A STUDY ON THE INFLUENCE OF DIFFERENT MODES OF COOLING ON THE STRENGTH OF GLAZED ALUMINA PORCELAIN

Abstract

The lab processing techniques for porcelain can significantly affect strength of a porcelain dental restoration, and thereby, its clinical performance. In this context, a study was done to assess the influence of different rates of cooling on the strength of glaze fired alumina reinforced porcelain. Fracture toughness and microhardness were the parameters used to assess strength. Vickers microhardness indentation was used to quantify fracture toughness and microhardness. And, Image J Analyzer, a software tool was used for measuring the cracks created in the porcelain on indentation. The study also attempts to assess if a change in the medium of cooling, can affect strength. The rapidly cooled groups (both in air and water), had higher strength relative to slow and medium cooled groups.

Keywords: Cooling rates; microhardness; fracture toughness; alumina reinforced porcelain; glaze firing

Authors

Dr. Aathira Kuruvilla
Department of Prosthodontics
Mar Baselios Dental College
Ernakulam, Kerala, India

Dr. Babu Cherian
Department of Prosthodontics
Mar Baselios Dental College
Ernakulam, Kerala, India

Dr. Jacob Kuruvilla
Department of Community Dentistry
Mar Baselios Dental College
Ernakulam, Kerala, India

I. INTRODUCTION

Dentistry has given much significance to meeting esthetic demands of patients. The developments in the field of ceramics have accelerated the evolution of dentistry in this regard. However, brittleness and low tensile and shear strengths render the ceramic material prone to fracture under forces of mastication. Porcelain fracture has been attributed to "crack propagation". Greater extent of crack propagation would imply a higher chance for fracture. A fracture usually initiates at a surface flaw, and spreads through other flaws in the material. Fracture toughness (Kc) quantifies the ability of a material to resist propagation of cracks. And, it can be quantified by measurement of radial cracks created in the material with a loaded microindenter.

There are several parameters to assess the clinical potential of dental ceramics. The evaluation of strength may hold a clue to long term restorative success, along with other clinical and technical factors in the success of ceramics as a restorative material. This study focuses on the effect of different modes of cooling of glaze fired alumina porcelain on its Vickers microhardness and fracture toughness values. These are parameters related to the strength of ceramics.

1. **Fracture toughness as a strength parameter:** "Toughened glass" for car windscreens and glass doors is fabricated by a process called thermal tempering. Here, the glass is heated to a critical temperature and then rapidly quenched to room temperature by air jets or, occasionally, an oil bath [1]. This process creates a high residual compressive surface stress on the material. And, makes the material capable of resisting forces that leads to initiation and propagation of cracks, and thereby fracture.

   Just as commercial tempering is used to strengthen glass [2], metal-ceramic restorations are also tempered by removing them from the furnace at high temperatures and allowing them to bench-cool in air at ambient temperatures. This has been a common practice by dental laboratories to improve the strength of veneering porcelain [3,4]. Strength is regarded as a parameter that affects the clinical performance of ceramic restorative material. But, with extremely brittle materials such as ceramics, high strength does not imply a higher fracture resistance. Fracture is caused by a propagating crack. A crack originates from flaws and spreads when the applied stress exceeds a certain threshold. This threshold level will also depend on the crack tip radius, flaw size and distribution, and fracture toughness, in very brittle materials.

   Fracture toughness is considered an important parameter in fracture mechanics for brittle materials. It is assumed to be independent of flaw size, specimen shape, and the stress concentration acting on the surface. It is characterized by a critical level of the stress intensity factor near the crack tip at which a crack will initiate to propagate.

