Influence of Strontium Oxide on Mechanical Response of Zirconia Toughened Alumina

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Submitted by

Lovleen Kumar Bhalla
(Roll no. : 710cr1176)

Supervisor:

Prof. Debashish Sarkar

Department of Ceramic Engineering
National Institute of Technology
Rourkela

CERTIFICATE

This is to certify that the thesis entitled "Influence of Strontium Oxide on Mechanical Response of Zirconia Toughened Alumina" submitted by Lovleen Kumar Bhalla (Roll No: 710CR1176) in partial fulfillment of the requirement for the award of Master of Technology degree in Ceramic Engineering at National Institute of Technology, Rourkela is an authentic work carried out by him under my supervision and guidance. To the best of my knowledge, the matter embodied within this thesis has not been submitted to any other university/institute for the award of any other degree or diploma.

Dr. Debashish Sarkar
Dept. of Ceramic Engineering
National Institute of Technology
Rourkela-769008

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Lovleen Kumar Bhalla

DECLARATION

I declare that,

a. The work presented in this thesis is an original content of the research done by myself under the supervision of my supervisor.

b. The project work or any part of it has not been submitted to any other institute for any degree or diploma.

c. I have followed the guidelines prescribed by the Institute in writing my thesis.

d. I have given due credit to the materials (data, theoretical analysis and text) used by me from other sources by citing them wherever I used them and given their details in the references

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Lovleen Kumar Bhalla

Abstract

Zirconia toughened alumina (ZTA) is widely used in total hip arthroplastices. ZTA components are comprised of an alumina-rich composition where zirconia is evenly dispersed in the alumina matrix. Incorporation of strontia (SrO) in ZTA matrix is subject to enhance the fracture toughness and strength through development of elongated grains and cell proliferation *in vivo* also. In this perspective, the aim of this investigation was to study the effect of SrO on the physical and mechanical properties of ZTA. ZTA pellets were prepared with varying weight percentage of strontia as dopant. The pellets were uniaxially pressed at 5 ton force and then sintered at 1600^{o}C for 3 hours. The effects of SrO addition on properties such as density, microstructure, Vickers hardness, fracture toughness, compressive strength and tribology are studied. The SrO profoundly reacts with alumina and forms strontium aluminates at an optimum processing conditions. These strontium aluminates have elongated structure, which reduces the resultant grain size of matrix and enhancement of sintered density. This developed microstructure assists to improve the mechanical and tribological response of strontia doped ZTA composites.

Keywords : Alumina, Zirconia, Strontia, Mechanical Properties, Friction, Wear.

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Chapter 1

Introduction

Engineering ceramics have been used as components in orthopedic implants since the 1970s, when Boutin and Blanquaert (1981) [1] began to use an artificial hip joint comprised of alumina, Al_2O_3, in a 10 year study between 1970 and 1980. In the 1980s, alumina biomaterials underwent evolutionary changes in manufacturing technology, resulting in greater density, lower porosity, and increased fracture strength. Thus, the technology underlying both the composition and fabrication of contemporary high performance ceramics for orthopedic implants has evolved over the past four decades.

Zirconia, ZrO_2 was introduced in orthopedics because of its improved fracture toughness and mechanical strength relative to alumina. Zirconia owes its higher fracture toughness to a stress induced phase transformation from its metastable tetragonal phase to its stable monoclinic phase [2] at ambient temperatures.

ZTA components are comprised of an alumina-rich composition where zirconia is evenly dispersed in the alumina matrix. These ceramics exhibit superior strength and toughness compared to conventional alumina and zirconia, further detailed in this review. Ceramic composites thus represent a major new advancement of clinically available orthopedic biomaterials.

In this article, we concentrate on the advancements that have been made in understanding the performance of zirconia-toughened-alumina (ZTA) [3].

1.1 Composition and properties of ZTA

Zirconia toughened alumina (ZTA), is an alumina matrix composite ceramic, in which alumina is the primary or continuous phase (70–95%) and zirconia is the secondary phase (30% to 5%), is a material that combines the advantageous properties of monolithic alumina and zirconia. Under the condition that most of the zirconia is retained in the tetragonal phase, the addition of zirconia to alumina results in higher strength and fracture toughness with little reduction in hardness and elastic modulus compared to monolithic alumina ceramics. Currently, there are two commercially available ZTA biomaterials for hip arthroplasty applications: Biolox Delta by CeramTec Medical Products (Plochingen,Germany) and AZ209 by KYOCERA Medical (Osaka, Japan) [4].

1.2 Properties

ZTA composites have mechanical properties that are often better than monolithic alumina or stabilized zirconia. They achieve these properties by using several mechanisms: controlling the phase transformation in the zirconia particles, blocking crack growth by controlling grain shape, and strengthening the alumina phase itself through control of grain size and various additions.

1.2.1 Phase transformation

In zirconia, the stress-induced phase transformation from the metastable tetragonal phase to the monoclinic phase at ambient temperatures results in a 3–5% volume expansion and approximately 7% shear [5]. The induced volume change and strain oppose crack propagation, thereby improving the fracture toughness of the ceramic. This phase transformation may also lead to micro cracking, which enhances fracture toughness by effectively distributing the stress ahead of the main crack. However, micro cracking is beneficial only if it remains limited. extensive micro cracking will reduce strength. The theoretical mechanism expected to occur in commercial ZTAs is depicted below. The stress induced by the crack and the loss of constraint by the surrounding matrix leads to tetragonal-to-monoclinic transformation in the zirconia grain.

