interface between the plates, which causes premature fracture of the assembly. During the first stage of the mechanism of penetration, the most important factor is to keep the integrity of the ceramic so that it can erode the greatest possible amount of mass of the projectile that is, delaying the startup of the fracture of the ceramic material. This factor is decisive for the choice of the ceramic material to be used.

It is not possible to ensure an effective correlation between ballistic performance and a single characteristic or property of the material, due to the dynamic nature of the event occurring at intervals of time ranging from Nano- to microseconds. Thus, ballistic tests under certain conditions are always required to determine the effectiveness of the protection systems. The development of ceramic ballistic requires careful assessment of physical and mechanical properties of the material in order to obtain adequate performance ballistic plates to the level of protection required.

1.2 STATEMENT OF PROBLEM:

Ballistic vests used by Nigerians soldiers are imported from foreign countries (Cynthia 2015). Also, Nigerians has overtaken Iraq, Latin American countries and Afghanistan as the world biggest importer of armoured vehicles it is estimated that about 30 percent customers for armoured vehicles worldwide come from Nigeria. In the past years, manufactures of armoured vehicles have exported an estimated 800 to 900 units to Nigeria at cost of N60 billion (Bukola, 2003).

Every year, about 60 sworn police officers are shot dead in the line of duty. At same time, about 20 saved by wearing armour. Had all the officers shot in recent years been earing armour when shot, another 15per year would likely been save from gunshot wounds (OTA – Congress, 1992).

In Nigeria, we have not been able to produce this ballistic wears for our used, which we can eventually offer maximum protection from firing flying bullets on the battle field (Adze, 2014). The inference one can draw from this is that we only depend on the protection of other countries. The country only endangers the lives of it citizens if there is an international, dispute, bilateral or social – economic problem with any of the countries we procure the hardware. Therefore, it's necessary to intensify research toward armour technology in Nigeria.

1.3 AIM AND OBJECTIVES:

1.3.1 AIM:

Formulate and evaluate the effect of temperature on the microstructure characteristics of a sintered alumina ceramics for high impact application.

1.3.2 OBJECTIVES:

- 1. Formation and development of alumina ceramics
- 2. Microstructural characterizations of the sintered samples.
- 3. Analysis and comparing the result of the developed ceramics sample with the established/standard result.
- 4. Recommendation for further research work.

1.3 RESEARCH JUSTIFICATION:

The research is to build up the knowledge on using alumina as a material for amour technology as the need of armour wears by security operatives is indisputable on strategic assignment involving weapon flying bullets. As Nigeria solely depends on the importation of armour hardware, formulation of light weight plate using alumina for Nigeria security independence and it would generate employment.

1.4 SCOPE

The research is for formulation of light weight ceramics plate using pure alumina (Al_2O_3) particulate and other additives. To determine microstructural characterizations of the developed light weight ceramics plate for possible high impact application.

1.5 SIGNIFICANCE OF STUDY

From the history of ceramics, ceramics have been considered one of the most important materials for light weight armour application due to their low density, high compressive strength, hardness (Silver *et al.*, 2014). Hence, the significance of studying ceramics materials on armour application is to establish and domesticate technology of producing ceramics components of body armour, bullet proof doors and armoured vehicles.

CHAPTER TWO

2.0 LITERATURE REVIEW

Efficient armour requires hard, tough and lightweight materials with significant penetration resistance and energy absorbing capabilities also High hardness steel was used as one of the first engineered armour materials (Kiran & Niranjan K, 2015). However, with increasing demands on reducing weight, researchers and designers explored other materials. Aluminum alloys, other lightweight metals such as titanium and their alloys, were hence used as armour materials. Ceramics are stronger than metals under heavy compressive loads acting in the vicinity of areas subjected to impact (Kiran & Niranjan K, 2015). Their much lower density makes them attractive lightweight alternatives to metallic armour. But processing of ceramics requires high temperature and pressure. Hence, they are more expensive than metals. Another drawback of ceramics is their brittle behavior causing heavy degradation on impact. They, therefore, have lower capability to withstand multiple hits than metallic armour. Attempts were made to improve the ductility of ceramics by embedding ceramic fibers inside bulk ceramics. Such materials are popularly known as ceramic matrix composites (CMCs). However, CMCs are difficult to process and even more expensive than conventional ceramics. Their use is hence, restricted to specialized applications. Other approach to improve the energy absorption of ceramics was by using ceramic-metal or ceramic-FRP composite armour. Such composite armour consists of a ceramic layer backed by a composite or metal layer. The ceramic layer provides primary ballistic impact resistance. The inner composite or metal layer is for secondary energy absorption. It also serves as the backing for brittle ceramics. Due to the high specific strength and specific stiffness of FRP composites used as backing layer, ceramic-composite armour is one of the lightest alternatives to monolithic metallic armour (Kiran & Niranjan K, 2015). There are other forms of armour using only FRP composites such as fabric or textile armour. These are suitable for lower threats such as handgun bullets. They cannot sustain more lethal threats (Kiran & Niranjan K, 2015).