   The concept of quantifying fracture toughness in brittle materials with indenter was first developed by Palmqvist. In ceramic materials, the use of the Vickers indentation technique for the evaluation of the fracture toughness has become outstanding due to the simplicity of specimen preparation. It requires only the provision of small size of specimen surface, enabling generation of large quantity of measurements. This technique has been used to evaluate fracture toughness of dental porcelain, composite resin, as well as human enamel and dentin. [5-9]

2. **Relationship of different modes of cooling with fracture toughness and hardness values in dental ceramics:** A study conducted by Haim Baharav et al concluded that rapid cooling of glazed porcelain reinforced with aluminium oxide had better fracture toughness values relative to medium and slow cooling.[10] Niwut Juntavee et al in a study on the fracture toughness of different feldspathic porcelains, observed that fast cooled procedure resulted in greater toughness of porcelain [5]

   It is however interesting to note that fast cooling protocols are not followed for veneering zirconia porcelains. Zirconia is a poor thermal conductor, and can maintain a higher thermal gradient for a longer time compared to other porcelain material. Rapid cooling thus leads to formation of weak tensile zones within the veneering porcelain with increased risk of fracture. Hence, slow cooling protocols are followed for zirconia-based restorations to reduce the development of tensile zones, and thereby decrease the chances of chipping fractures.[11]

II. METHODOLOGY

The method involved the evaluation of fracture toughness and microhardness of alumina porcelain (Shofu Inc., Kyoto, Japan) ceramic discs. Forty specimens, ten each from four different groups was tested with Vickers microhardness indenter.

A standardized rigid plastic mold for forming ceramic disc of 8 mm x 0.6 mm was fabricated. The ceramic disc could be ejected from the mold by piston pump mechanism. A separating medium, Picosep (Renfert, Germany) was applied onto the mold. The alumina reinforced porcelain powder is condensed and packed into the plastic mold. A ceramic disc of 8mm x 0.6mm was ejected from the mold. The ceramic disc was subjected to bisque firing in the ceramic furnace, and mildly polished. Glaze (Renfert, Germany) was applied to the ceramic disc. The ceramic disc was then subjected to glaze firing in the ceramic furnace.

Cooling of ceramic disc was done at different rates in the following protocol:

Following glaze firing, 10 ceramic discs each of the given dimensions are cooled in four different cooling protocols as described below:

- **Rapidly cooled group:** After completion of firing, the ceramic discs on the firing platform are immediately lowered to its inferior most position. The ceramic discs were removed from the vicinity of the furnace, allowing them to cool to room temperature.

- **Medium-cooled group:** Specimens were subjected to a medium rate of cooling by lowering the firing platform to 6cm, at the rate of 3cm for 4minutes. Then the specimens are removed from the vicinity of the furnace.

- **Slow-cooled group:** Here, the specimens were subjected to slow cooling by lowering the tray to 2cm from the entrance of the furnace for 12 minutes. The furnace is then switched off. And the specimens are allowed to cool to room temperature.

- **Rapidly cooled in water:** The ceramic discs are rapidly cooled as in the first group. But, here the specimens are cooled by quenching in water.

- Each ceramic disc was tested with digital Vickers microhardness indenter Shimadzu HMV- 2TAW. A 300gf test load was applied for 14 seconds. Five indentations were made on each disc, and averages were taken to assess Vickers microhardness value, and crack length. An optical image is obtained on testing each sample with the digital microhardness indenter. Vickers microhardness values was obtained from the axis of indentation, while the crack length was assessed with ImageJ analyzer, which was downloaded from the National Institute of Health website.

The fracture toughness was calculated by the following formula:

1. $Kc = 1[P/D]^{3/2} \div \pi^{3/2} \tan \omega$

Kc= fracture toughness (residual stress intensity factor),

ψ= indenter cone angle (136/2=68)

P =peak contact load and D= radius of radial crack

III. RESULTS

The statistical analysis was done with SPSS16.0 statistical package. The observed data was abstracted using mean and standard deviation. Further data analysis to estimate the level of differences between the groups were done using ANOVA analysis.

