

Dental Porcelain (Core) with Improved Fracture Toughness and Microhardness

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Abstract: To study the effect of different cooling rates after glaze firing on Radius of radial crack, Fracture toughness and Microhardness of Alumina reinforced porcelain. Total 30 porcelain discs reinforced with aluminium oxide of 8mm diameter×0.6mm thickness were prepared. Specimens were divided into three groups, each group containing ten discs. They were positioned at the entrance of the furnace with a temperature of 593°C for ten minutes and then elevated to a non vacuum furnace while the temperature was increased to 970°C at a rate of 50°C per minute for glaze firing and each group was cooled at a different rate. After cooling, indentations on each disc were made with a Vickers indenter with a microhardness tester loaded with one kilogram for twenty seconds. Statistical analysis revealed significant differences in fracture toughness of porcelain for each of the cooling rates. The slow-cooled group had significantly lower fracture toughness than the group with medium cooling rate and the group with rapid cooling rate. Highest fracture toughness value was obtained for rapidly cooled group. The highest VMH value was also obtained for the rapidly cooled group.

Keywords: Aluminous porcelain, hardness, firing & fracture toughness

1. Introduction

'Dental ceramics' is one of the fastest growing fields of esthetic dental material research and development. Current technology in dental ceramics is advancing at a rapid pace constantly producing new materials. Standard of esthetics in dental porcelain has now reached a stage where the development of new porcelains meet ever increasing demands on their optical and physical properties. Their excellent biocompatibility, wear resistance, colour stability and translucency have made them the most reliable esthetic restorative material in the field of dentistry.

In spite of all these advantages, there is one disadvantage that precludes its use as a universal restorative material, i.e., its potential to fracture. Initially some researchers reported decreased strength of all-ceramic crown systems in comparison to conventional porcelain fused to metal restorations. Its brittleness and low tensile and shear strengths render the porcelain restoration liable to fracture during mastication. A well-established explanation for porcelain fracture is 'crack propagation'. A fracture commonly originates at a surface flaw, propagates through others in the material. During cooling after firing, separation at grain or inclusion boundaries can cause crack development with restoration failure. It has recently been advocated that a stronger and tougher core material would improve the reliability and the life time of an all-ceramic restoration. A significant improvement in the fracture resistance of porcelain was reported when it was reinforced with alumina (40-50% Al₂O₃). The ability of a material to resist crack propagation is quantified by its fracture toughness (K_{IC}). Fracture toughness is calculated by

measurement of radial cracks created in the material by a loaded microindenter.

Present study is to assess the effect of different cooling rates after glaze firing of alumina reinforced porcelain, on fracture toughness and microhardness. The present study has got its significance because by this study we will be able to know the ideal mode of cooling rate, to increase fracture toughness and Vickers microhardness, which can prolong the longevity of the ceramic restoration.

2. Review Literature

It was reported in a study that the aluminous porcelain was significantly tougher than feldspathic porcelain. These differences in K_{IC} were attributed to differences in the nature of crack-microstructure interaction occurring in the two types of porcelain [1].

In studies, the effects of sandblasting and coating techniques on volume loss, surface morphology and surface composition of In-Ceram ceramic. Conclusion was that sandblasting of all ceramic clinical restorations with feldspathic glass materials should be avoided, but for In-Ceram ceramic the volume loss was within an acceptable range and similar to that of noble metals [2]. When three new ceramic crown core materials to compare their biaxial flexural strength and indentation fracture toughness. Ten specimens of Empress, In-Ceram, and Procera AllCeram ceramics were prepared according to their manufacturers' recommendations. There was no statistically significant difference between the fracture toughness of Procera (4.48 MPa) and In-Ceram ceramics (4.49 MPa); however, both

ceramics had significantly higher fracture toughness ($p < 0.005$) than toughness of Empress ceramic (1.74 MPa) [3].