Ceramics were used for armour applications during the late 1960s and early 1970s. Early experiences on the behavior of Al₂O₃ ceramics subjected to ballistic impact were documented in 1979 (Lanz, 2001). Some of the widely used ceramics for armour applications are Al₂O₃, B₄C, SiC, TiB₂ and WC. Armour ceramics can be classified on the basis of density (Gooch and Burkins, 2001). Ceramics with density greater than RHA (Rolled Homogeneous Armour) are high density ceramics. Those with density lower than RHA are low density ceramics. Typical high density ceramic is WC, whereas low density ceramics include Al₂O₃, B₄C, SiC and TiB₂. Another method to classify ceramics is based on the manufacturing process (Callister, 2003). Some widely used processes for ceramic armour are

sintered, pressed-sintered, reaction-bonded3 and gel cast. Pressing can be uniaxial and isostatic. It can be at room temperature or under heated conditions (Kiran & Niranjan K, 2015).

The long process of moving ceramics from craft to science based technology conducted under the direction of engineers was underway in the 1800s and has continued to the present day watchman cited (Cahn, 2001). Since the ceramics have been developed for an immerse range of advanced application were materials are required that can resist thermal shock, provide help temperature and electrical insulation, act as abrasives, have a better (compressive) strength weight ratio. More recent breakthrough have included: in 1965, the development of photovoltaic cells which convert light into electricity (solar cells); in 1987, the discovery of a superconducting ceramics oxide with 60 degrees centigrade higher temperature than metallic superconductors with potential use in integrated circuits is high speed computers, recently in the early 1990s the development of a range ceramics materials called "SMART" materials that sense and react to variables surface conditions (Kiran & Niranjan K, 2015). An example of use is in the triggering airbags. The range of ceramics developed to exploits their special properties to meet at range of demanding engineering applications is vast. It now includes:

2.0.1 ELECTRICAL CERAMICS

Phosphors for cathode ray tube screen which have been used until recently for computers, TV, radar and oscilloscope screens and computer monitor:

- Piezo electric (used in sonar and ultra-sonic), pyro electric, electro-optic and magnetic ceramics
- Ceramics conductors (e.g. the varistor), supersonic conductors,
 superconducting ceramics
- Ceramics capacitors

2.0.2 MAGNETIC CERAMICS

- Ferrite as the ceramics core of the 1948 giant synchrontron (replacing the metallic core)
- Computer memory
- Telecommunication

2.0.3 THERMAL INSULATION AND REFRACTORY MATERIALS

- Refractory materials
- Ceramics tiles for thermal insulation against high re-entry temperatures of the space shuttle
- Alumina ceramics for missiles and rocket nose cones

Silicon carbide and molybdenum disilicide for rocket

2.0.4 NUCLEAR POWER

- Fuel elements as a ceramics compound
- Control rod element in ceramics form

In line with other advanced materials the recent changes in focus from micro to a Nano-structured approach has resulted in engineering ceramics with high temperature super-plasticity; better mechanical strength (fracture behavior approaching quasi ductility, strength comparable to single crystal, improved reliability and improved high temperature properties and a range of new functional properties such as high electrical conductivity and elevated thermal conductivity Sajgalik, 2004)

2.1 ARMOUR MATERIALS CLASSIFICATIONS

Armour materials can be classified into three main groups, namely

- 1. Metallic
- 2. Ceramics
- 3. Composite

2.1.1 METALLIC ARMOUR

Metals are still the most widely used materials in armour design. The main advantage of these materials is that, they are capable of carrying structural and fatigue loads while offering efficient protection. They are less expensive compared to the other materials.

The most commonly used metallic materials in armour fighting vehicles are steel. The main properties such as toughness, hardness, good fatigue strength, ease of fabrication and joining and relative low cost make it a popular material for armoured vehicle hulls (Hazel, 2006).

