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RESEARCH ARTICLE - MEDICAL TECHNIQUES

Evaluation the Effect of different Cooling Cycles on the Hardness Surface of the Conventional Feldspathic Zirconia

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Abstract

Cooling rate is the main fact in success and life span of all ceramic restoration through its effect on mechanical properties and producing a residual tensile stress, crack propagation and failure restorations. The goals of this study is to assess the impact of diverse cooling cycles (slow cooling – fast cooling) on the surface hardness of the Zirconia (VM9). A total of 30 conventional Y-TZP Zirconia (Vita VM9) disks were fabricated according manufacturers recommendation. The samples were partition into three categories depending on the cooling system. Each group consisted of ten specimens in diameter (2mm×10mm). Control group: samples are unescorted by any change. Fast cooling group: these specimens were fast cooled after second firing ($910C^0$ -$600C^0$) with opening Oven muffle 25% withholding time for 5 minute and remove from the furnace to cool at room temperature. Slow cooling group: specimens were slow cooled after second firing ($910C^0$ -$400C^0$) with opening Oven muffle 25% withholding time for 5 minute and remove from the furnace to cool at room temperature. Each specimen was subjected to hardness test in load 9.8N at 15s using Digital microvickers Hardness tester, Scanning electron microscope. The statistical analysis revealed that, the highest vickers hardness mean value was for the control group (690.57 ± 69.9563) and for second group (618.12± 53.6164) and for third group (631.75±65.3858), The facts were statistically examined by applying ANOVA test (P- value) testes which revealed significant differences($p=0.038$) ($p<0.05$) among groups. Conclusion**:** The impact of cooling cycle on the hardness surface measurements of the Zirconia (Vita VM9) between the three groups was significant. The slow cooling shows a higher value of (VH) Hardness and recommended for Zirconia than the fast cooling.

Keywords: Zirconia; Cooling Rate; Vickers Hardness (HV); Mechanical Properties; Scanning Electron Microscope; Crack

1. Introduction

Yattria stabilized tetragonal zirconia (Y-TZP) was presented in dentistry as framework materials for long span posterior fixed dental prostheses and anterior single tooth restoration due to many reasons' superior mechanical properties, chemical stability, good dimensional, high biocompatibility and high esthetic demand [1]. Zirconia is an occurring in several different forms which accrued at trio crystal structure: monoclinic (from room temperature to 1170°C), tetragonal (1170–2370°C), and cubic (above 2370°C) [2]. There are many factors might effect on the Zirconia phase transformation and mechanical properties such as sintering process, microstructure, heating and cooling rate, grain size, lower thermal conductivity and thermal diffusivity and surface treatment by mechanical manipulation (machining sandblasting, grinding, polishing) [3]. The mechanical characteristics and longevity of all ceramic Zirconia restoration are influenced by cooling protocol [4].The slow cooling for Zirconia restoration was suggested owing to the fact that fast cooling after final firing procedure creating high residual tensile stress and This facilitates crack propagation [5].The hardness test measures the abilities of materials to resist plastic deformation .The mechanical characteristics of dental materials can be proved by implementation a hardness test. This process is done by exerting a constant load via a diamond pointer and then measuring with the help of a screen connected to the device. One of the most current types use for examining the hardness of material is the Vickers Hardness device [6].

Nomenclature		**Symbols**	
ANOVA	Analysis of Variance	µm	Micrometer
FPDs	Fixed Partial Dentures	P	p value
LSD	Least Significant Difference	˚C	Degree centigrade
SPSS	Statistical Package for the Social Science	M	Minute
SD	standard deviation	Maxi	Maximum
Sign	significant	Mini	Minimum
Y-TZP	Yttrium Full Stabilized Tetragonal Zirconia Polycrystal	VAC	Vacum
VHN	Vickers hardness unit		

2. Materials and Methods

2.1. Preparation of conventional feldspathic Zirconia (VitaVM9) discs

Thirty wax discs were made from a sheet of modeling base plate wax with dimensions (2 mm in thickness×10 mm in diameter). Then punched with stainless steel ring to produce the silicon mold for specimens [7]. After applying separating medium on the silicon mold, the Zirconia feldspathic ceramic (VITA VM9) base Dentin powder mixed with VITA modelling fluid by using a clean brush to get homogenized creamy mixed and inserted into the silicon mold with a vibration movement to remove all the excess liquid using absorbent paper and leave to harden before moving samples and removing them from the mold [6]. After the samples are removed from the template, they are entered into a vacuum sintering furnace (VITA VACUMAT 6000 M). The burning schedule has been implemented until the maximum temperature is reached dependent on the industrialist's recommendations as shown in Tables 1 and 2 and applying cooling protocol with the opening oven muffle 25% for 5 minute and remove the samples from the furnace to cool at room temperature for 1 hour as shown in Fig. 1. A rubber bur is used to remove all excess of ceramic samples. In order to have a smooth surface, a ceramic disc from one side were polished by abrading first with wet 600 grit aluminum oxide abrasive paper and finally with wet 1200 grit aluminum oxide abrasive paper by using a grinder- polisher device. After finishing the polishing procedure for all samples, the thickness and diameter of each sample was checked by using digital caliper device [8].