- In this study,
  - The rapidly cooled group had the least crack length.
  - The rapidly cooled group had the highest fracture toughness values.
  - The rapidly cooled group also had the highest Vickers microhardness strength.
  - In the rapidly cooled group, higher VMH values, higher fracture toughness values, lower crack length values were noted in the group cooled in air relative to the group cooled in water.
  - Vickers microhardness values and fracture toughness values showed positive correlation.

Table 1: Data expressed in mean and standard deviation

Descriptive	Cooling rates	N	Mean	±	Standard deviation
Vickers	Rapid cooling in air	10	511.0286	±	16.07569
microhardness values	Medium cooling in air	10	467.3738	±	04.42086
(Kg/mm^2)	Slow cooling in air	10	444.8110	±	12.91117
	Rapid cooling in	10	496.3406	±	11.30576
	water				
	Total	40			
Crack length(μm)	Rapid cooling in air	10	19.7710	±	2.61431
	Medium cooling in air	10	30.9427	±	1.06636
	Slow cooling in air	10	39.7347	±	3.30354
	Rapid cooling in	10	22.6844	±	1.42036
	water				
	Total	40			
Fracture	Rapid cooling in air	10	2.5509 ± .40044		
toughness((Kc)	Medium cooling in air	10	1.276 ± .06518		
	Slow cooling in air	10	0.8820 ± .12917		
	Rapid cooling in	10	2.0360 ± .17488		
	water				
		40			
	Total				

Highest mean Vickers microhardness value, highest fracture toughness value, least crack length is observed in the rapidly cooled group (both cooled in air and water), relative to medium and slow cooled groups.

Means plots

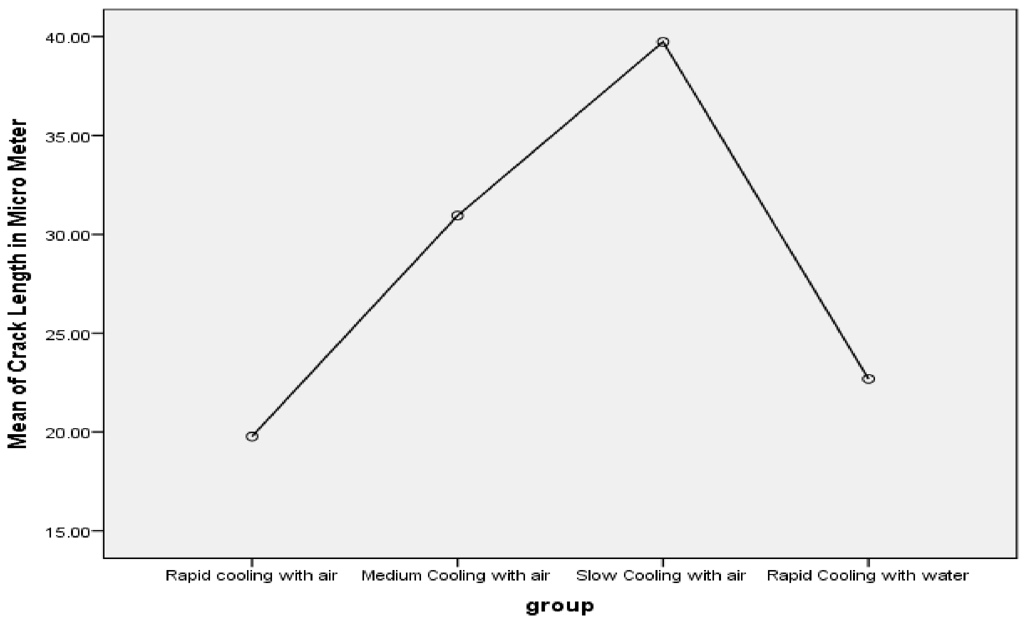

Figure 1: Means plot of crack length across differently cooled groups.