Steel armour can be studied in four main groups which are Rolled Homogeneous Armour (RHA), High hardness armour (HHA), Variable Hardness Steels and perforated Armour (Hazell, 2006)

2.1.1.1 ROLLED HOMOGENEOUS ARMOUR (RHA)

Rolled homogeneous armour (RHA) is usually used in depth of penetration testing as a benchmark material (Hezell, 2006). Therefore, it is used to describe and compare the performance of different armour materials chemicals composition (Hezell, 2006). The classification of RHA according to UK Ministry of Defense Standard for Armour plate (DEF STAN, 2004) are given below

Table 2.1: Composition of RHA

С	Mn	Ni	Cr	Mo	S	P
0.18-0.32	0.65-1.50	0.05-0.95	0.00-0.90	0.30-0.69	0.015	0.015
					(max)	(max)

Source: (DEF STAN, 2004)

Table 2.2: Classification of RHA

Classification	Description	Hardness	Tensile	Elongation
		(BHN)	Strength	(%) Min
		2 0	(MPa)	
Class 1	Readily weld-able	262-311	895-1,050	15
u .	steel subjected to		ă	a .
	structural loads			- 4
Class 2	Readily weld-able	255-341	895-955	14-16
H	steel to protect		G	
	against AP	3	•	
19 19	ammunition			
Class 3	Readily weld-able	470-540	1,450-1,850	8
# ±	higher hardness			
	steel			g.
	manufactured in	8		
	thin sections	6		
Class 3A	Readily weld-able	420-480	1,200-1,600	9
	higher hardness			5
·	steel	-8		

	manufactured in			
	thin sections			
Class 4	Higher carbon and	475-605	1,450-2,000	7
	alloy contents			
	higher hardness			
2	armour for thick			u .
	sections	# # # # # # # # # # # # # # # # # # #	1	
Class 5	High alloy content	560-655	1,800-2,400	6
	armour with very	*		
	high hardness	78		
9	used for special			
	applications such	2		
5	as perforated	*		
	armour		1	5

Source: (DEF STAN, 2004)

2.1.1.2 HIGH HARDNESS ARMOUR

High hardness armour (HHA) on the other hand, is the name given to a class of homogeneous steel armour which has hardness valueless exceeding 430 BHN (Hazell, 2006).

2.1.1.3 VARIABLE HARDNESS STEEL (VHS)

Variable hardness steel plates introduce some advantages with varying through thickness properties. By surface hardening of one side of thick low-carbon steel plates. It is possible to incorporate both hard disruptive and tough absorbing properties in single materials (Hazell, 2006).

The main advantages are that; the more ductile backing layer is able to arrest crack propagation in the armour plate while the hard front layer is able deform or fracture the threat. The effectiveness of dual-hardness armour (DHA) is given by a comparison in the table below

Table 2.3: The effectiveness of dual-hardness armour (DHA)

Armour steel	Density (kg/m³)	Thickness (mm)	Area Density
41	8	9	(kg/m^3)
HHA (550BHN)	7830	14.6	114
HHA (550HHN)	7850	12.5	98
HA (600-440 BHN)	7850	8.1	64

Source: (Ogorkiewicz, 1991)

2.1.1.4 ARMOUR PERFORATION

In perforated armour, holes are introduced into the steel plates. These holes in high hardness steel plate have been shown to be an effective way of disrupting and fragmenting incoming projectiles. The mechanism can be regarded as edge effect (Chocron *et al.*, 2001) also studied the impact of the 7.62 mm APM2 Projectile against the edge of a metallic target and a photograph of a fractured core due to aforementioned edge effect.

ALUMINUM ALLOYS

Aluminum alloys also provide a versatile choice for an armour design engineer. The main advantage is that; it has a relatively low density while the tensile strengths range from 60-600MPa. It can be deduced that equal mass of aluminum armour will have larger volume compared to steel, which leads to improvement in rigidity. Material properties of some commonly used aluminum alloys in the table below (Hazell, 2006).

Table 2.4: Materials properties of some aluminum alloys currently used in AFVs.

Alloy	Proof Strength	Ultimate	Elongation	Hardness
Li .	(MPa)	tensile (MPa)	(%)	(HV)
Type 5083	(0.1 %) 278	386	6	~100
Type 5017	(0.1 %) 278	490	8	~160
Type 5039	(0.2 %) 420	475	10	~150

Source: (Hazall, 2006)

However, there are some disadvantages associated with aluminum alloys. The harder alloys that are suitable as armour are susceptible to stress corrosion cracking (Hazell, 2006). This type of failure occurs when the aluminum alloy is attacked by a corrodant while it is lower than that of yield strength and the residual stress induced during machining, assembly or welding can lead to failure.

These alloys also possess lower spall strength than steel so that they are prone to scabbing. This makes it necessary to employ a spall liner behind the armour. The ballistic grade form (Ti-6Al-4V) of titanium also provides good alternatives to high strength and hardness (UTS 900 - 1300 MPa, BHN 300 - 350). However, high cost related with titanium alloys is a prominent shortcoming.