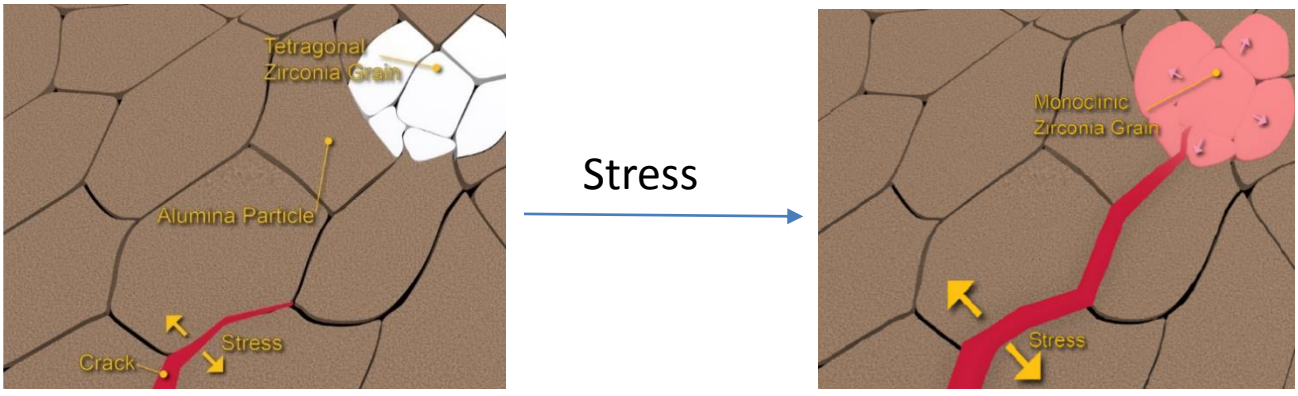


Figure 1.1: Example of phase transformation and crack propagation

1.2.2 Using platelets to block crack growth

An additional toughening mechanism in ZTAs consists of using platelet-like crystals to block or deflect crack growth. These crystals are depicted in both the KYOCERA Medical AZ209 and the CeramTec Biolox Delta technical documentation. The CeramTec and KYOCERA Medical formula utilizes strontium oxide crystals to enhance toughness and diffuse crack energy [6]. Addition of strontium oxide creates strontium aluminate composites, which form rod structures with higher crack propagation energy. These rods possess a maximum length of 3 μm and account for about 3% of the volume. Figure below illustrates the platelet toughening mechanism with the depiction of the Delta strontium aluminate rod. The frames in Figure depict crack propagation through alumina grains until the crack is deflected by the strontium aluminate rod. Incorporating multiple reinforcing mechanisms throughout the structure of the material makes the component more reliable because it becomes more effective in deflecting cracks closer to the surface and in avoiding fracture.

Chapter 2

Literature Review

2.1 Background

ZTA ceramics have lower density, higher hardness and a good compressive strength compared to metals, on the other hand these materials are brittle, this reduces their ability to withstand multiple impacts. Brittleness also facilitates propagation and coalescence of microcracks and may lead to the damage and comminution of ceramic due to impact and penetration.

For the design of advanced ceramic materials, the knowledge on the mechanical properties of ceramics is very essential [7]. Fracture toughness and hardness are two major parameters that manufacturers focus on. Fracture toughness shows the ability of ceramic to withstand the multiple impacts and crack propagations, while hardness affects erosion and plastic deformation of the impact. However, in femoral head design the dynamic properties of ceramic are more important. This is due to the difference in the behavior of materials in high strain rate compared to its behavior under a static load. Many approaches have been tried with the aim at improving the toughness of ceramics. One of them is the adding of zirconia into the alumina (ZTA) to give better properties compared to pure alumina. However, some additional additives are added to improve the properties of ZTA. ZTA is also very popular in total hip arthroplasties [8].

2.2. Consolidation

In the fabrication of advanced ceramics, it is important to get a high green density and defect free green body before sintering. Attaining a density close to theoretical is only possible after only attaining of high compact green body through judicious ceramic processing technique. The different consolidation techniques implemented for preparing green bodies are uniaxial dry pressing [9], cold isostatic pressing (CIP), slip casting, gel casting (thermal and non-thermal) and tape casting. In the present work, consolidation has been done by uniaxial pressing.

2.3 Sintering

Sintering is a processing technique used to produce materials with controlled density and microstructure by applying high temperature below its melting point [10]. The main sintering techniques used for fabricating advanced ceramics are hot pressing, hot isostatic pressing (HIP), spark plasma sintering (SPS), vacuum sintering, and microwave sintering. It is also observed in the literature that atmospheric sintering is successfully used for the fabrication of ZTA ceramics.

2.4 Mechanical Response

Research by Coble, Wang et al. [11], and Rittidech et al. [12] indicated that the presence of MgO in the Alumina matrix significantly affect the mechanical properties of bulk Alumina. The addition of small amounts of MgO (0.25 wt.%) enable the Alumina to sinter to near-theoretical density [6]. Wang et al. [13] showed that MgO addition can efficiently improve the sinterability of Alumina. The Alumina-magnesia ceramic system has higher Vickers hardness values compared to the conventional alumina ceramic system because MgO reduces the grain size.

Azhar et al. [14] concluded that the introduction of fine MgO particles into ZTA will increase its Vickers hardness and wear-resistance properties. Besides hardness, fracture toughness is also important so that a cutting insert will be able to perform without experiencing premature failure [15]. Tsukuma and Shimada [16] found that fracture toughness and hardness were very dependent on the amount of Cerium Oxide added into tetragonal zirconia polycrystals (TZP).