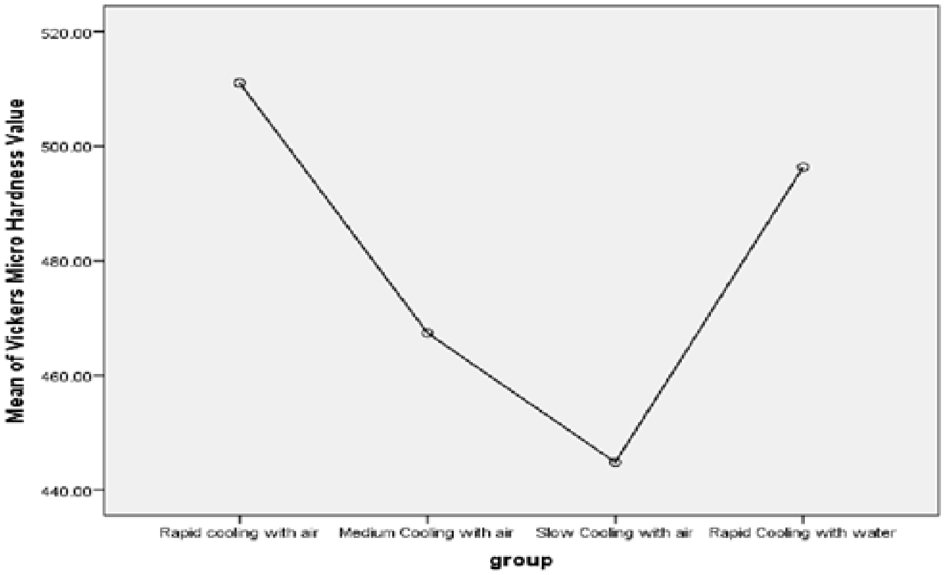

Figure 2: Means plot of Vickers Microhardness values across differently cooled groups

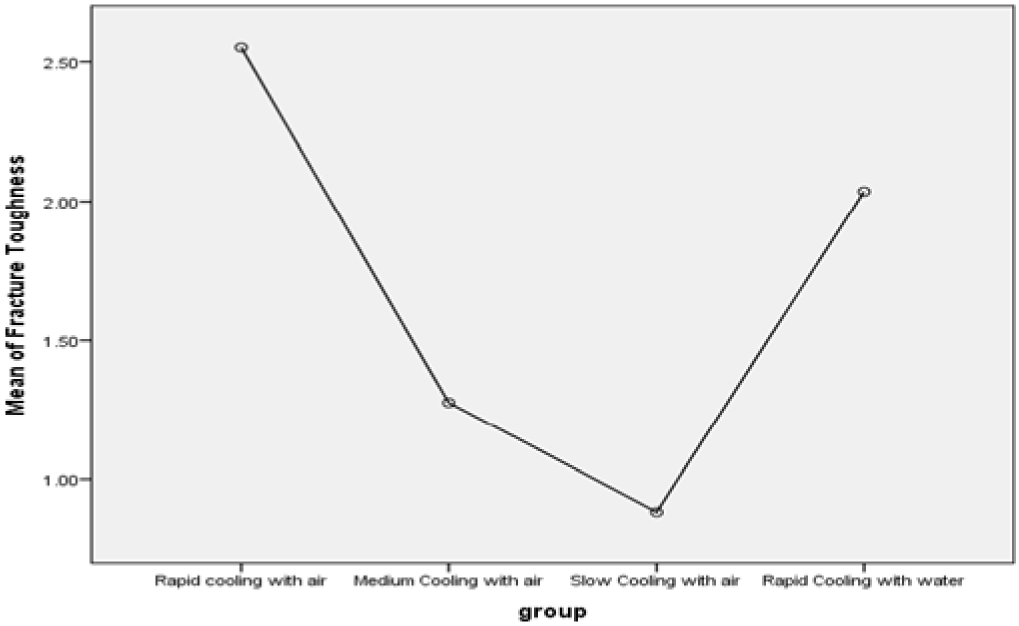

Figure 3: Means plot of fracture toughness across differently cooled groups

One way ANOVA analysis

One way ANOVA analysis shows significant increase in Vickers microhardness values, fracture toughness values, and significant decrease in crack length values, in rapidly cooled group, relative to medium and slow cooled groups. The p value was found to be .000, for the three parameters (Vickers microhardness values, crack length and fracture toughness) observed.