2.1.2 CERAMICS ARMOURS

It can be anticipated that the resistance of a given material to penetration mainly depends on its compressive strength (Rosenberg *et al.*, 2009). Ceramic materials which possess high compressive strength and hardness values are good candidate materials as for the armour designer because of their relatively low density (Hazell, 2006). High strength that are an order of magnitude higher than those of metals. Then, it seems possible to make an assumption that ceramic faced targets will be efficient for armoured protection (Rosenberg, 2009).

The cost of ceramics tiles is taken into consideration besides its performance. A comparison of some ceramic materials with prices is given in the table below (Roberson, 2004).

Table 2.5: Relative cost of ceramic materials for armour applications

Ceramic	Bulk density	Hardness	Kıc ^a (MPa.m ²)	Relative cost
	(kg/m^3)	(HV)		
98(%)Al ₂ O ₃	3,800	1,600	4.5	1.0
RB ^b SiC	3,100	3,200/2,2700	~4.5	2.5
Sintered SiC	3,150	2,700	3.2	4.5
HP ^c SiC	3,220	2,200	5.0	9.0
HP B ₄ C	3,520	3,200	2.8	16.0

Source: (Roberson, 2004).

a = Fracture Toughness

b = Reaction Bonded

c = Hot Pressed

2.1.3 POLYMERIC ARMOURS

Polymeric composite materials possess high specific strength and specific stiffness and they are able to absorb significant part of kinetic energy induced by projectile impact. They also have relatively lower densities (Adze, 2014). These materials consist of laminates of matrix bonded reinforcement fibers. The function of the matrix is to provide a medium for the diffusion of load to the stronger and stiff fibers (Adze, 2014).

Table 2.6: Typical fiber materials are S-glass, E-glass, aramid carbon and boron. Some properties of these materials are presented in the table below. (Edwards, 2000)

Fiber	Bulk density	Tensile	Young's	Failure strain
	(kg/m^3)	strength (MPa)	modulus	(%)
			(GPa)	
Aramid	1440	2990	60	3.6
(low modulus)			1	
Polyethylene	970	3200	99	3.7
(high modulus)				
E-glass	2600	3500	72	4.8
S-glass	2500	4600	86	5.2
Carbon (high	1780	3400	240	1.4
strength)		2 W		
$C = \sqrt{\Gamma} \cdot 1 = 1$	2000)			

Source: (Edwards, 2000)

2.2 ARMOUR CONFIGURATION

Armour configuration can be classified in three main groups according to the way they treat the threat; these groups are passive, reactive and active armours (DENIZ, 2010).

2.2.1 PASSIVE ARMOUR

Passive armour is designed to absorb the kinetic energy of a kinetic energy projectile or a shaped charge jet. Special combinations of high strength materials and geometrical design are used to achieve desired mechanism against aforementioned threats (DENIZ, 2010).

2.2.2 SLOPPED ARMOUR

This armour is placed obliquely rather than having a vertical surface. The thickness of armour can be increased by this way. The second purpose is to ricochet or deflect incoming kinetic energy threats (DENIZ, 2010).

2.2.3 SPACE ARMOUR

It's commonly used to defeat shaped charge jet by increasing the distance the jet has to travel to penetrate the armour configuration. Moreover the internal layer can be designed to tumble and deflect incoming kinetic energy threats (DENIZ, 2010)

2.2.4 SLAT ARMOUR

It works by holding off the shaped charge device from the skin of the vehicle, and increase the way the jet has to travel so that stand off effect can occur (DENIZ, 2010).

2.2.5 COMPOSITE ARMOUR

This armour makes use of special combination of steels, ceramics and other materials to absorb and diffuse the damage caused by threat (DENIZ, 2010).

2.2.6 REACTION ARMOUR

Reactive armours make use of elements which are sandwiched between two metal plates. They react upon impact of a threat and use special mechanism to defeat the threat and use special mechanism to defeat the threat. This armour can be classified as

- 1. Explosive reaction armour
- 2. Non explosive reaction armour
- 3. Non energetic reactive armour
- 4. Electromagnetic reactive armour

2.2.7 ACTIVE ARMOUR

Active armours make use of sensors to detect incoming threats and are designed respond to intercept, disrupt or deflect this threat (DENIZ, 2010)

2.3 SINTERING: Sintering is the process of compacting and forming a solid mass of material by heat or pressure without melting it to the point of liquefaction. Ceramics sintering is a process used in the manufacturing of pottery and other ceramics objects. These objects are made from substances such as glass, alumina, zirconia, silica, magnesia, lime etc. some ceramics raw materials have allowed affinity for water and low plasticity index than clay, requiring organic additives in the stages before sintering.