Table 1 Firing schedule of the Zirconia VM9 (first dentin firing)

Onset Temperature Degree (°C)	Drying Time (min)	Rise time (min)	Temperature rate of raise (°C/min)	Definitive temperature (°C)	Holding time (min)	VAC (min)
500 °C	6.00 min	7.27 min	55	925 °C	1.00 min	7.27 min

Table 2 Firing schedule of the Zirconia VM9 (second dentin firing)

Onset Temperature Degree (°C)	Drying Time (min)	Rise time (min)	Temperature rate of raise (°C/min)	Definitive temperature (°C)	Holding time (min)	VAC (min)
500 °C	6.00 min	7.16 min	55	910 °C	1.00 min	7.16 min

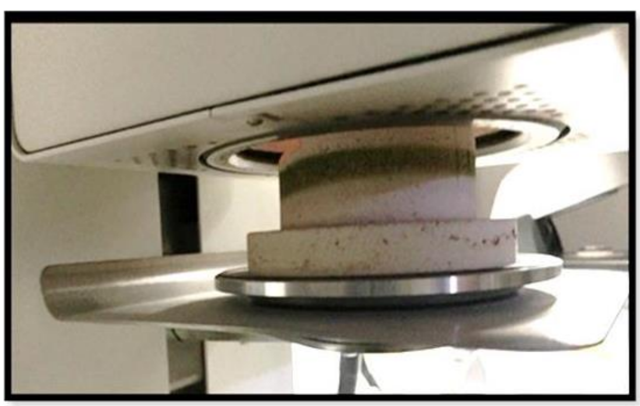

Fig. 1 The oven muffle opening 25%

2.2. Cooling protocols [9,10]

After finishing and polishing, the samples were spitted into 3 sub-groups:

- Control group: n= 10, from 910°C manufacture firing to cooling at room temperature.
- Fast cooling group: n=10, from 910°C manufacture firing to 600°C with opening of the oven muffle 25% holding time 5 min and the samples left to cool at room temperature for 1 hour.

Slow cooling group: n=10, from 910°C manufacture firing to 400°C with opening of the oven muffle 25% holding time 5 min and the samples left to cool at room temperature for 1 hour.

2.3. Vicker's hardness testing

Through the usage of the Vickers hardness device, the surface hardness of the material is measured. The samples are installed on a horizontal platform and the indicator was under load 9.8N for 15s. Samples are measured and the hardness readings appear immediately after the pointer departs. The samples were tested three times (left, middle, right) [11], as appeared in Fig. 2.

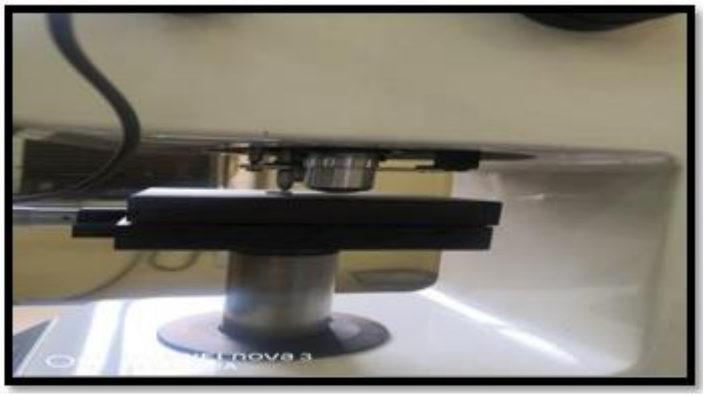

Fig. 2 Vickers diamond pyramid indenter while indent is being placed

2.4. Scanning Electron Microscope (SEM)

In this study, an electronic scanning microscope apparatus was used. One sample was selected from each group and coated with gold to increase conductibility. The surface of each specimen was recorded photographically at different magnification (1000X and 2000X) [11], as shown in Fig. 3 for control group, Fig. 4 for fast cooling group and Fig. 5 for slow cooling group.

