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Research Article

Investigation effect of nano ZrO2 contents and temperature on fracture toughness of in-ceram zirconia dental materials

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ABSTRACT

Objective: This study describes a general factor-based experiment that applied to researching and investigating on hardness and fracture roughness of in-ceram zirconia both with and without nano zirconia reinforcement. The aim of the study was to evaluate the impact of factors such as the Nano zirconia contents and sintering temperature. **Methods:** Vickers microhardness method for and the fracture toughness (Indentation Strength) was measured on 5 specimens for each ceramic. SEM and optical microscope have used to measure the volume fraction of each phase, the dimensions, and shapes of the grains and the crack pattern. Experimental design (general factorial method) has used for running experiment. DX7 software used for all statistical analysis. **Results:** Results show that the nano zirconia improves fracture toughness and hardness, but by increasing, the percentage of nano zirconia this effect will be inverse and the fracture toughness of samples reduced.

Keywords: In-ceram, Nano zirconia, fracture toughness, hardness

INTRODUCTION

Ceramic biomaterials such as alumina and zirconia are historical materials that they were anticipated to function as an inert material in the body especial in dental healthcare [1]. The problem of stained, decayed, and terminally malodorous dentures by using these materials have been solved [2]. From these bioceramics, zirconia offers so far the best mechanical properties [3].

Zirconium oxide (ZrO2) is a mineral that is found in nature in the form of baddeleyite (zirconium earth) [4]. It features a Mohs' hardness of 7-9 and – similar to aluminium oxide – is suitable for producing abrasive discs. It has also proved its reliability for domestic and industrial cutting instruments. Due to its high strength and fracture toughness, it is also suitable for the production of hip prostheses. It has also used as opacifier for metal ceramic opaque materials [5]. For this purpose, however, synthetically produced ZrO2 used, since the natural material exhibits excessive contamination [6]. Yttria-tetragonal zirconia polycrystals (Y-TZP) successfully applied as a dental materials with long term prosperous in the orthopedics field. The flexural strength of zirconia is from 800 to 1,000 MPa [7], which is very high compared to other dental ceramics. But any way the microcracking and low fracture toughness of ceramics is main problem and failure cause in dental materials. In the last years nanomaterials technology rapidly grows on intriguing reality in materials science. The "nano dimension" permits the development of multifunctional material [8].

One of the most representative zirconia-based dental ceramics are In-Ceram Zirconia (IZ) (Vita Zahnfabrik, Bad Sa¨ckingen, Germany). IZ, which was developed by adding 33% 12 mol% CeO_2 partially stabilized zirconia to In-Ceram Alumina (IA), is an attempt to combine the toughening mechanism due to the phase transformation of zirconia to the versatility and ease of use of the partially-sintered glass-infiltrated alumina. Like IA, IZ is available as either slip or dry-pressed. Literature has shown contradictory results and modest improvement of the strength and fracture toughness of IZ over IA.[9 –13].There is a lack of studies about effect of nano zirconia contents and sintering temperature on mechanical properties of IZ. From the above topic the aim of this research investigation of the effect of nano ZrO_2 particle contents and sintering temperature on the fracture toughness and microhardness of in-ceram zirconia.

MATERIAL & METHODS

In this research VITA In-Ceram® ZIRCONIA (Germany) production, Zro2 nano particle (Nano SAY) have been used. Sample prepared according vita company manual and nano particles mixed with vita in- ceram powder after good stabilized in vita solution with stirring and ultrasonic mixing for 30 min. specimens were prepared with the slip-casting technique after mixing nano particle with in-ceram zirconia, according to the manufacturer's recommendations. All specimens were shaped by placing an open-ended mold on a base made from the supplied special plaster die material. The IZ slips were poured into the mold and dried before sintering in a high-temperature furnace (In-Ceramat II, Vita). After sintering, the specimens infused with a special infiltration glass. Excess glass removed by sandblasting with 50-µm Al_2O_3 at a maximum pressure of 0.3 MPa. The specimens were ground with diamond disks of nominal grit 120, 70, 30, and 15 µm to the final dimensions (1-mm thickness, 10* 30 mm).

In this research experimental design (general factorial method) have used for running experiment. Design of experiments is a statistical technique, which can used for optimizing such multivariable systems. Using design of experiments, the optimum condition having satisfactory performance can be arrived with minimum number of experiments without the need for studying all possible combinations experimentally. Further, the input levels of the different variables for a particular level of response can also be determined. The variables chosen for the study designed. DX7 software used for all statistical analysis. Factorial designs used primarily for screening significant factors, but can also be used sequentially to model and refine a process. The general factorial design allows you to have factors that each has a different number of levels. It will create an experiment that includes all possible combinations of your factor levels. All factors should be categorical (i.e. batch type, tool type, process method) rather than numeric [14]. As the experiments in this study are factor based, all possible factor combinations can experimentally studied. The most important experiment design known as 2k, and is widely used in experiments where k factors and each of their two levels investigated. Nano zirconia with four percentage 0, 1, 2, 4, and 8% and sintering temperature in 4 level (900, 1000, 1100, and 1180) have been choose as a variable. By using these method and software the experiments have been choose with the samples composition and sintering temperatures that listed in table 1.