Work under taken by Azhar et al. [17] showed that the addition of MgO increases the Vickers hardness up to 1740 HV. However, the fracture toughness of the ZTA composite decreased with further addition of MgO due to a finer Alumina grain size. The decrease in fracture toughness causes the ZTA insert to become vulnerable to failure during the machining process.

2.5 Improving Hardness

Yuanlong Li, Hyoun-Ee Kim and Young-Hag Koh [18], reported on how to increase the surface hardness of ZTA composites by simple surface treatment with boehmite sol. This treatment improved the hardness significantly, with little decrease in flexural strength and fracture toughness.

2.7 Toughening mechanism

The two major mechanisms that were playing role in the toughening the ZTA were stress induced transformation and microcrack toughening [19]. In transformation toughening, once the cracks travel, the stress concentration at crack tips can change zirconia from tetragonal to monoclinic phase. The phase change resulted in volume expansion around the crack tip and the neighboring grains became compressed thereby impeding the crack propagation. To overcome such compressive stress, more energy is required to propagate the cracks. A combination of grain reduction and phase transformation could toughen ZTA matrix. The transformation is the cause of crack deflection, with increasing the percentage of Zirconia in the specimen. Microcracks were generated due to the volume expansion in transformation of tetragonal to monoclinic phase in the cooling part of sintering. Microcracks extended

in the stress field or affected the crack growth, which could absorb the fracture energy.

Although these hardness and fracture toughness measurements are considered to be essential parameters that provide basic knowledge on the behavior and failure mechanisms of the ZTA samples, they are only limited to the static behavior of the sample.

The indentation fracture toughness (K_{IC}) was derived from the average crack length. For a ratio c/a >2.5 (present study), where c is the crack length and a is the half diagonal length of the indentation impression [20], K_{IC} is calculated using the following equation:

$$K_{IC} = 0.0752.P/c^{3/2}$$

2.6 Tribological Response

The volume expansion (~4%) caused by tetragonal to monoclinc phase transformation exerts some stress to the neighboring grains. The accumulated stress fields lead to surface roughening, interconnected surface and subsurface cracks (defects) resulting increased friction, grain pull-outs and ultimately fast failure of the material [21]. To improve the wear properties, Kamiya et al. [22] synthesized

particulate (Al_2O_3 or SiC) reinforced ZTA and reported lowest wear rate when tested under collision (erosive wear test). In a different study, He et al. [23] Observed a mild wear with a very low wear rate of 2×10^{-8} mm^3 /N m for ZTA under reciprocal dry sliding (against stainless steel plate) at contact pressure of 300 MPa. Different investigators studied these composites with various types of oxide added in small amounts with the intension of modifying microstructures, improving mechanical properties, chemical stability.

2.7 Objective

The objective of this work is to study the effects of strontia on mechanical behavior of Zirconia toughened alumina.

- To determine optimum SrO content is Zirconia toughened Alumina as dopant.
- To study the effect of SrO on density, grain size of Zirconia toughened Alumina.
- To study the mechanical response of SrO doped Zirconia toughened Alumina.
- To study of tribological behavior of SrO doped Zirconia toughened Alumina.

Chapter 3

Experimental Procedure

3.1 Batch Preparation

In this experiment different specimen samples were prepared with following raw materials:

1. Alumina
2. Zirconia
3. Strontium nitrate

The composition of different samples with their indicator names is given in the table below:

Table 3.1: Specimen composition

Sample no.	**wt.% Alumina**	**wt.% strontia**	**Wt.% zirconia**	**Indicator Name**
1	100	0	0	A
2	99.5	0.5	0	AS
3	99.25	0.75	0	AS_1
4	99	1	0	AS_2
5	94.5	0.5	5	AZ_1S
6	89.5	0.5	10	AZ_2S

3.1. Characterization of powders

3.1.1. X-Ray Diffraction analysis

Phase analyses of the calcined powders were executed by x-ray diffraction (XRD) using Cu Kα radiation in Phillips PANalytical (Model: PW 1830diffractometer, Netherland).The XRD arrangement was furnished with one-dimensional compound silicon strip detector for high quality diffraction data. The powder samples were placed on a non-diffracting sample holder positioned in the Bragg-Brentano diffractometer setup. The calcined powder obtained from different calcination temperature and different powder synthesis processes were analyzed.

3.2. Pellet preparation

The important process parameters of the pellet preparation are:

1. Addition of binder
2. Type of compaction
3. Heating rate
4. Type of sintering

The binder used was 3 wt. % PVA (poly vinyl alcohol) solution. Different batches of powder were filled into a circular die made with carbon steel and uniaxially

pressed at 5 ton force. The dwell time for pressing was 120 seconds. During pressing of ceramics, the three stages are:

1. Rearrangement
2. Deformation
3. Fragmentation

3.3 Sintering of Compacts

Sintering is a processing technique to produce materials with controlled density and microstructure by subjecting the pre-consolidated green body to higher temperature below its melting point.

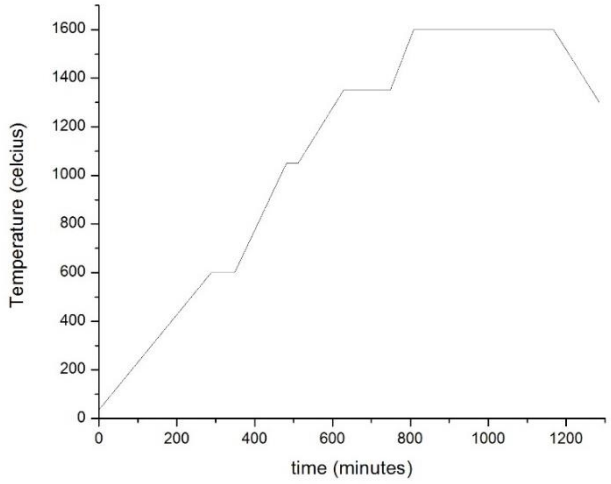


Figure 3.1: Sintering Profile

The driving force for sintering is the reduction in the free energy of the system. Both densification and coarsening result is a reduction in free energy of the system.

