A comparison of mechanical properties of zirconia ceramic calcined at different temperatures with zirconia glass ceramic composite

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Abstract: Glass-ceramics have higher crystallinity, low porosity, high strength, toughness, translucency, stable at high temperature, machinability, bioactivity and biocompatibility. These properties can be controlled by base-glass composition and also by controlled heat treatment. So that the purpose of this study was to figure out the role of zirconia content on crystal structure and mechanical properties.

The goal of this work was to evaluate the mechanical behaviour of zirconia ceramic calcined at various temperatures with zirconia-glass ceramic composite. Difference in mechanical properties of same composition, calcined at various temperature was observed because of the difference in microstructure. A composite material containing zirconia and glass ceramic was prepared and tested for its microhardness. It was concluded that they had excellent mechanical properties than zirconia.

Keywords: Calcination, Mechanical properties, Ceramic, Composite.

I. INTRODUCTION

Zirconia(ZrO_2) present in solid state having mono-clinic crystal structure at room temperature. Transformation occur at 1100°C to tetragonal form, with 3 to 9% volume contraction.Zirconia transformed into cubic structure above 1490°C. The melting point of zirconia is 2700°C[1].

Applicability of zirconia is mostly in its 'stabilized' state, by heating, zirconia undergoes phase modifications which can be disruptive or not useful for mechanical properties. Phase changes can be stopped by adding some stabilizer like yttria, and the resulting material will have greater thermal and mechanical properties [2].

Zirconia-glass ceramics are bio-compatible, chemically inert and having good esthetic appeal, that's why mostly used in dental application and prosthetic restoration. But the main drawback is that they have low fracture toughness and low tensile strength [3].

There are various mechanism responsible to enhance the mechanical properties of glass–ceramic like compressive stress reinforcing mechanism, transformation toughening and bridging mechanism [4].

In this study, the role of calcination temperature and zirconia on mechanical properties was also investigated. Zirconia(ZrO2) shows three crystalline phases at various temperature. It shows monoclinic at lower temperatures, tetragonal phase between 1000°C and 2370°C and cubic phase in between the melting point temperature i.e 2370°C and 2700°C. However, the various phases and the phase transformations may be advantageous to develop ceramics with Superior mechanical properties [5].

Glass–ceramics found limited in application because of their poor mechanical properties i.e micro-cracks or flaws are expected to be generated in the laboratory during synthesis or fabrication and these flaws can later lead to clinical failure. This can be avoided by adding a second crystalline phases such as fibers and particles to the glass matrix. Zirconia particles are widely utilized in glass matrix as the second crystalline phase to enhance their mechanical properties [6].

The morphology of the powder is strongly influenced by calcinations temperature. This modification are due to changes of specific surface area, crystallite size, pore size, degree of crystallinity, phase composition and shrinkage rate during calcinations [7].

The flexural strength of composites material reinforced by micro-sized zirconia is reported to be somewhat higher than nano-sized zirconia composites. It was due to the homogeneous distribution of micro-sized zirconia as compared to the agglomerated nano particles. On another side toughness values of nano-sized samples have been reported to be slightly more than other micro-sized zirconia and glass-ceramic samples. And this has been attributed to toughening mechanism involving deflection and pinning of micro cracks [3].

II. EXPERIMENT

A. Preparation of sample

Four sample were prepared for this study. Three sample were of nano yttria stabilized zirconia. These powders were obtained through a method described in another work [8]. The powders were calcined at various temperature viz. 700,800 and 900°C.The fourth composition was prepared by using zirconia and silica glass(33.33 wt % zirconia).

B. Powder compaction

Powder was compacted using a steel die and punch into a disc of 10 mm in diameter with varying thickness. die and the punch assembly are shown in figure 3.1. To obtain compaction, stearic acid solution was used for lubrication of the die and punch wall and Poly vinyl alchohol(PVA) solution was used as binding agent to form green compact.

Fig 3.1: Compaction die along with mortar and pestle

C. Powder pressing

A uniaxial press with a maximum available load of 400 kg.cm^{-2} was used to obtain the disc shape sample. A 2 gm of powder was poured in a steel die of 10 mm in diameter to get disc like samples.20 kg.cm^{-2}load was used for 15 s.Figure 3.2 show the uniaxial press machine.

Fig 3.2: Uniaxial pressing for compaction

D. Sintering

Sintering is the process of densifying a material by heating it up to liquefaction temperature without melting it.

Sintering temperature (1450^{0}C) and holding time (6 hours) was selected based on literature to ensure densification.