Post hoc Tests-After ANOVA analysis, post hoc test, Tukey HSD test was performed for further detailed analysis by multiple comparison among the groups for each parameter assessed.

Table 2: ANOVA analysis

Descriptives		Sum of squares	df	Mean square	F	Significance
Vickers microhardness (Kg/mm^2)	Between groups	26274.261	3	8758.087	61.193	.000
	Within groups	5152.413	36	143.123		
	Total	31426.675	39			
Crack length(µm)	Between groups	2420.145	3	806.715	154.376	.000
	Within groups	188.123	36	5.226		
	Total	2608.268	39			
Fracture toughness((Kc) ($MN/m^{3/2}$)	Between groups	16.851	3	5.617	106.044	.000
	Within groups	1.907	36	.053		
	Total	18.758	39			

Dependent variable: Vickers microhardness.

Table 3: Tukey HSD test; Vickers microhardness data

(I) group	(J) group	Mean Difference (I-J)	Std. Error	Sig.
Rapid cooling with air	Medium Cooling with air	43.65480*	5.35019	.000
	Slow Cooling with air	66.21760*	5.35019	.000
	Rapid Cooling with water	14.68800*	5.35019	.044
Medium Cooling with air	Rapid cooling with air	-43.65480*	5.35019	.000
	Slow Cooling with air	22.56280*	5.35019	.001
	Rapid Cooling with water	-28.96680*	5.35019	.000
Slow Cooling with air	Rapid cooling with air	-66.21760*	5.35019	.000
	Medium Cooling with air	-22.56280*	5.35019	.001
	Rapid Cooling with water	-51.52960*	5.35019	.000
Rapid Cooling with water	Rapid cooling with air	-14.68800*	5.35019	.044
	Medium Cooling with air	28.96680*	5.35019	.000
	Slow Cooling with air	51.52960*	5.35019	.000

*. The mean difference is significant at the 0.05 level.

The above table illustrates that there is a significant increase in Vickers microhardness values in the rapidly cooled group relative to medium and slow cooled groups. It is noted that different modes of cooling significantly affect the Vickers microhardness value. This post hoc test shows significant difference between the groups, given that the mean difference is significant. The rapidly cooled group in air shows significantly higher VMH (p value: .044), relative to the group cooled in water. Tukey HSD

Dependent variable: Crack length in micrometers

Table 4: Tukey HSD test; Crack length data

(I) group	(J) group	Mean Difference (I- J)	Std. Error	Sig.
Medium Cooling with		-11.17173*	1.02231	.000
air				
Rapid cooling with air	Slow Cooling with air	-19.96373*	1.02231	.000
Rapid Cooling with		-2.91343*	1.02231	.035
water				
Rapid cooling with air		11.17173*	1.02231	.000
Medium Cooling with	Slow Cooling with air	-8.79200*	1.02231	.000
air	Rapid Cooling with	8.25830*	1.02231	.000
water				
Rapid cooling with air		19.96373*	1.02231	.000
Medium Cooling with		8.79200*	1.02231	.000
Slow Cooling with air	air			
Rapid Cooling with		17.05030*	1.02231	.000
water				
Rapid cooling with air		2.91343*	1.02231	.035
Rapid Cooling with	Medium Cooling with	-8.25830*	1.02231	.000
water	air			
Slow Cooling with air		-17.05030*	1.02231	.000

*. The mean difference is significant at the 0.05 level.