Sintering is a competitive process of grain growth and densification.The sintering profile is described in Figure 3.1.

3.4. Characterization of compacts

3.4.1 Density

The bulk density and apparent porosity of the pellets was measured using Archimedes principle. The following formulae were used:

Bulk Density = $\frac{D}{W-S} \times \rho$

Apparent porosity = $\frac{W-D}{W-S} \times \rho$

Where,

D = Dry weight

W = Wet weight

S = Suspended Weight

ρ = density of liquid

3.4.2 Microstructure

The microstructures of the sintered sample were taken under Field Emission Scanning Electron Microscope (FEI Nova NanoSEM450) system. The electron gun having a field-39 emission cathode delivers precise probing beams at low and high electron energy, bringing about both superior spatial resolution, minimum sample charging and damage during operation.

3.4.3 Grain Size

The average grain size was mesured by taking averages of grain sizes appearing in the FESEM micrograph.

Mean grain size = $\frac{1}{n}\sum_{i=1}^{n} x_i$

Standard deviation = $\frac{1}{n-1}\sum_{i=1}^{n}(x_i - \bar{x})^2$

3.4.4 Hardness

The formula for the Vickers hardness is given by the following equation

$$HV = \frac{1.8544F}{d^2}$$

Where,

F = applied load (3 Kgf)

d = mean diagonal of indentation

3.4.5 Fracture Toughness

The fracture toughness is also calculated from the Vickers indentation by the formula:

$$K_{IC} = 0.0752.P/c^{3/2}$$

Where,

c = crack length

p = load applied

3.4.6 Compressive strength

In the study of strength of materials, the compressive strength is the capacity of a material or structure to withstand loads tending to reduce size. It can be measured by plotting applied force against deformation in a testing machine. Some materials fracture at their compressive strength limit; others deform irreversibly, so a given amount of deformation may be considered as the limit for compressive load. The compressive strength was measured using universal testing machine.

3.4.7 Brazilian Disk Test

Tensile strength formula = $2p/\pi dt$

Where,

p = maximum applied load

d = diameter of specimen

t = thickness of sample

3.4.8 Friction/ wear

In materials science, wear is erosion or sideways displacement of material from its "derivative" and original position on a solid surface performed by the action of another surface.

The wear test was performed using Ball plate tester.

Test parameters:

Sliding time = 2 hours

Normal Load = 15 N

Speed = 30 rpm

Track Diameter = 2 mm

Chapter 4

Results and Discussion

4.1 XRD Analysis

The composite XRD pattern of different weight % strontia doped in alumina matrix has been shown in Figure 4.1. The relative volume percentages of alumina, strontia and strontium aluminate has been calculated from XRD and tabulated in Table 4.1.

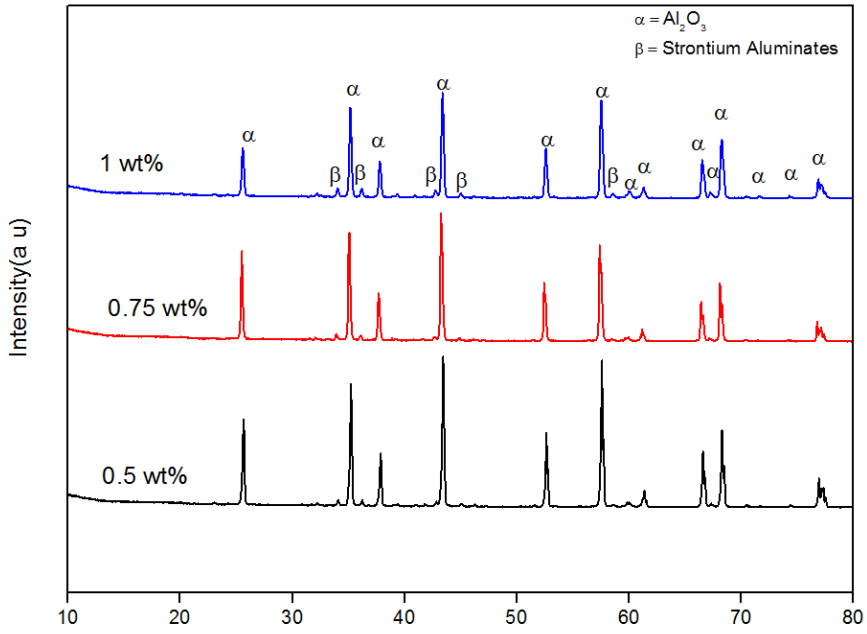


Figure 4.1: Composite XRD pattern of varying wt. % strontia doped alumina

Table 4.1: Relative vol. % of Alumina, Strontia and Strontium Aluminates after sintering

Sample	Vol. % Alumina	Vol. % Strontia	Vol. % Strontium aluminate
AS	98.825	0	1.175
AS_1	86.827	12.368	0.805
AS_2	87.09	11.78	1.13

From the above XRD data, it is observed that SrO remains unreacted when added in excess of 0.5 wt. %.

4.2 Density

The calculated value of bulk density and apparent porosity has been tabulated in Table 4.2.