T1=900^{0}C at 3hr

T2=1300^{0}C at 3hr 20 min

T3=1450^{0}C at 2hr 30 min

T4=1450^{0}C at 6hr

T5=1000^0 C at 1hr 30 min

T6=200^{0}C at 1hr 20 min

E. Density measurement

The geometrical measurements of the sample were done by Vernier caliper having 0.001mm least count. The mass was measured by maintaining 0.1 mg accuracy. The density measurement was done using the ratio mass/volume for both green body and sintered samples. The density of the disc was also measured using Archimedes principle.

F. Mounting and polishing

Cold Mounting and polishing for Vickers hardness calculations were done by using Acrylic repair material.

Figure 3.3 shows the material used for mounting the samples and mounted samples.

Fig 3.3: Mounting material and mounted samples

Sand paper of grit size C200 was used for primary polishing of samples and then mirror finishing were achieved using emery paper of grade P500, P1000, P1500 and P2000 micron in successive polishing. Figure 3.4 shows the Duel head polishing machine.

Fig 3.4: Duel head polishing machine

G. Hardness measurement

Vicker hardness were measured using standard diamond indenter as shown in figure 3.5 at two different loads of 1kgf and 2 kgf. To obtain higher accuracy average of three measurements for each load were calculated.

Fig 3.5: The Vickers Hardness tester

Optical Olympus microscope was used to analyze the indented part of the surface. Figure 3.6 shows the profile of the indentation. It clearly depict that the indentation left the two diagonals named d_1 and d_2 on surface of the sample. Mean values of d_1 and d_2 was used in hardness calculation.

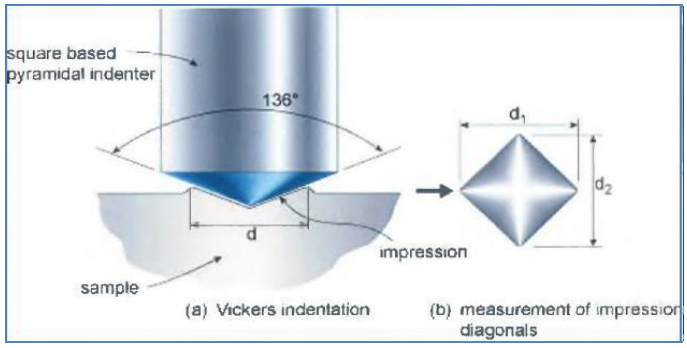


Fig 3.6: The profile of the indentation[2]

The Vickers hardness was measured using the following relation.

$$H_V = \frac{2 \sin 136^\circ}{D^2} \approx 1.845 \frac{F}{D^2} \quad (1)$$

Where:

Hv: is the Vickers hardness in ($kgf.mm^{-2}$)

F: is the load in (kgf)

D: is the arithmetic mean of two diagonals, d_1 and d_2 in (mm)

H. Scanning electronic microscopy (SEM)

A scanning electron microscope(SEM)was used to observe the grain boundaries of the sintered samples (in backscatter mode). To obtain nice SEM image real surface of sintered sample were etched with acidic solution to get clear view of the grain boundaries.

EDS analysis was also done to know the elemental identification and quantitative composition of the material.

I. Fracture morphology

Fracture morphologies of indented samples were observed under optical microscope having 100X to 1000X magnification range.

III. Results and Discussion

A. Density and Sintering behavior

Figure 4.1 shows the density graph for the green and sintered samples. It was noticed that density of sintered sample is greater than green samples, in that sample calcined at 800^0C had the highest density which is 4.898 g/cm^3.

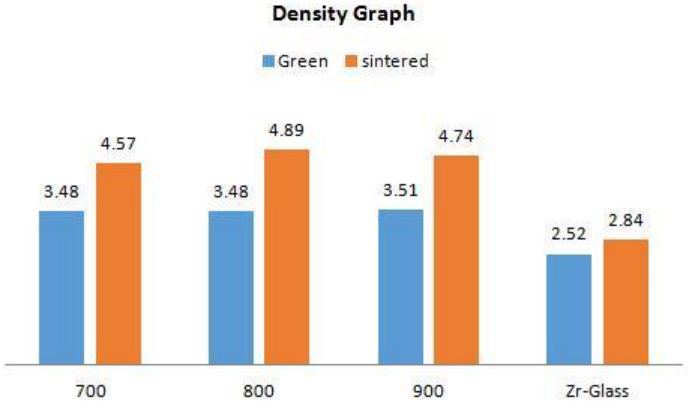


Fig 4.1: Green and sintered densities of the samples.