Reports are there about the flexure strength under static and cyclic loading and the fracture toughness under static loading of six restorative ceramic materials [4]. The lifetime of a ceramic is dependent on the presence of incidental cracks and their gradual propagation under the conditions of the oral cavity. The objective of the study was to examine the long-term strength of glass-infiltrated alumina- and various zirconia ceramics currently used in CAD/CAM systems to manufacture crown and bridge frameworks. Based on these parameters, lifetime diagrams were generated which allowed the evaluation of the long-term behavior. The results showed that in a moist environment, the glass-infiltrated alumina- and some zirconia ceramics have a high susceptibility to subcritical crack growth. Zirconia ceramics with an alumina oxide content of 0.25 wt %, exhibited the highest initial and most favorable long-term strength, and should therefore be suitable for crown and bridge restorations [5].

Studies were conducted to evaluate the influence of the bending test configurations and the crosshead displacement speeds on the fracture toughness (K_{Ic}) of dental porcelains obtained with the indentation strength in bending (ISB) method. Conclusion made was that the crosshead displacement speed can cause statistically different results of fracture toughness obtained with the ISB method [6]. New generation all-ceramic designing and materials present various options, both in material selection and fabrication techniques. Success of the ceramic restoration depends on clinician's ability to select the appropriate material to match functional and esthetic demands [7]. The surface quality of dental ceramics influences the formation of bacterial biofilm. In the oral environment, the dental plaque forms a constant threat leading to periodontitis and other conditions such as peri-implantitis. Adherence of microbial species to dental ceramics and the subsequent formation of biofilms on their rough surfaces act as favourable factors to plaque-related systemic diseases [8].

3. Materials and Methods

Metal mould for the test specimen 8mm diameter and 0.6mm thickness, Test specimen made in inlay casting wax, Blue opaque inlay wax-Hindustan inlay wax, Bionline petroleum jelly IP, Disc abrasives-LM Abrasives, Mitutoyo micrometer, Investment material-Lamina west, Alumina reinforced porcelain powder (Vitadur-N 338, Vitazahnfabrik, Badsackingnen, Germany) & Evanson's gauge. Equipments used for characterizations are Ceramic furnace-Jelrus wizard, Sand blaster-Duostar-Bego and Microhardness indenter-Clemex, Digital, 100X-1000X.

3.1 Methodology

Total 30 porcelain discs reinforced with aluminium oxide of 8mm diameter \times 0.6mm thickness were prepared. Specimens were divided into three groups, each group containing ten discs. They were positioned at the entrance of the furnace with a temperature of 593°C for ten minutes and then elevated to a nonvacuum furnace while the temperature was

increased to 970°C at a rate of 50°C per minute for glaze firing and each group was cooled at a different rate. After cooling, indentations on each disc were made with a Vickers indenter with a microhardness tester loaded with one kilogram for twenty seconds. The diameter of radial cracks that emanated from Vickers indentation were measured within one minute after indentation.

3.2 Preparation of test specimen

Total 30 porcelain discs reinforced with aluminium oxide of 8mm diameter \times 0.6mm thickness were prepared in the following manner: A circular Metallic mould was prepared to make wax patterns at specified dimensions that is 8mm diameter and 0.6mm thickness. The mould consisted of two semicircular parts which, when assembled together formed a circular mould space for the wax pattern. Into this, the molten wax was poured to the appropriate level which was retrieved after proper hardening. The application of petroleum jelly facilitated easy retrieval. Investment moulds were prepared by using the wax patterns. Then the mould space was filled with porcelain slurry and vibrated and the excess moisture was blotted dry. The porcelain discs were dried in front of an open door of a furnace at 593°C for 10 minutes and then placed inside the furnace and vacuum fired with a temperature increase of 50°C per minute upto 970°C. The investment material was separated from porcelain discs by sandblasting. Porcelain discs were polished using abrasives. 30 porcelain discs were divided into three groups, each group containing ten discs. They were positioned at the entrance of the furnace with a temperature of 593°C for ten minutes and then elevated to a nonvacuum furnace while the temperature was increased to 970°C at a rate of 50°C per minute for glaze firing. After firing each group was cooled at a different rate as specified.

- 1) Rapidly cooled group:-Specimens were subjected to rapid cooling by lowering the firing platform to its most inferior position and removing specimens from the vicinity of the furnace and allowing them to cool to room temperature.
- 2) Medium-cooled group:-Specimens were subjected to a medium rate of cooling by lowering the firing platform to a position 3cm from the entrance to the furnace for 4minutes followed by a position at 6cm for 4minutes and then removing specimens from the vicinity of the furnace.
- 3) Slow-cooled group:-Specimens were subjected to slow cooling by positioning the tray 2cm from the entrance to the furnace for 12minutes and then switching off the furnace and allowing specimens to cool to room temperature.