2.4 REVIEW OF RELATED WORKS (Literature Review)

(DEMĐR, *et al.*, 2008) Worked on the investigation on the ballistic performance of alumina/4340 steel laminated composite armor against 7.62mm armor piercing projectiles and some important mechanical properties for the 4340 steel and the alumina are given in Tables below respectively. One can see that increasing the tempering temperature decreased the hardness and strength of the steel significantly. On the other hand, the hardness of the alumina was found to be ~ 1900 HVN that was much higher than the 4340 steel.

Table 2.7: Table of increasing the tempering temperature decreased the hardness and strength of the steel significantly (4340 steel)

Heat treatment	Hardness	Yield strength	Tensile	Ductility
	(HRC)	(MPa)	strength (MPa)	(%Elongation)
1	40	1200	1550	13.7
2	50	1300	1600	13.0
3	60	1550	1855	12.0

Table 2.8: Table of hardness of the mechanical properties of alumina

Ceramic	Hardness (HV)	Compressive	Bending streng	th
		strength (MPa)	(MPa)	
Alumina	1900	2100	330	

Also studied that any ballistic threat has to be stopped without perforation at the backing layer of the composite for safety. For this reason, the criterion in this work was considered as whether the backing layer was perforated or not by the projectile. The samples, of which backing plates resisted to the projectile without perforation, were accepted as satisfactory.

(Dora, et al., 2015), Worked on the influence of sintering time on properties of alumina-based ceramic composite. The sintering time used was 1600°C and was heated 100°C per hour. The method adopted for the ceramics powder mixes, the specimens were compacted by uniaxial pressing. Also the research shows that the maximum values of hardness and mechanical bending strength were found at 5hours of sintering time.

(Arindam & Subrata, 2013) Worked on effect of Al₂O₃ content and process variables on structure and properties of Al-Al₂O₃ compacts, the research was of the effect of sintering on the ceramic material. he used scanning electron microscope to determine the size, shape and morphology of the aluminum and aluminum powder. during the experiment the specimen was pressed at various pressures from 115MPa to 290MPa in a pneumatic press of 25.4 mm die and then sintered at different temperatures of 573, 673, 773 and 873 K for one hour in an argon atmosphere. The result shows that on sintering, hardness of the sintered compact gradually decreases with increasing temperature.

(Regassa, et al., 2014), Worked on the modeling and simulation of bullet resistant composite body armor. Due to composite body armour outstanding mechanical properties, flexibility in design capabilities, ease of fabrication and good corrosion, wear and impact resistant. Composite body armor is an item or piece of clothing that is designed to protect the wearer against a variety of attacks. They can be

made to stop different types of threats, such as bullets, knives and needles, or a combination of different attacks. Body armor has 2 types which are soft body armor and reinforced body armour. The soft body armour which is used in regular bullet and stab proof vests and hard armor that is rigid while the reinforced body armor, and is used in high risk situations by police tactical units and combat soldiers.

(Silva, et al., 2014), Worked on mechanical characterization and ballistic test of alumina based armour plates. Three compositions (92, 96, and 99 wt% Al2O3) were tested for 10 mm thick plates processed in an industrial plant. Samples were pressed at 110 MPa and sintered at 1600°C for 6h. Relative density, Vickers hardness, and four-point flexural strength measurements of samples after sintering were performed. Results showed that the strength values ranged from 210 to 300 MPa depending on the porosity, with lower standard deviation for the 92 wt% Al2O3 sample. Plates (120 mm × 120 mm × 12 mm) of this composition were selected for ballistic testing according to AISI 1045, using a metallic plate as backing and witness plates in the case of penetration or deformation. Standard NIJ-0108.01 was followed in regard to the type of projectile to be used $(7.62 \times 51 \text{ AP},$ Level IV, 4068 J). Five alumina plates were used in the ballistic tests (one shot per plate). None of the five shots penetrated or even deformed the metal sheet, showing that the composition containing 92 wt% Al2O3 could be considered to be

a potential ballistic ceramic, being able to withstand impacts with more than 4000 J of kinetic energy.