Density results shows that zirconia-glass ceramic had high porosity as compared to other zirconia samples.

Shrinkage differences after sintering of the sample which was calcined at different temperature are shown in figure 4.2. Sample sintered at 800^0c has got highest shrinkage rate as compared to other two.

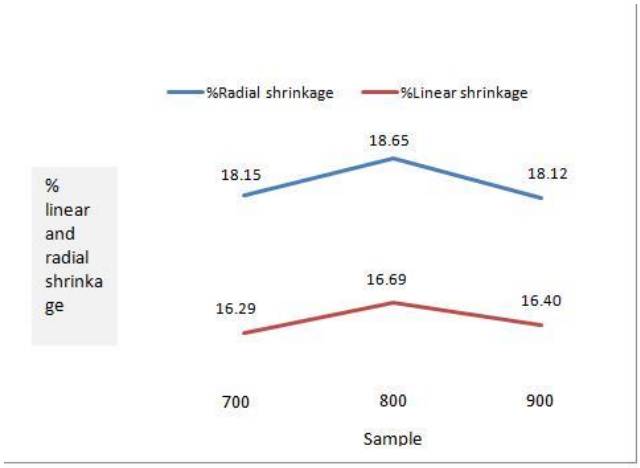


Fig 4.2: Percentage Linear and Radial Shrinkage after sintering of zirconia samples

A summary of the main properties are shown in Table I

TABLE I. Density and Vickers' hardness measurements

Composition	Green density (g/cm^3)	Calcination temperature(^{0}C)	Sintering temp(^{0}C)	Sintered density (g/cm^3)	Indentation load (kgf)	Vickers hardness (HV)	Avg. Crack length (μm)
ZrO_2	3.48	700	1450	4.57	1,2	75.80	87.05
ZrO_2	3.48	800	1450	4.89	1,2	114.4	75.19
ZrO_2	3.51	900	1450	4.74	1,2	35.99	85.53
ZrO_2-glass ceramic	2.52		1450	2.84	1,2,3,5	254.2	No Crack

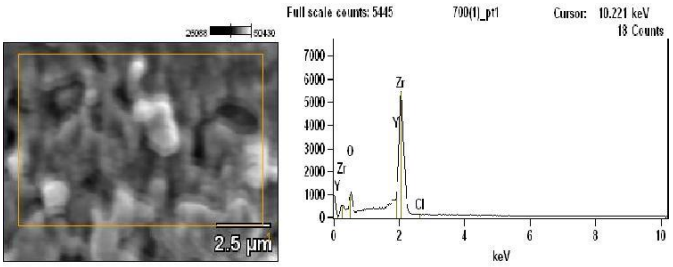
B. Micro structural analyses

SEM images reveal that sample calcined at 800^{0}C shows finer grain size, where as another two sample calcined at 700^{0}C and 900^{0}C has coarser grain size.

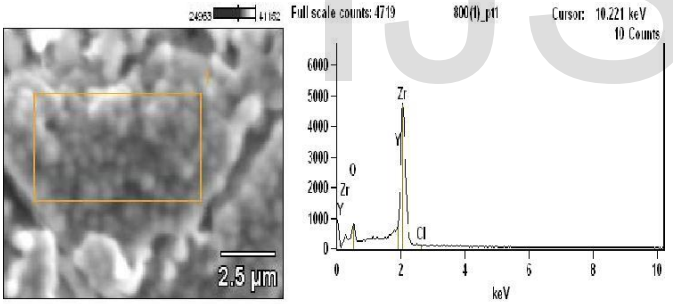
It seems that the uniform distribution of zirconia in glass matrix in zirconia-glass sample.

EDS analysis reveals the amount zirconia, yattria and silica contents in the specimens.

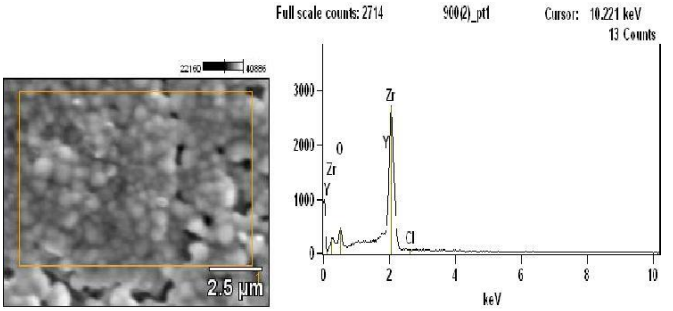
Following images shows the microstructure of sintered samples.