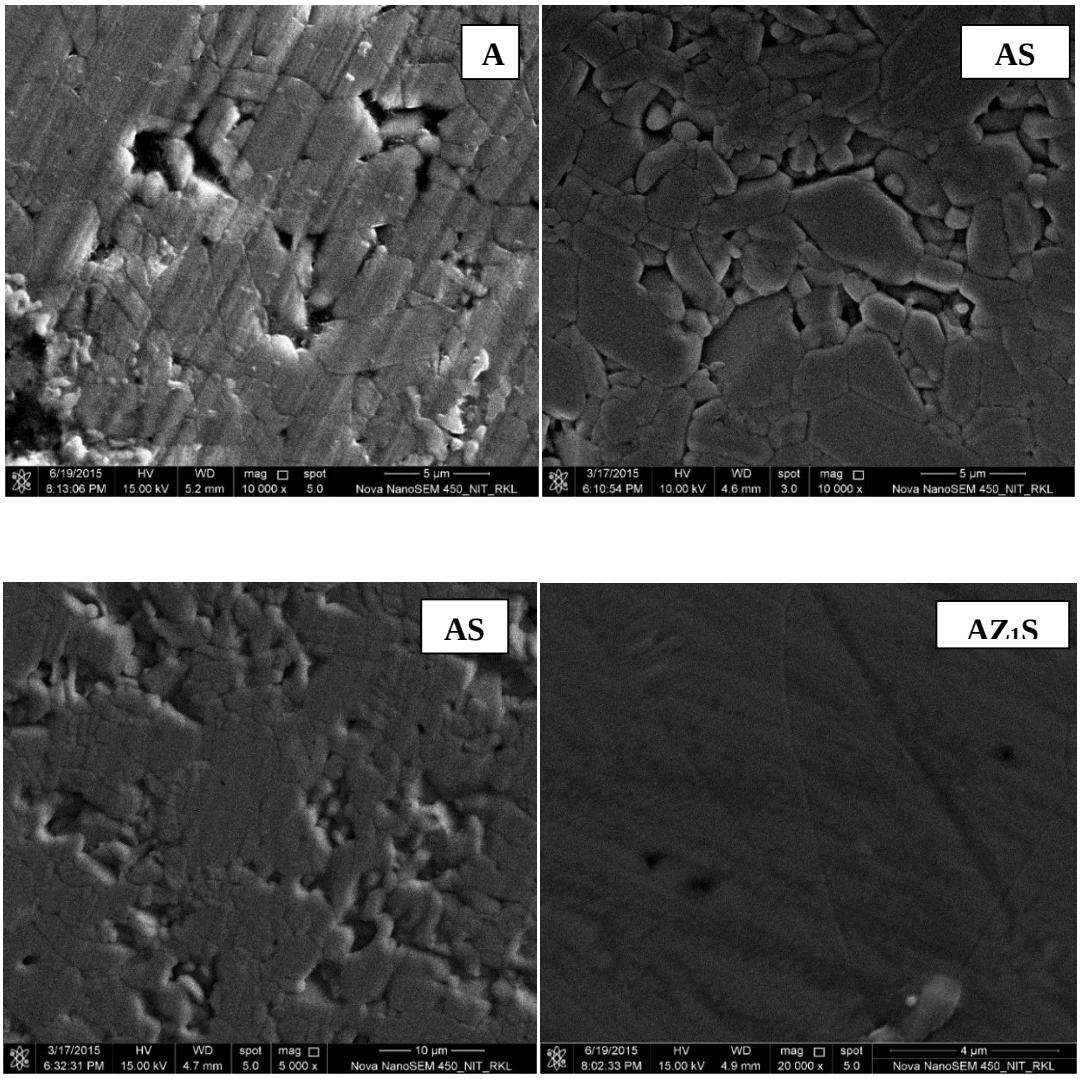
Table 4.2: Density Measurements

Sample no.	Indicator name	Bulk Density (g/cm^3)	Apparent Porosity (%)
1	A	3.07	0.8
2	AS	3.62	0.9
3	AS_1	3.53	0.85
4	AS_2	3.59	0.82
5	AZ_1S	3.85	0.7
6	AZ_2S	3.93	0.67

At the beginning, the density of Alumina sample increased from 3.07 g/cm^3 (0 wt.% SrO) to 3.93 g/cm3 (0.5 wt. % SrO), showing a 2.08% increase with the addition of SrO. Among them, the sample with 0.5 wt. % SrO and 10 wt. % ZrO_2 had the highest bulk density value (3.93 g/cm^3). SrO has the ability to inhibit the abnormal grain growth of Al_2O_3 due to a microstructure pinning effect, thus resulting in higher density.

4.3 Microstructure

The sintered specimens prepared were subjected to microstructural analysis. The following figure below shows the FESEM micrographs of the indicator specimens.



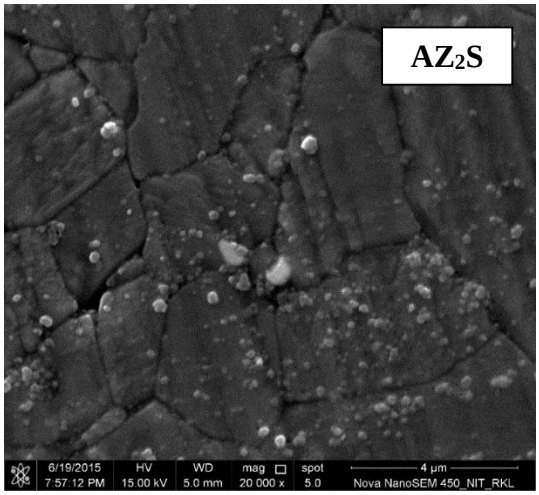


Figure 4.2: FESEM micrographs of A, AS, AS_1, AZ_1S, AZ_2S specimens

4.4 Grain Size

The average grain size calculated from the FESEM images are tabulated in Table 4.3. It can be observed that with the addition of zirconia and strontia, grain size increases.