3.3 Testing the specimens

Indentations on each disc were made with a Vickers indenter with a microhardness tester loaded with one kilogram for twenty seconds. The diameter of radial cracks that emanated from Vickers indentation were measured within one minute after indentation. Two readings were made for each indentation, and the average of the ten readings used to derive the fracture toughness for each disk. The fracture toughness was calculated by the following formula:

$$K_{c} = \frac{1}{\pi^{3/2} \tan. \psi} [P/D^{3/2}] \quad [1]$$

Kc fracture toughness (residual stress intensity factor), ψ indenter cone angle(136/2=68), P peak contact load and D radius of radial crack. Vickers microhardness (VMH) was calculated from the measurement of the axes of the indentation. The average measurement of five indentations was used for the VMH. The mean Kc and VMH values for each group were calculated.

3.4 Data analysis

Data are expressed in its mean values and standard deviation (\pm SD). Analysis of variance (One Way ANOVA) were performed as parametric test to compare different variables. Duncan’s Multiple Range Test was employed as post hoc analysis to compare the variables individually.

4. Results

Throughout the study the abbreviations and corresponding explanations are as follows:

Rapid -Rapidly cooled group, Medium-Medium cooled group, Slow-Slow cooled group, VHN-Vickers microhardness, Kc-Fracture toughness.

Table 1(a): Analysis of Variance (ANOVA) of crack distance (μm) comparing different cooling rates

Cooling Rate	N	Mean	\pm SD	Minimum	Maximum
Rapid	10	55.28 ^a	1.58	53.00	58.00
Medium	10	64.80 ^b	1.71	62.00	68.00
Slow	10	70.52 ^c	0.95	69.00	72.00

a, b, c: Means with same superscript do not differ each other (Duncan’s Multiple Range Test)

Table 1(b): Analysis of Variance (One Way ANOVA) statistics for crack distance

Comparisons	Sum of Squares	df	Mean Square	F	P value
Between Groups	5926.77	2	2963.39	1402.685	< 0.001
Within Groups	310.56	147	2.11		
Total	6237.33	149			

Table 2 (a): Analysis of Variance (ANOVA) of fracture toughness (Kc) ($\text{MN}/\text{m}^{3/2}$) comparing different cooling rates

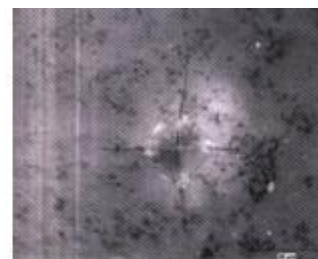
Cooling Rate	N	Mean	\pm SD	Minimum	Maximum
Rapid	10	1.77 ^c	0.08	1.64	1.88
Medium	10	1.39 ^b	0.06	1.29	1.49
Slow	10	1.22 ^a	0.02	1.19	1.27



(I) - rapid



(II) - medium



(III) - slow

Figure 1: Radial cracks at different cooling rates

a, b, c: Means with same superscript do not differ each other (Duncan’s Multiple Range Test)

Table 2 (b): Analysis of Variance (One Way ANOVA) statistics for fracture toughness

Comparisons	Sum of Squares	df	Mean Square	F	P value
Between Groups	7.74	2	3.87	1239.99	< 0.001
Within Groups	0.459	147	0.01		
Total	8.198	149			

Table 3 (a): Analysis of Variance (ANOVA) of Vickers Microhardness (Kg/mm^2) comparing different cooling rates

Cooling Rate	N	Mean	\pm SD	Minimum	Maximum
Rapid	10	608.29 ^c	34.73	551.25	660.16
Medium	10	442.54 ^b	23.48	401.04	482.41
Slow	10	373.09 ^a	10.10	357.72	389.50

a, b, c: Means with same superscript do not differ each other (Duncan’s Multiple Range Test)

Table 3 (b): Analysis of Variance (One Way ANOVA) statistics for VMH

Comparisons	Sum of Squares	df	Mean Square	F	P value
Between Groups	1460235.41	2	730117.71	1178.019	< 0.001
Within Groups	91108.27	147	619.78		
Total	1551343.68	149			

Data were analyzed using computer software, Statistical Package for Social Sciences (SPSS) version 10. Data are expressed in its mean values and standard deviation (\pm SD). Analysis of variance (One Way ANOVA) were performed as parametric test to compare different variables. Duncan’s Multiple Range Test was employed as post hoc analysis to compare the variables individually. For all statistical evaluations, a two-tailed probability of value, < 0.05 was considered significant.