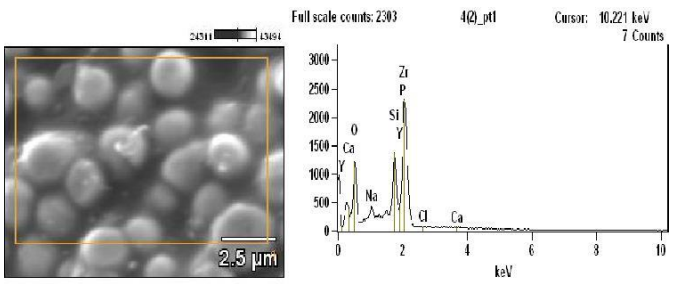
Sintered zirconia that was calcined at 700^{0}C



Sintered zirconia that was calcined at 800^{0}C



Sintered zirconia that was calcined at 900^{0}C



Sintered Zirconia-glass ceramic

C. Vickers hardness

Calcination temperature causes the resultant microstructure which is responsible for mechanical properties. Figure 4.3 shows the Vickers hardness data for all sintered samples.Vickers hardness of sample calcined at 800^{0}C was more as compared to other two calcined sample, but hardness of zirconia glass was too high as compared to remaining all sample.

Hardness values are summarized in table 4.1

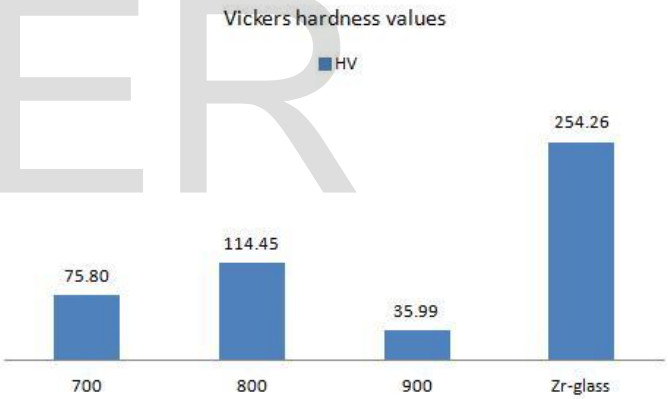
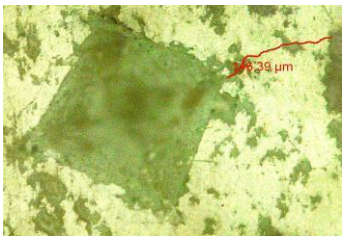


Fig 4.3: Vickers hardness of sintered zirconia and sintered zirconia glass-ceramic

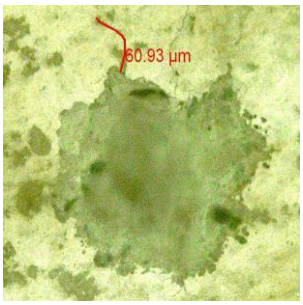
D. Fracture morphology

It was noted that cracks were obtained in all sample except zirconia glass sample. The sample calcined at 800^{0}C show minimum average crack length as compared to others and it is mentioned in table 4.1.

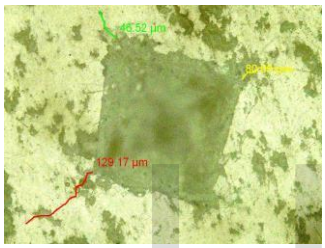
Following images was captured at 2 kgf load, which shows the crack generated in samples.



Sample calcined at 700



Sample calcined at 800



Sample calcined at 900

Zirconia glass was indented at 1kgf, 2kgf, 3kgf and 5kgf load, but still it was not possible to generate crack.

IV. Conclusion

The objective of this work was to study comparatively the mechanical properties of zirconia ceramic calcined at different calcinations temperatures with zirconia-glass ceramic composite.

The conclusions from this work are as follows.

The microstructure of zirconia-glass sample shows that the distribution of zirconia grains in silica matrix was uniform.

Microstructure of sample calcined at 800^0C show fine grain size as compared to other samples.

Hardness value, fracture resistance and density of sample calcined at 800^0C was higher as compared to other samples.

It was not possible to generate crack in zirconia-glass sample even at tests done at 1kgf, 2kgf, 3kgf and 5kgf load. In pure zirconia samples, there is crack formation at both 1kgf and 2kgf load.

On the basis of density results, it was conclude that zirconia-glass sample is much porous as compared to other three samples.

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