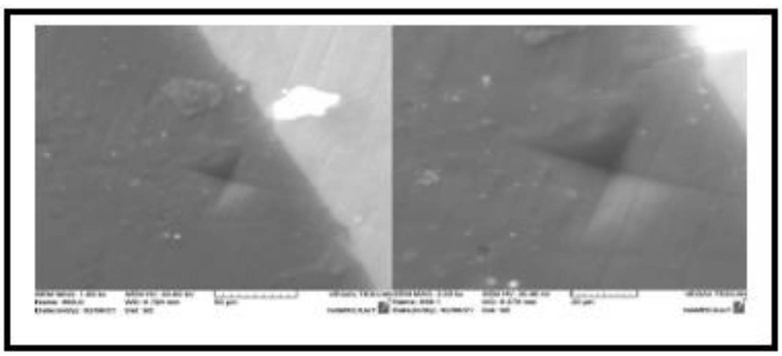

Fig. 3 Scanning electron microscope for Zirconia VM9 control group

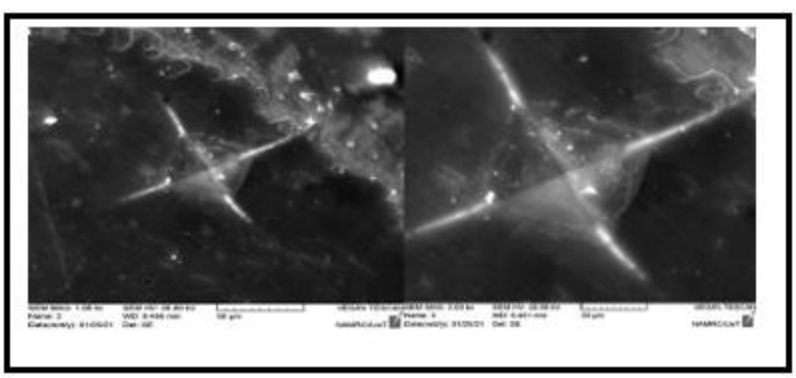

Fig. 4 Scanning electron microscope for Zirconia VM9 fast cooling group

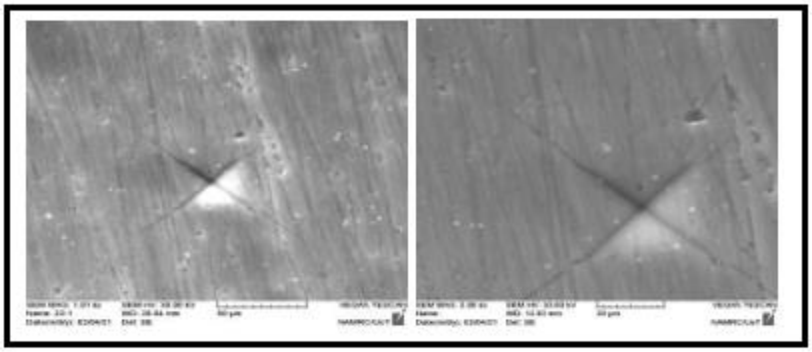

Fig. 5 Scanning electron microscope for Zirconia VM9 slow cooling group

2.5. Statistical analyses

IBM SPSS statistics program Version 21 used One-way ANOVA for done the statistical analysis and significant (P<0.05) of the current study.

3. Result

In this study, the highest mean value of Vickers hardness in Zirconia VM9 was found in control group (690.57 ± 69.9563) while the lowest mean of Vickers hardness was found in fast cooling group (618.12± 53.6164), the statically analysis represent a significant difference (ANOVA test :P= 0.038 , P<0.05) between Zirconia VM9 tested groups as shown in table 3.

Table 3 Mean distributions of hardness test to Zirconia VM9 among tested groups

Zirconia VM9	N	Mean	Std. Deviation	Std. Error	Rang		ANOVA test (P value)
					Mini.	Maxi.	
Control group	10	690.57	±69.9563	22.1221	590	780	P=0.038
Fast cooling group	10	618.12	±53.6164	16.955	549.7	720.5	Sign.
Slow cooling group	10	631.75	±65.3858	20.6768	553.4	735.8	(P<0.05)

LSD test (P-value) in Table 4 showed multiple comparison that there was a significant difference at (P=0.017, P<0.05) between control and fast cooling groups, further showed a significant difference at (P=0.048, P<0.05) between control group and slow cooling group, ultimately appear Non-significant difference at (P= 0.634, P>0.05) between (fast and slow) cooling groups.