4.1 Inference

Statistical analysis revealed significant differences in fracture toughness of porcelain for each of the cooling rates. The slow-cooled group had significantly lower fracture toughness than the group with medium cooling rate and the group with rapid cooling rate. In the present study,

- 1) Lowest radius of radial crack was observed for rapidly cooled group.
- 2) Highest fracture toughness value was obtained for rapidly cooled group.
- 3) Highest VMH value was also obtained for the rapidly cooled group.

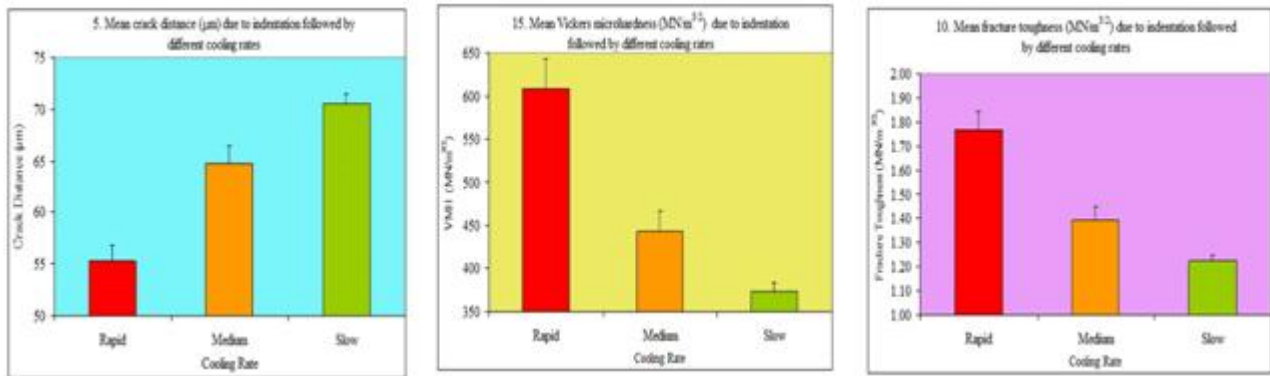


Figure 2: Crack distance, Vickers Hardness & Fracture toughness at different cooling rates

5. Discussion

The term "ceramics" is derived from the Greek word "keramos" meaning "burnt stuff" [9]. Ceramic is a material made essentially from a non-metallic mineral (as clay) by firing at a high temperature; whereas a porcelain is a ceramic material formed of infusible elements joined by lower fusing materials and composed essentially of kaolin, quartz and feldspar.

A ceramic restoration consists essentially of two components viz. an inner core and an outer veneer. In all-ceramic restorations both core and veneer are of ceramic. They are capable of producing the best cosmetic effect of all dental restorations. The first all-ceramic crown was developed by Land in 1886 and was known as the porcelain jacket crown (PJC) [10]. Because of the tendency of this type of restoration to fracture it was usually limited to single anterior tooth primarily incisors.

In 1965 McLean and Hughes developed a porcelain jacket crown with an inner core of aluminous porcelain containing 40-50% Alumina crystals to block the propagation of cracks [11]. The refractory inner core of the restoration that surrounds the preparation layered with conventional porcelain restoration is approximately twice as strong as the traditional porcelain jacket crown.

In the last two decades research has focused on strengthening dental ceramics by modification of the porcelain's microstructure to overcome its susceptibility to brittle fracture. Three mechanisms strengthen ceramics and they all require incorporation of a second phase of heat generated crystal production to increase the energy necessary for crack propagation.

Crack-tip interactions: Obstacles in the microstructure impede crack motion by reorienting or deflecting the path of fracture.

Crack-tip shielding: Events triggered by high stresses in the crack-tip region act to reduce stress: i.e., in transformation toughening. (Eg: Zirconia reinforcement).