The above table illustrates significant differences in crack length across different cooling protocols, given that the mean difference is significant at the 0.05 level. The rapidly cooled group showed significantly less crack length relative to medium and slow cooled groups. The rapidly cooled group in air shows significantly less crack length relative to the group cooled in water. (p value: 0.035)

Tukey HSD

Dependent variable: Fracture toughness

Table 5: Tukey HSD test; Fracture toughness data

(I) group	(J) group	Mean Difference (I- J)	Std. Error	Sig.
Medium Cooling with		1.27490*	.10292	.000
air				
Rapid cooling with air	Slow Cooling with air	1.66890*	.10292	.000
Rapid Cooling with		.51490*	.10292	.000
water				
Rapid cooling with air		-1.27490*	.10292	.000
Medium Cooling with	Slow Cooling with air	.39400*	.10292	.003
air	Rapid Cooling with	-.76000*	.10292	.000
water				
Rapid cooling with air		-1.66890*	.10292	.000
Medium Cooling with		-.39400*	.10292	.003
Slow Cooling with air	air			
Rapid Cooling with		-1.15400*	.10292	.000
water				
Rapid cooling with air		-.51490*	.10292	.000
Rapid Cooling with	Medium Cooling with	.76000*	.10292	.000
water	air			
Slow Cooling with air		1.15400*	.10292	.000

*. The mean difference is significant at the 0.05 level.

The above table illustrates significant increase in fracture toughness relative to slow and medium cooled groups. It is also noted that there is a significant increase in fracture toughness in the rapidly cooled group in air (p value: .000), relative to the group cooled in water.