The disks used to evaluate the fracture toughness (IF) and hardness were further polished with diamond paste of 9, 6.5, and 1 µm. Three mirror-like polished disk specimens for each material indented with a Vickers indenter mounted on a universal testing machine (KOPA PAJHOHESH) at loads of 50N. Four acceptable indentations measured per load, for 10 measurements per material.

The criteria for acceptability were:

(1) presence of only four radial cracks, with the crack length (measured from the middle of the indentation) 2.5 times longer than the half diagonal of the indentation; (2) symmetry of the indentation; (3) absence of chipping; (4) absence of large pores along the perimeter of the indentation; and (5) all cracks emanated from the corner of the indentation.

Measurements performed with scanning electron microscopy and optical microscope within a few hours after indenting. No particular steps were taken to control the presence of moisture. Fracture toughness calculated according to the following equation:

KIC= 0.016(E/H)1/2(P/c3/2)

where 0.016 = material-independent constant; P = indentation load; and c = crack length measured from the middle of the Vickers indentation [15-17].

Some indentations were made with a Vickers indenter applied for 15 seconds at loads of 10 N used to calculate hardness, as recommended by ASTM C1327-99.26.

RESULTS

ANOVA table and the mean values of fracture toughness and hardness is reported in table 1 and 2. This result shows the significant optimization analysis.

Figure 1 and 2 shows the effect of variable on the hardness and fracture toughness. In table 2, the Model F-value of 7.49 implies the model is significant. There is only a 0.14% chance that a "Model F-Value" this large could occur due to noise. Values of "Prob > F" less than 0.0500 indicate model terms are significant. In this case A are significant model terms.

Table 1: experiments and operating conditions

No.	T(^{0} C)	ZrO_2 %	N0.	T(^{0} C)	ZrO_2 %
1	1180	0	11	1000	2
2	1100	0	12	900	2
3	1000	0	13	1180	4
4	900	0	14	1100	4
5	1180	1	15	1000	4
6	1100	1	16	900	4
7	1000	1	17	1180	8
8	900	1	18	1100	8
9	1180	2	19	1000	8
10	1100	2	20	900	8

According the prediction model in the 1180 centigrade the maximum hardness will achieve and it is predictable because in high temperature the sintering process has been completed and the hardness increased. According to table 3 the Model F-value of 7.5 implies the model is significant. There is only a 0.38% chance that a "Model F-Value" this large could occur due to noise. Values of "Prob > F" less than 0.0500 indicate model terms are significant.

Figure 2 shows SEM image of samples in varius percent of nano zirconia.

DISCUSSION

In- Ceram zirconia are particularly vulnerable to slow crack growth. For these, the aim of this study is improvement of fracture toughness and crack growth.

According to figures by increasing the temperature, hardness of samples increased and fracture toughness improved. In high temperature transformation mechanism may occur and Through that transformation, zirconia has been shown to have relatively high mechanical strength and the mechanism is called Transformation toughness [19].

Table 2. Analysis of variance (ANOVA) for selected factorial model to predict Hardness

Source	Sum of Squares	DF	Mean Square	F Value	p-value
Model	2.672E+005	7	38169.26	8	< 0.0001
Temperature	2.194E+005	3	73135.33	15.41	< 0.0003
ZrO2	47778.80	4	11944.70	9.45	0.012
Residual	61186	12			
R-Squared	0.8912				

Table 3. Analysis of variance (ANOVA) for selected factorial model to predict Fracture toughness

Source	Sum of Squares	DF	Mean Square	F Value	p-value
Model	3.50	7	0.5	7.5	< 0.0031
Temperature	2.55	3	0.85	12.34	< 0.0013
ZrO2	1.55	4	0.24	5.9	0.012
Residual	1.9	12			
R-Squared	0.8312				