Crack-bridging: Second phase crystalline structure act as a 'bandage' to prevent crack from opening further.

According to the reviewed articles the success of a ceramic restoration depends on three main factors i.e. strength, accurate fit and aesthetics. Various authors have mentioned

that susceptibility of ceramics to brittle fracture is the major disadvantage and this feature restricts their use in dental restorations [12]. They suffer from an inability to absorb appreciable quantities of elastic strain energy prior to fracture. Measure of the absorbing ability of strain - energy of a brittle material is the critical stress intensity factor (Fracture toughness or K_{Ic}). Strength, resistance to thermal shock and susceptibility to erosive wear, all are basically controlled by this parameter. Dispersion strengthening is a process by which the dispersed phase of a different material (such as alumina, leucite, zirconia, etc.) is used to stop crack propagation as these crystalline phases are more difficult to penetrate by cracks [13]

Fracture toughness has got prime importance in determining the various aspects of mechanical behaviour of a brittle material. Knowledge of fracture toughness (K_{Ic}) of dental ceramics is an essential starting point if the resistance to fracture of ceramic - based dental prosthesis has to be improved

Anusavice et al reported that tempering (rapidly cooling ceramic after firing) treatment enhanced resistance of opaque and body feldspathic porcelain to crack initiation [14]. Morena et al stated that K_{Ic} is not sensitive to the size and density of surface flaws, which are in turn controlled by the manner in which test specimens are prepared. This was also supported by the studies conducted by Rosenstiel and Porter [15].

Present study is an attempt to assess the effect of different cooling rates after glaze firing of alumina reinforced porcelain, on fracture toughness and microhardness. Fracture toughness values of alumina reinforced porcelain at three different cooling rates were observed.

Haim Baharav et al studied the effect of different cooling rates on fracture toughness of glazed porcelain reinforced with aluminium oxide. They concluded that rapid cooling after glaze firing can result in higher fracture toughness than that in medium and slow cooling. The results of the present study were consistent with their observations [19].

In the present study fracture toughness was determined by the indentation fracture technique with a digital microhardness tester. Only small quantity (a few grams) of sample is required for this method [17] So this method is particularly suitable for expensive materials like dental ceramics. According to Susanne et al use of indentation

fracture technique for finding the fracture toughness values of porcelain was highly useful in reducing the number and size of specimens needed [18].

Amin et al also supported that indentation fracture toughness and ultrasonic test methods exhibited lower coefficient of variation compared to conventional methods [19] and they stated that small specimens were required to produce an acceptable number of data for statistical analysis. The basis of this technique is the series of cracks that form around a Vickers hardness indentation in a brittle material. These cracks (radial cracks) appear to emanate, when viewed from the above, from each of the corners of the indentation. The size of the crack is an inverse function of fracture toughness [20].

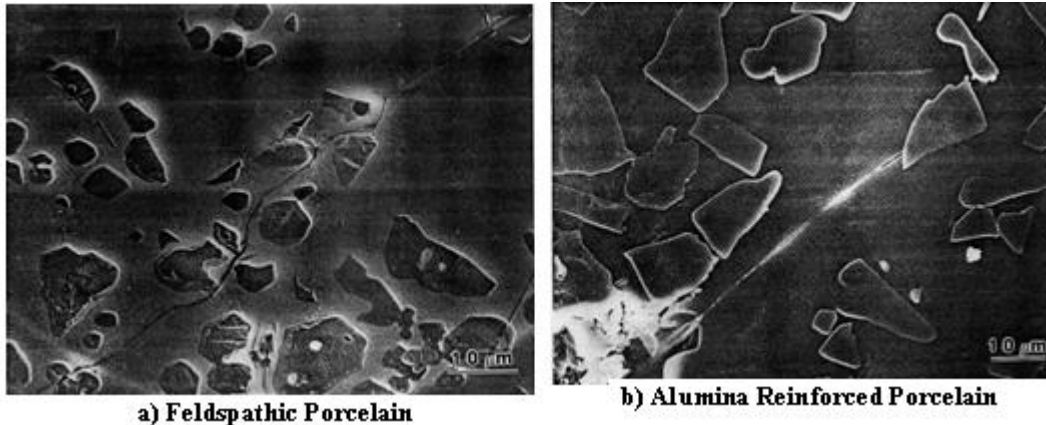


Figure 2: SEM images of crack propagation

In the present study total 30 porcelain discs reinforced with aluminium oxide of 8mm diameter \times 0.6mm thickness were prepared. Specimens were divided into three groups, each group containing ten discs. They were glaze fired and each group was cooled at a different rate.