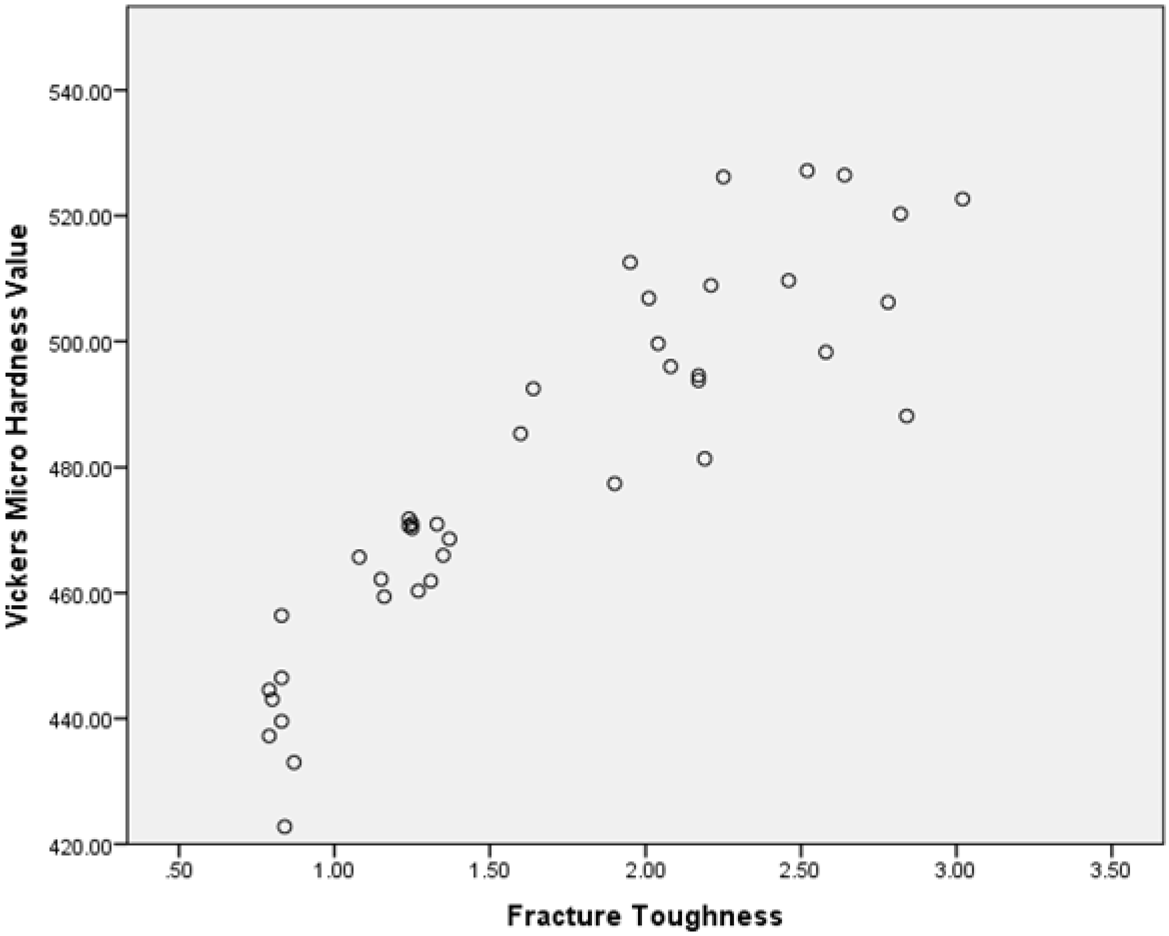

Figure 4: Correlation between Vickers Microhardness values and fracture toughness

The above graph points to a positive correlation between Vickers microhardness and fracture toughness

IV. DISCUSSION

The study concludes that different cooling rates affected the fracture toughness, microhardness values, and thereby the strength of ceramics. As the rate of cooling transitioned from rapid through medium to slow, crack lengths increased and Kc decreased. Following firing of ceramic restoration, different temperature zones form within the porcelain. The inhibition of free expansion or contraction of adjacent areas within porcelain results in formation of residual stress. In slow cooling cases and to some degree in medium cooled ceramics, the outer layer, cooling first, develops tensile stresses while contracting. This tensile stress is compensated by the compressive stresses that develop in the center of the ceramic material, which is the last to contract. But this situation is reversed in rapid cooling cases. During rapid cooling, the outer surface becomes rigid and cannot adapt to changes of interior volume. So, in these cases, compressive stress form on the surface, and tensile stress at the center. The residual compressive stresses on the outer surface of porcelain increases resistance to fracture.

REFERENCES

[1] Callister, W.D. (2007), Materials science and engineering: An introduction (7th ed.; New York: John Wiley & Sons New York).
[2] Rawson, H. (1974), 'Physics of glass manufacturing processes', Physics Technol, 5, 91- 114.
[3] DeHoff, P.H., Anusavice, K.J., and Gray, A.E. (1990), 'Tempering as a means to strengthen porcelain-fused-to-metal restorations', Biomed Sci Instrum, 26, 167-174.
[4] Asaoka, K., Kuwayama, N., and Tesk, J.A. (1992), 'Influence of tempering method on residual stress in dental porcelain', J Dent Res, 71, 1623-1627.
[5] Niwut Juntavee, Wichuda Chinklang, Sirinthip Chaleekler. Effects of different cooling procedures on fracture toughness of feldsparthic dental porcelains. KDJ Vol 2 No.2 July-December, 1999
[6] Morena R, Lockwood P E, Fairhurst CW. Fracture toughness of commercial dental porcelains. Dent Mater 1986;2:58-62.
[7] Anusavice KJ, Lee RB. Effect of firing temperature and water exposure on crack propagation in unglazed porcelain. J Dent Res 1985:64 (Abstract # 1092).
[8] Anusavice KJ,Gray A, Shen C. Influence of initial flaw size on crack growth in air tempered porcelain. J Dent Res 1991; 70:131-6.
[9] Hassan R, Caputo AA , Bunshah RF. Fracture toughness of human enamel. J Dent Res 1981; 4; 820-7.
[10] Baharav H, Laufer BZ, Mizrachi A, Cardash HS. Effect of different cooling rates on fracture toughness and microhardness of glazed alumina reinforced porcelain. J Prosthet Dent 1996; 76:19-22.
[11] Al-Amleh B., Lyons K., and Swain M. (2010), 'Clinical trials in zirconia: A systematic review', J Oral Rehabil, 37, 641-652.