Table 4.3: The calculated grain size of different specimens

Sample no.	Name	Average(μm)	Standard deviation
1	A	2..39	2.16
2	AS	2.75	1.58
3	AS_1	2.69	1.05
4	AS_2	2.57	0.95
5	AZ_1S	2.23	1.56
6	AZ_2S	2.30	1.08

4.5 Mechanical Properties

4.5.1 Hardness

The Vickers test conducted gave the hardness following the equation in Section 4.4.4. The hardness value of the specimens has been tabulated in Table 4.4. The following table shows the hardness of specimens prepared

Table 4.4: Values of Hardness for different specimens

Sample no.	Name	Vickers Hardness(HV)
1	A	1207
2	AS	1274.6
3	AS_1	1248
4	AS_2	1260
5	AZ_1S	1421.9
6	AZ_2S	1658.6

With the addition of strontium oxide, the hardness increases. On addition of zirconia the hardness increases because zirconia toughens the alumina matrix.

4.5.2 Fracture Toughness

The fracture toughness has been calculated using the equation in Section 4.4.5. Table 4.5 represents the values of fracture toughness.

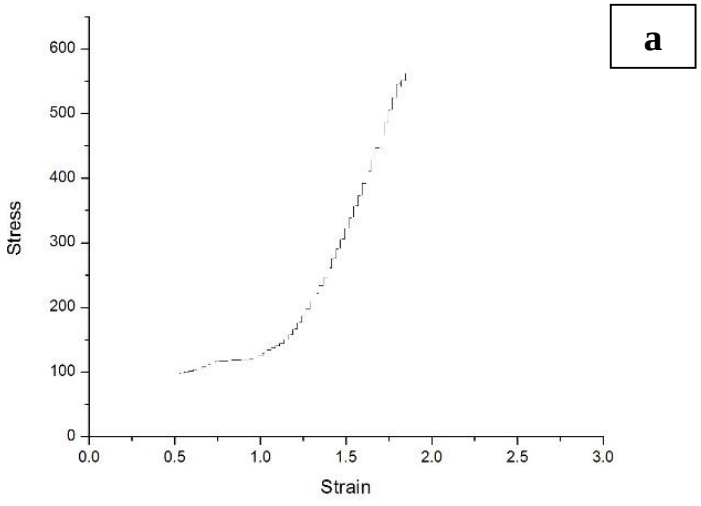
Table 4.5: Fracture toughness values of the specimens

Sample no.	Name	Fracture toughness (MPa $m^{1/2}$)
1	A	3.80
2	AS	3.95
3	AS_1	3.01
5	AZ_1S	4.32
6	AZ_2S	4.45

With the increase in strontium oxide content, the fracture toughness of the specimens increases.

4.5.3 Compressive strength

The compressive strength of the following specimens has been carried and the plot of stress vs. strain curves is shown in the figures below.



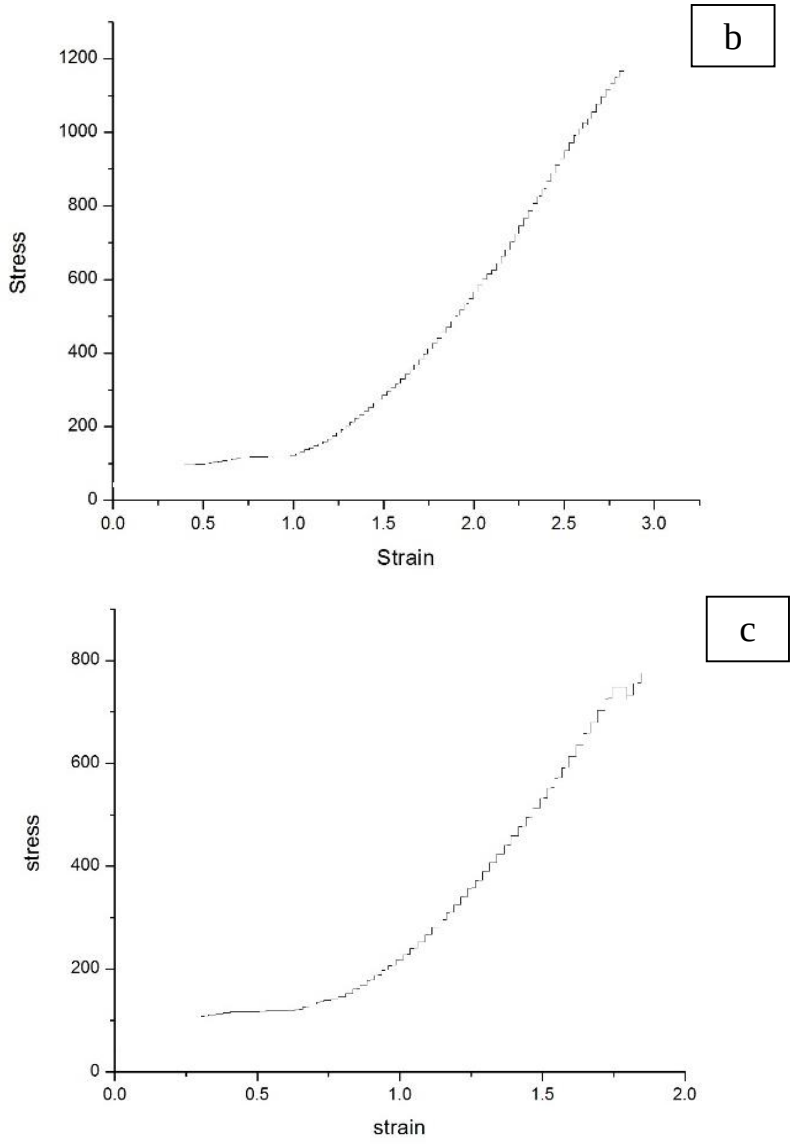


Figure 4.3: Stress vs. Strain plot for (a) A, (b) AZ_1S and (c) AZ_2S specimens.