In the first group, test specimens were subjected to rapid cooling by lowering the firing platform to its most inferior position.

In the second group, test specimens were subjected to a medium rate of cooling by lowering the firing platform to a position 3cm for 4 minutes followed by a position at 6cm for 4minutes and then removing these specimens from the vicinity of the furnace.

In the third group, test specimens were subjected to slow cooling by positioning the tray 2cm from the entrance to the furnace for 12minutes and then switching off the furnace and allowing these specimens to cool to room temperature.

After cooling, indentations on each disc were made with a Vickers indenter with a microhardness tester. The mean fracture toughness (Kc) and Vickers Microhardness (VMH) values for each group were calculated. The results of the present study were inconsistent with Anusaviceet al's observations [4].

In the present study, glazed specimens were used which was supported by Haim Baharav et al's observations [21]. In their study they compared effect of glaze thickness on fracture toughness and Vickers hardness number of Alumina-reinforced porcelain. They stated that minimal and

In the present study, Kc was found to be inversely proportional to radius of radial crack which was in consistent with the above mentioned literature. Morena et al [1] stated that basic difference between the crack propagation in feldspathic and alumina reinforced porcelain was that indentation cracks deflected away from the leucite crystals in feldspathic porcelain whereas cracks interact directly with the alumina dispersed phase in the latter. They observed that Kc value for alumina reinforced porcelain was nearly 1.5 times that for feldspathic porcelain[1]. These difference can be reconciled from the Scanning Electron Microscopic (SEM) micrograph of crack microstructure interactions.

maximum thickness of glaze layers on alumina-reinforced porcelain resulted in a surface that was harder and more resistant to fracture than moderate glaze thickness.

Russel et al examined the effects of surface finish on a conventional feldspathic ceramic material (Vita VMK 68, Vita Zahnfabrik) and an aluminous ceramic material (Vitadur N 338, Vita Zahnfabrik). They observed that polishing of all the ceramic materials significantly increased strength [22]

Statistical analysis revealed that there is significant difference in fracture toughness, microhardness and radius of radial cracks among the three groups. From observations and statistical analysis present study comes to the conclusion that different rates of cooling after firing a glazed alumina reinforced porcelain created various degrees of crack propagation. Rapidly cooled group showed smallest degree of crack propagation, greatest fracture toughness and Vickers microhardness values. The findings of the present study are confirmed by the studies conducted by Haim Baharav and Ben Zion Laufer. Studies by the Morena and Fairhurst also supported the inference of the present study.

6. Conclusion

During the last two decades the search for 'metal free and more natural looking restorations' led to the development of several new all ceramic systems with superior aesthetic qualities such as translucency and fluorescence similar to natural tooth. But still irrespective of its superior aesthetic

quality, fracture of the all ceramic crowns is considered as a problem. So use of these materials is limited to low stress situations.

In the present study, effect of different cooling rates on crack propagation, fracture toughness and Vickers microhardness of alumina reinforced porcelain were evaluated.

The following conclusions were drawn,

- 1) Rapidly cooled group showed least amount of crack propagation.
- 2) Rapidly cooled group showed highest fracture toughness value and
- 3) Rapidly cooled group showed highest Vickers microhardness value.

It can be concluded that rapid cooling can contribute to increased fracture toughness and Vickers microhardness in the alumina reinforced porcelain. Elevated fracture toughness will definitely prolong the longevity of the ceramic restoration and increase hardness will improve the material's ability to withstand plastic deformation.

The significance of the study was that we were able to know the ideal mode of cooling to increase the fracture toughness and microhardness which can inturn prolong the longevity of the ceramic restoration.

7. Future Scope

By noting the advantages and disadvantages of various firing cycles, the clinician and laboratory personnel can decide the ideal firing cycles and method of cooling after firing according to the situational demands. In future further studies can be planned for other core porcelains also.

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