Table 4 Less significant difference (LSD test) for hardness test to Zirconia VM9 among tested groups

Zirconia VM9		LSD test (P-value)
Control group	**Fast cooling group**	P=0.017 Sign. (P<0.05)
	Slow cooling group	P=0.048 Sign. (P<0.05)
Fast cooling group	**Slow cooling group**	P=0.634 Non sign. (P>0.05)

4. Discussion

The results of this study showed an increase in VHN was for the control group that start from 910 C^0 (manufacture firing) to cool to room temperature followed by slow Cooling group from 910 C^0 (manufacture firing) to 400 C^0 and fast Cooling group from 910 C^0 (manufacture firing) to 600 C^{0}with significant differences between the among groups P=0.038 Sign.(P<0.05) and the lower VHN was for fast Cooling group from 910 C^0 (manufacture firing) to 600 C^0 , this could be related to decreasing the microstructures porosity and increasing hardness of Y-TZP with increasing sintering temperature during its fabrication. With a processing parameters defects are introducing through the advanced period and are gets worse after sintering. Hence, the ultimate optical and mechanical properties of Y-TZP extremely rely on the preparing methods and parameters, this agreed with [14]. The mechanical properties of zirconia were found to be dependent on its grain size [15].

The prolonged and higher sintering temperatures it causes a development of larger grain sizes which effect on the final stability and consequences the mechanical properties of the zirconia [14]. Depending to the consequences of this study measurements in this in vitro study, there is a significant difference between the conventional control cooling group and fast Cooling group and non-significant difference between fast Cooling group and slow Cooling group. This may be attribute to, Zirconia had three various shapes that appears relying on temperature: monoclinic at room temperature, tetragonal above to 1170°C, and cubic greater than 2370°C. The period of transmissions are able to be turned, and in free crystals are connected with a volume development of about 0.5% in the situation of the cubic to tetragonal transition, and about 4% for the tetragonal to monoclinic transmission. On cooling, the tetragonal → monoclinic transformation is of special interest because sintered products of pure zirconia can break into pieces due to stresses generated by the lattice expansion that accompanies the tetragonal → monoclinic transformations, this excepted with [16]. In this study, the finding explained that may, Volume changes and transformation are sufficient to make the immaculate material unseemly for applications requiring an intaglio solid structure:(cubic to tetragonal Approximation of 2.31%),(tetragonal to monoclinic 3-5%) this indicates that ingredients made of pure zirconium oxide would exploding because of growing in volume grains dimension and tension [16]. So the increasing at grain size it will effect on the hardness of zirconia and reduce it, this is proven by [15].

High compressive stresses consist of volume expansion that can create crack propagation and failure, this may be ascribed to [17]. At monoclinic phase the intense transformation can create a mutilation of materials such as a decreasing in the strength and the fracture of the materials, this supported by [18]. The Zirconia characterize of low thermal conductivity and high thermal expansion coefficient, because of that can be stress accumulation during cooling in zirconia-based composites.it makes these materials very sensitive to thermal shock and the high residual tensile stresses develop with fast cooling zirconia restorations [18]. The compressive stress as a result of rapid cooling veneering porcelain, which might move forward the resistance of the surface layer, but a possibility formation superior tension stress at the zirconia/ porcelain interface [19]. At fast cooling methods formation, a higher values of compressive residual stress, this result may be connected to the elevated discrepancy of the thermal coefficient expansion amongst zirconia infrastructure [4].

The zirconia is a very poor thermal conductor and for this slow cooling is recommend for zirconia restorations, in order to prevent the growth and increase of residual tensile stresses and to provide stability of zirconia-based restorations, corresponding with [20]. Much research supports the theory that residual stresses arise as a result rapid cooling and a slow-cooling protocol advised to equalize the heat dissipation from zirconia and veneering porcelain and to improving the fracture resistance of the veneer [21]. SEM analysis for surface topography of Zirconia VM9 control group as shown in Fig. 3 indicate shortened crack exuding from the corner of indentations were obviously discernible and the crack is very small and its measure 20μm the cracks presented fine linear appearance [22]. Image analysis for (Fig. 4) at the angle of an indentation orthogonal cracks emitting with different crack lengths along the X and Y-axes with the surrounding area affected [22]. For Fig. 5 the picture showed initiated crack originated from the corner of the Vickers indentation due to applied load was also measured in micron (20μm) in a very straight line with a small size and without any distortions [23].

5. Conclusion

In the present study, it was concluded that the control and slow cooling groups were the best and had a greater hardness mean value and are recommended more than the fast cooling groups.

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