The following table summarizes the Ultimate compressive strength observed for specimens A, AZ_1S and AZ_2S, respectively. The maximum compressive strength is obtained for composition 94.5 wt. % Alumina, 5 wt. % Zirconia and 0.5 wt. % strontium oxide.

Table 4.6: Ultimate compressive strength

Sample name	Compressive Strength(MPa)	Maximum Strain
A	686	1.846
AZ_1S	1178.6	2.832
AZ_2S	786	2.531

The secondary phases play significant roles in affecting the mechanical properties of ceramic composites. The ceramic composites were shown to have maximum hardness, fracture toughness and compressive strength when the strontium aluminate phase was at its highest percentage. As a conclusion, compared to other compositions, ZTA ceramics system with 0.5 wt.% SrO has the optimum properties.

4.8. Friction/Wear

4.8.1 Friction

The following plots below shows the coefficient of friction vs. sliding time for different specimens (**Figure 4.7 - 4.9**).

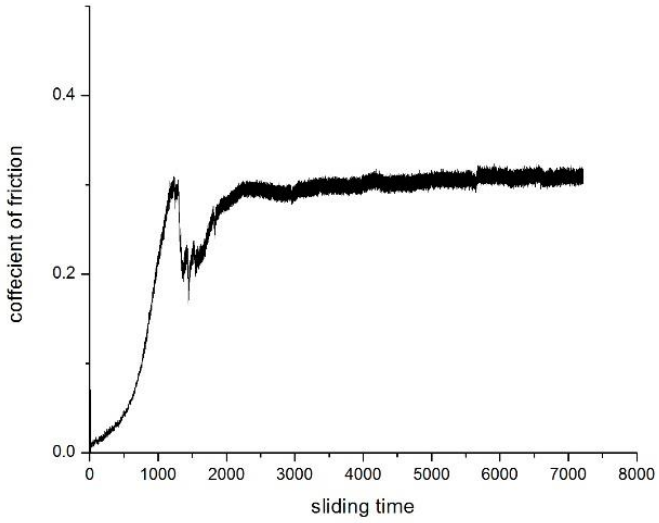


Figure 4.4: Coefficient of friction vs. sliding time for specimen A

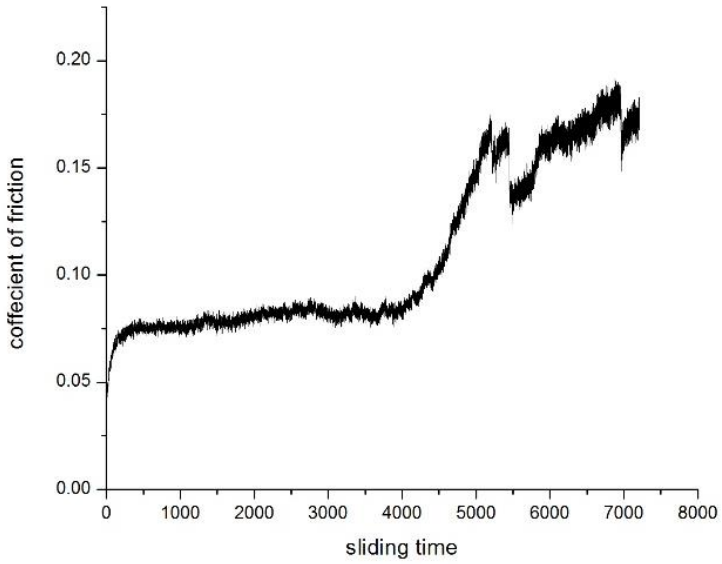


Figure 4.5: Coefficient of friction vs. sliding time for sample AZ_1S

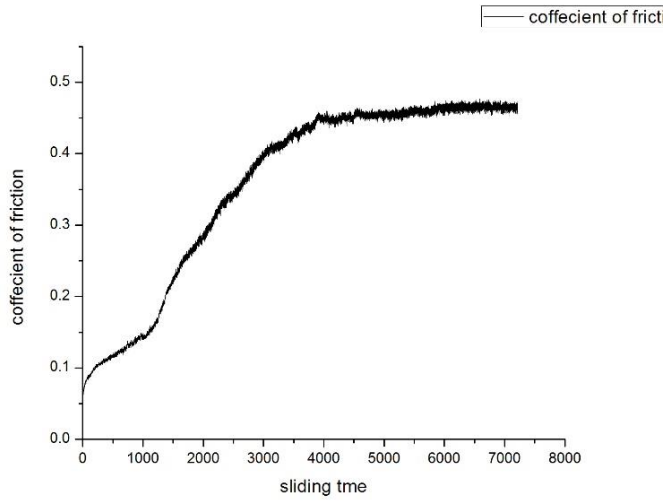


Figure 4.6: Coefficient of friction vs. sliding time for sample AZ_2S

The coefficient of friction is lowest for 94.5 wt. % Alumina, 5 wt. % Zirconia and 0.5 wt. % strontium oxide.