(Fawaz, et al., 2003), Worked on numerical simulation of normal and oblique ballistic impact on ceramics composite armour. The finite element results are compared with experimental data from different sources both for normal and oblique impact, respectively. Simulation of the penetration processes as well as the evaluation of energy and stresses distributions within the impact zones highlight the difference between normal and oblique ballistic impact phenomena. The findings show that the distributions of global kinetic, internal and total energy versus time are similar for normal and oblique impact. However, the inter laminar stresses at the ceramic composite interface and the forces at the projectile—ceramic interface for oblique impact are found to be smaller than those for normal impact. Finally, it is observed that the projectile erosion in oblique impact is slightly greater than that in normal impact.

(Vanichayangkuranont, et al., 2013) Worked on the Numerical Simulation of Ballistic Impact on Ceramic Armor. A preliminary investigation of ballistic impact on ceramic armor was carried out by recourse to numerical simulation using finite element method. Following our earlier study on the effect of stress wave propagation from dynamic loads, the current computational model was developed with built-in brittle failure criteria, aiming to correlate with experimental ballistic

testing. It was found that the rate that the ceramic armor absorbed the bullet kinetic energy raised as the armor thickness increased.

(Sanusi O.M, et al., 2016) Worked on Development of Wood-ash/Resin Polymer Matrix Composite for Body Armour Application. The usual waste from wood after combustion, wood ash, was used as the additive in the development of the PMC (Polymer Matrix Composite). Chemical composition of the prepared wood ash was determined using X-ray florescence spectrometry. The mechanical properties of the developed PMC were evaluated and discovered to have been enhanced through the inclusion of the wood ash particles. 2.3% inclusion of wood ash resulted in the tensile strength value of 104N/mm2 against 86 N/mm2 for neat reinforced resin. The impact strength of the PMC increased from 50 J/mm2 (without wood ash) to 112 J/mm2 while 25.4 HRF was recorded for the hardness value of the PMC with wood ash against 23.4 HRF for the neat reinforced PMC. The microscopic analysis also revealed that the wood ash particles were uniformly distributed in the matrix without indication of segregation. When subjected to ballistic tests, the developed PMC successfully arrested the 9 x 19 mm ammunitions after perforating some layers of the sandwiched sample.

(DENIZ, 2010) States that the ballistic testing of armour is the most important part of it. It is impossible to test every condition and it is necessary to limit the number of tests to cut huge costs. With the introduction of hydrocodes and high

performance computers; there is an increasing interest on simulation studies. This study deals with the numerical modeling of ballistic impact phenomena, regarding the ballistic penetration of hardened steel plates by 7.62 mm AP (Armor Piercing) projectile. Penetration processes of AP projectiles are reviewed. Then, a survey on analytical models is given.

(Christian, et al., 2002) Worked on Influence of material properties on the ballistic performance of ceramics for personal body armour. They conducted penetration test on four different ceramics materials which are alumina, modified alumina, silicon carbide and boron carbide. His experiments consisted of impacting ceramic tiles bonded to aluminum cylinders with 0.50 caliber armour piercing projectiles. The results are presented in terms of ballistic efficiency, and the validity of using ballistic efficiency as a measure of ceramic performance was examined. In addition, the correlation between ballistic performance and ceramic material properties, such as elastic modulus, hardness etc.

CHAPTER THREE

3.0 METHODOLOGY

3.1 MATERIALS

The major materials for the experiment are;

- 1. Alumina powder (Al₂O₃)
- 2. Additives (magnesium oxide MgO and calcium oxide CaO)
- 3. Binders (polyvinyl alcohol)

3.2 EQUIPMENT

Equipment used are listed below

- 1. Mold
- 2. Pressing machine (110MPa)
- 3. Furnace
- 4. Mortar and pestle
- 5. Microstructural testing machine
- 6. Electronics balance
- 7. Power hack saw

3.3 EXPERIMENT PROCEDURE

3.3.1 MATERIAL SELECTION

This study required magnesium oxide (MgO), calcium oxide (CaO), high purity powdered alumina (Al₂O₃) and polyvinyl alcohol (PVA). Alumina powder was the major materials used for the research, produced by AdValue Technology, LLC, United States. Magnesium oxide (MgO) and calcium oxide (CaO), served as hardening additives and grain stabilizer to the powdered alumina respectively. The polyvinyl alcohol (PVA) served as a binding agent. The chemical analysis of 99.999% Al₂O3 as provided by the manufacturer is shown below:

Table 3.1: Composition/information on ingredients

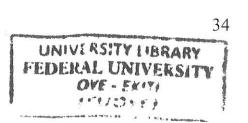
Ingredient	Formula	Percent	CAS No.
Aluminum oxide	Al ₂ O ₃	≥99.999%	1344-28-1