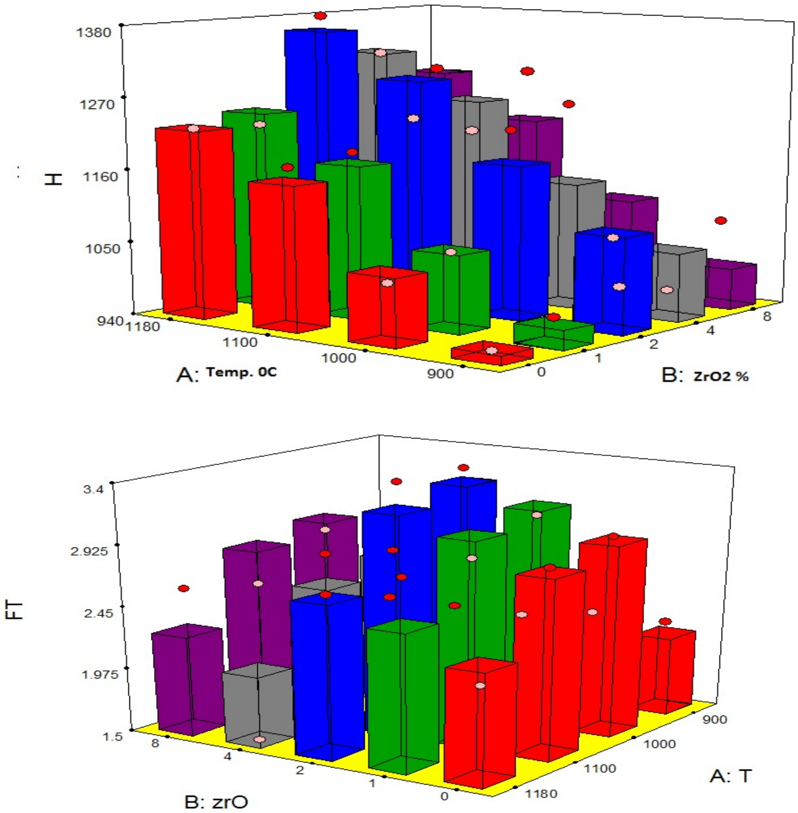

Figure 1: the effect of parameters on hardness (a) and Fracture toughness of samples

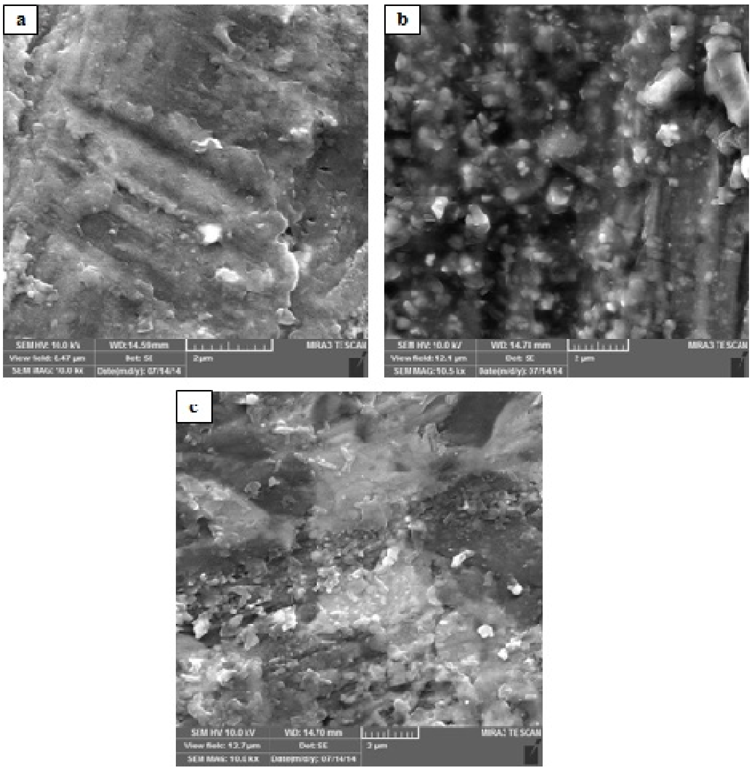

Figure 2: SEM image of sample with a) 0% b) 2% and c) 8% nano zirconia

High temperature also increases shrinkage and mickrocrack nucleation in ceramic materials [20]. According to the results by increasing the nano zirconia until 2% the fracture toughness and hardness increased. This is may be because of decreasing grain size of sample and improve the resistance of crack grow. Fine particles size has good distribution and low porosity [21].

Physical properties of the zirconia depend on particle size, surface area, crystallite size and the presence of impurities [22]. In high percent of ZrO_2, the fracture toughness decrease. In the present of Zro_2 in high percentage, the improvement effect diminished and cause decrease properties.

Inceram samples showed that the mean fracture toughness is 2.6(MPa m½) and the mean hardness is 1150 V. The test revealed a statistically significant difference between the groups and fracture toughness. There is a statistically significant difference between groups that reveals that the nano particles has good effect on in-ceram fracture toughness and hardness.

Figure 1 shows the SEM micrograph of samples in present of various concentration of nano zirconia. According this figure in the sample with 2% of nano zirconia, surface morphology is more homogeneous than others. By increasing the nano zirconia percentage the zirconia powder distribution in sample decrease and it may be by agglomeration effect and decrease fine structure. This effect decreases the properties of sample that show in previous result and it can be justification of result.

Result shows that by increasing hardness the frcture toughness decreesed that it is predictable. Adding zirconia nano particles to the in-ceram the fracture touness improved and this is becous of fine grain size but in high percent of nano particle the properties decreased.

CONCLUSION

The present study has shown that the effect of nano zirconia reinforce and sintering temperature on fracture toughness, and hardness in dental ceramics. Heat treatment of samples in high temperature increase hardness and decrease fracture toughness. By the way, heat treatment in low temperature the sintering process is incomplete and the samples did not show good properties. According the model the best condition can achieved in 2% of nano zirconia content and sintering temperature of 1100 centigrade.

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