4.8.2 Wear depth

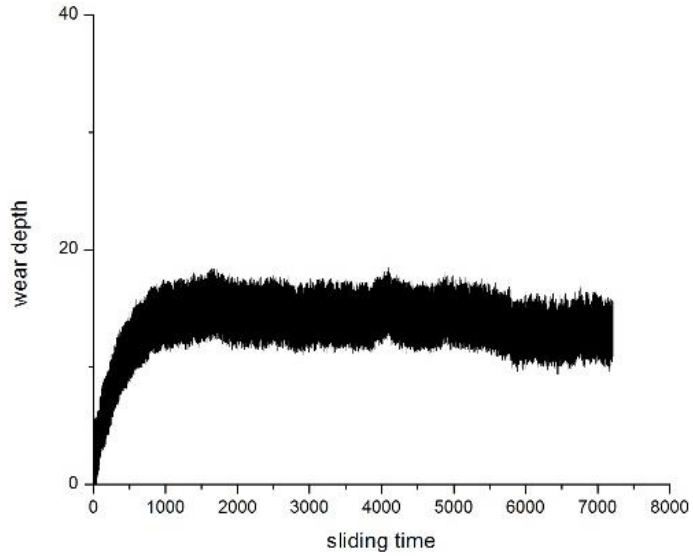


Figure 4.7: Coefficient of friction vs. sliding time for sample A

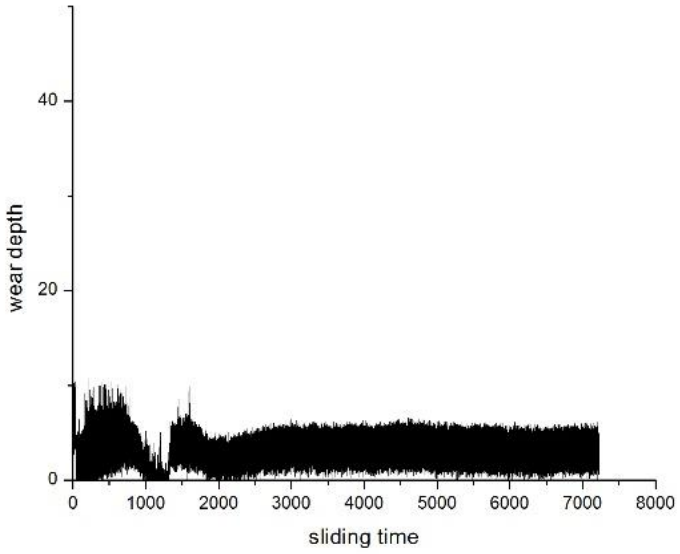


Figure 4.8: Wear depth vs. sliding time sample AZ_1S

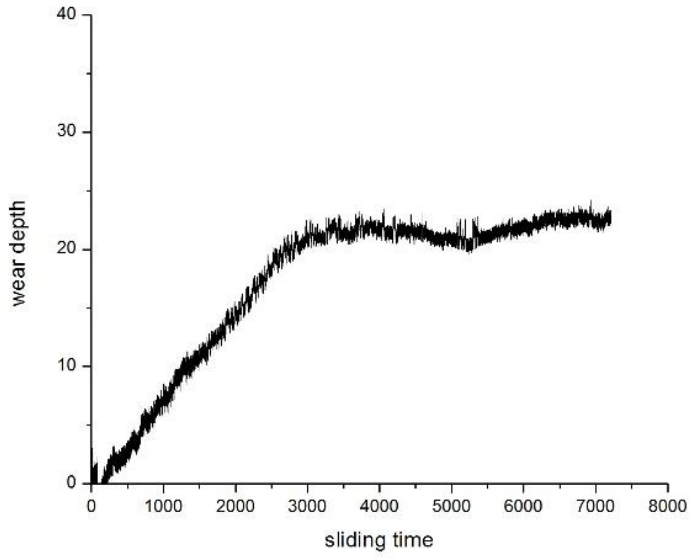


Figure 4.9: Wear depth vs. sliding time sample AZ_2S

Table 4.7: Summary of friction/wear test

Sample name	**Wear width track (μm)**	**Coefficient of friction**
A	183.16	0.30
AZ_1S	99.29	0.18
AZ_2S	178.5	0.46

The lowest value of coefficient of static friction (μ= 0.18) is obtained for AZ_1S and the highest value of coefficient of static friction (μ = 0.46) is obtained for AZ_2S after the wear test. The value of coefficient of static friction for other composite A is calculated to be 0.3. Thus from the results obtained, it is evident that AZ_1S, which contains SrO, shows a very low wear rate and also a low value of coefficient of static friction among all the composites of different composition.

Chapter 5

Conclusion

5.1 Conclusions

1. An optimum amount of 0.5wt% SrO is found beneficial to achieved elongated grain of strontium aluminate and sintered density of ZTA. Unreacted SrO beyond the optimum amount degrades the both mechanical and tribological responses.
2. Due to pinning effect of SrO on ZTA, the grain size decreases, which causes increase in density. SrO doped ZTA exhibits relative density of 98 %.
3. Strontium oxide increases the hardness and fracture toughness of alumina due to the formation of elongated grains, which absorbs the crack propagation energy. The maximum hardness obtained is 1658 HV. Maximum value of fracture toughness is 4.7 MPa $m^{1/2}$.
4. The compressive strength also increases with addition of Strontium oxide because of reduced grain size. The maximum compressive strength is 1178.6 Mpa.
5. Coefficient of friction and wear track width decreases with the addition of Strontium oxide. Minimum coefficient of friction is 0.18 and minimum wear track width is 99.29 μm.

Chapter 6

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