Table 3.2: Physical and chemical properties

Appearance: white powder

2980
2050
3.97
Insoluble in water
Odorless

- 3.3.2 The method to be used to achieve this experiment is via sintering of the alumina which includes:
 - Mixing the powder (alumina), additives (magnesium oxide MgO and calcium oxide CaO), binder (polyvinyl alcohol)
 - Mixed powder is then introduced into the prepared mold and uniaxially pressing it to form a green body
 - Sintering at temperature 1300 °C to 1600 °C



Certification of Test for Alumina Powder

Conclusion	Qualified	Inspector	echn	0/2	
Impurity Total		\$ 10× 10 ⁻¹ %			
	Ti	≤3	0.94	Pass	
	Zn	\$3	<0.4	Pass	
	Pb	≤3	<0.1	Pass	
7, 705	Cr	≤3	< 0.84	Pass	
	Ca	≤3	1.0	Pass	
Total impurity (×10 ⁻⁴), %s	Fe	≤5	1.5	Pass	
	Cu	≤3	<0.98	Pass	
	Mg	≤3	1.0	Pass	
	Na	≤5	1.1	Pass	
	Si	≤5	1.79	Pass	
Impurity Testing	Elements	Standard	Test Resulsts	Conclusio	
Inspection Quantity	20kg				
Inspection Method	Testing + Visual Check				
Appearance	White Powder				
Crystal Form	α Alumina				
Batch Quantity	20kg				
Batch Number	161110				
Item Code	ALO-5N				
Manufacturing Date	12/31/2016				
Product Name	High Purity Alumina Powder				

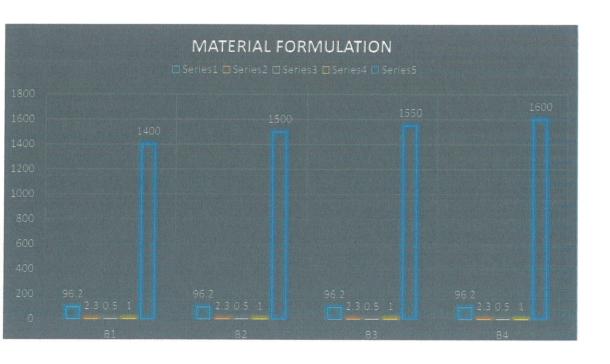
Date:

3.3.3 Material Formulation

The material formation weight percentage adopted for the research is as shown in table 3.2

Table 3.3: Proportion of material formulation of the ceramics

Designation	Al ₂ O ₃	CaO	MgO	PVA	Sintering Temperature
	%wt	%wt	%wt	%wt	°C
B1	96.2	2.3	0.5	1	1300
B2	96.2	2.3	0.5	1	1400
В3	96.2	2.3	0.5	1	1500
B4	96.2	2.3	0.5	1	1600



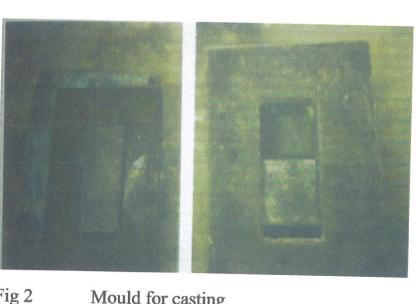
After the appropriate formulation, the materials were mixed thoroughly in the mortar with pestle in order to avoid significant density gradients and textures in the green body, which can be amplified during sintering, leading to distortions and internal mechanical stresses.

Fig 1 The mortar and pestle



3.3.4 **MOLD FABRICATION**

The mild steel mold which was used for forming process after the materials has been mixed properly.



Mould for casting

3.3.5 SINTERING

This is the process whereby the ceramics material has gone through the process of mixing the powder with binder, deflocculant, water and the ceramics powder then sprayed because from the mixing a slurry is formed after which the dried powder is put into a mold and pressed it to form a green body. The green body is sintered to 1300°C – 1600 °C at the heating rate of 25°C per min in a furnace.

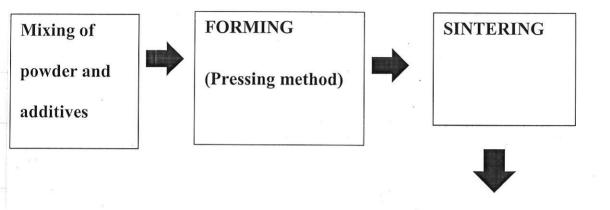
Fig 3 Electric furnace for sintering



3.4: MICROSTRUCTURE TEST:

Scanning electron microscope combined energy dispersive X-ray analysis is a good tool in many field of science and it is found in many applications of problems of archaeological ceramics. Scanning electron microscope yields high resolution images of ceramics surfaces also the interior can be studied either as a polished section or as a fracture surface. SEM provides pictures which can easily be interpreted in terms of structural details (FROH, 2004). To carry out this test the equipment is available at Ahmadu Bello University, Zaria, kaduna state.

FLOW CHART BELOW



Microstructural test

(SEM)

CHAPTER FOUR

RESULTS AND DISCUSSION

MICROSTRUCTURE CHARACTERISTICS:

SEM (Scanning Electron Microscope) images of the dry pressed alumina samples at different sintering temperatures

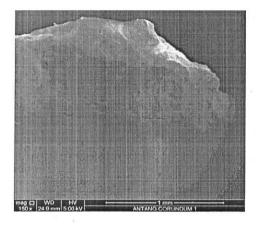


Fig 4a: SEM image of the sintered alumina at 1500°C (X150)

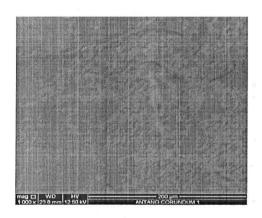


Fig4b: SEM image of the sintered alumina at 1500°C (X1000)

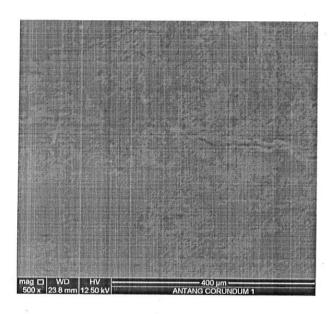


Fig 4c: SEM image of the sintered alumina at 1500°C (X500)

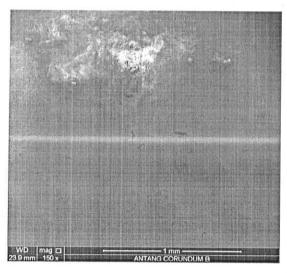


Fig 5a: SEM image of the sintered alumina at 1400°C (X150)

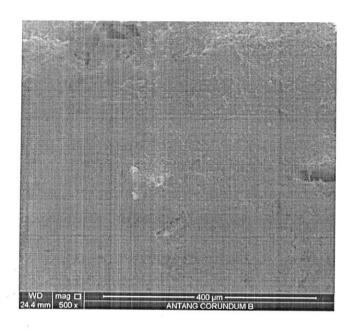


Fig 5b: SEM image of the sintered alumina at 1400°C (X500)

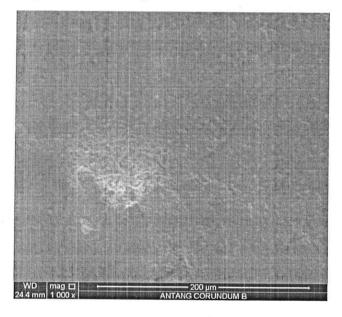


Fig 5c: SEM image of the sintered alumina at 1400°C (X1000)

The difference in the microstructures of the two samples show the effect of the temperature on the result. From fig 1a the microstructure from the result of the test the temperature causes some deformation on the sample like the cracks shown and also from the fig 2a the temperature affected the grain size and also gave it a shiny surface.

Based on the observation the reactivity of the alumina of fig 2 sintering temperature 1400°C, the alumina ceramics particles have an influence on the grain size of the microstructure and ensure homogeneous microstructure.

With the increase of the sintering temperatures there were a small reduction of the values of hardness and fracture toughness of this material and there was consequent reduction of the average grain size. This reduction is in the toughness and it resulted of the lesser way to crack propagation as shown in Fig 1, therefore it has a great reduction of the amount of grain boundaries (Santos, *et al.*, 2013).

The microstructure of the samples shows the effect of sintering temperature on the ceramics.

CHAPTER FIVE

5.0 CONCLUSION AND RECOMMENDATION

5.1 CONCLUSION

Conclusively, in this evaluation process the influence of the sintering temperature on the Al₂O₃ ceramic lead to reduction in grain size as the temperature was increased, and its influenced the properties of the ceramics under study. The results indicate that the temperature has influence in the densification of this material. Also, it has a soft reduction in the hardness and fracture toughness according to (Santos, *et al.*, 2013), related with the grain growth. Alumina having small grain size and high density yield good mechanical properties which are improved strength, hardness and wear resistance (Khan, *et al.*, 2016)

Fine grain size has high density of grain boundary that refines deformation of specimen in loading.

5.2 RECOMMENDATION FOR FURTHER RESEARCH WORK

This research work recommends sample A with regards the small grain size that indicates improved strength, hardness and fracture toughness. Further investigation may be carried out in the area of the effect of the additives on the mechanical properties.



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