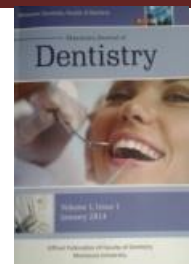




## Influence of Speed Sintering Protocol on Fracture Resistance of Translucent Fixed Restorations .



Rana Ashraf Mohamed Abdel Baset<sup>1</sup>, Walid Abd El-Ghafar Al-Zordk<sup>2</sup>, Mohamed Hammed Ghazy<sup>3</sup>

1Postgraduate student, Faculty of Dentistry, Mansoura University, Egypt.

2Assistant Professor, Fixed Prosthodontic Department, Faculty of Dentistry, Mansoura University, Egypt.

3Professor, Fixed Prosthodontic Department, Faculty of Dentistry, Mansoura University, Egypt.

### Abstract:

**Objectives:** The aim of this *in vitro* study was to investigate the influence of speed sintering protocol on the fracture resistance of the translucent zirconia.

**Materials and Methods:** Forty intact maxillary premolars were used in this study. All teeth were divided according to the type of zirconia into two main groups (n=20): Group K receiving KATANA HTML zirconia and Group Z receiving Zolid fx ML zirconia, Then, each group subdivided according to sintering protocol into two subgroups (n=10): KC and KS refer to KATANA HTML sintered by conventional and speed sintering protocols, respectively, while ZC and ZS refer to Zolid fx ML sintered by conventional and speed sintering protocols, respectively. The teeth were prepared by the aid of dental surveyor. After cementation with self-adhesive resin, the specimens were subjected to thermal cycling for 5000 cycles and mechanically loaded for 120,000 cycles. Then, the specimens were loaded to fracture. Mode of failure was assisted using stereoscopy and scanning electron microscope micrographs.

**Results:** The highest mean value of fracture resistance was recorded in KC subgroup (3083.79 ±844.88) followed by KS subgroup (2864.41 ±692.15) vs ZC (2342.85 ±434.04) and ZS (1829.18 ±299.38) subgroups. Two-way ANOVA showed no interactions between variables (P=.448).

**Conclusions:** Increasing the yttria content had negative impact on zirconia fracture resistance. Although, the sintering protocols did not influence the fracture resistance of the tested zirconia, Zolid fx ML was affected by the speed sintering protocol. Despite of the present results, the tested zirconia can be used safely at premolar area.

**Keywords:** Translucent zirconia, Speed sintering, Fracture resistance.

### Introduction

Nowadays, patients are demanding dental prostheses that combine pleasant esthetics and durable at the same time. For this reason, the ceramic materials are gaining more popularity since they possess tooth-like appearance with acceptable mechanical properties.<sup>1</sup> Among oxide ceramics, Zirconia is gaining more attention due to high biocompatibility, higher mechanical properties with flexural strength 900-1200 MPa and acceptable optical properties.<sup>2</sup> Zirconia is a polycrystalline ceramic, which presents in three different forms depending on the temperature which are: monoclinic, tetragonal and cubic forms. The classical pure zirconia exhibits in the monoclinic crystalline structure at room temperature, which is stable up to 1170°C. Above this temperature, a phase transformation to the tetragonal crystalline structure occurs, which is stable up to 2370°C; beyond that the cubic crystalline structure is derived.<sup>3</sup> Many dopants or stabilizers, particularly yttrium oxide, were added to zirconia in order to stabilize it in its tetragonal form.<sup>3</sup> The firstly introduced zirconia was stabilized by 3 mol% yttria. This generation showed higher mechanical properties and biocompatibility, however, its problem was high opacity so it was used as a framework to be veneered with other translucent ceramic.<sup>4</sup> The main drawback of this generation was the chipping of the veneering material. They explained that the failure could be related to the different

coefficient of thermal expansion between two materials or poor thermal conductivity associated with the poor thermal diffusivity the zirconia substructure.<sup>5</sup> In order to eliminate the risk of chipping, the manufacturers tended to increase the yttria content to approximately 4 and 5 mol% with decreasing the alumina content from 0.25 weight % to 0.05 weight %.<sup>6</sup> This resulted in introduction of monolithic translucent zirconia which provided higher translucency due to better light transmittance that increased by 43% to 45% as the cubic phase increased with less porosity. However, increasing the cubic phase by 25 to 45% resulted in decrease the fracture toughness and flexural strength to 700-800 MPa when compared to the conventional 3Y-TZP.<sup>6</sup>

From a clinical point of view, conventional sintering times for zirconia are time wasting process that may extend to 4 to 12 hours involving slow heating and cooling rates with increased dwell time.<sup>7, 8</sup> This, in turn, extended the treatment times with multiple treatment appointments.<sup>8</sup> Currently, the chairside dentistry is gaining more attention due to decreased time and cost with high productivity and increased patient comfort. This results in introduction of speed sintering protocol that could be achieved at temperature range 1500 to 1600 °C and dwell time of 30 minutes.<sup>9</sup> It is a matter of fact that sintering parameters, either time or temperature, affect the

microstructure, optical and mechanical properties of zirconia.<sup>7,10</sup> A previous investigation included testing many types of monolithic zirconia ceramics. It recorded that increasing the sintering temperature resulted in an improved translucency attributed to the increased grain size with decreased grain boundaries that is considered as light scattering centers, however, it recorded no impact on flexural strength.<sup>11</sup> Even though the increased grain size aim to better translucency, it makes the material more prone to transformation<sup>12</sup> which have a negative impact on its mechanical properties.

This *in vitro* study aimed to evaluate the fracture resistance of different translucent zirconia brands following conventional and speed sintering protocols. The null hypothesis states that there is no significant influence of speed sintering on the fracture resistance of different translucent zirconia fixed restorations.

odontogenic tumor<sup>(3)</sup>. Recently, the status OKC was addressed and classified into the category of cyst. Based on WHO (2017) histological classification of odontogenic tumors and odontogenic cysts<sup>(15)</sup>. Many efforts have been made to understand the pathogenesis of odontogenic cysts, but many of them have been unsuccessful<sup>(16)</sup>.

Blood supply is an essential factor for the growth of odontogenic epithelium. Because there is no vascular system in the epithelium, apoptosis will happen if the connective tissue does not provide the necessary blood supply<sup>(17)</sup>. Angiogenesis occurs in physiologic and pathologic processes including embryogenesis, wound healing, and inflammation<sup>(18)</sup>. In odontogenic cysts, the connective tissue stroma has an essential role in the preservation of epithelial tissues and minor alterations in the epithelium are followed by corresponding changes in the stroma, such as angiogenesis<sup>(17,19,13)</sup>.

#### Material and method :

Forty intact human maxillary premolars freshly extracted for orthodontic reasons with homogenous dimensions and morphology were collected for the present study (ethics approval number: M03080120). Any stain or remnant soft tissues were removed and all teeth were disinfected with diluted sodium hypochlorite solution (Clorox Bleach, Clorox Co., Cairo, Egypt). In order to facilitate handling, the teeth were fixed into blocks of epoxy resin (CMB Kemapoxy 150 3D, AlAhram, Giza, Egypt) that were made with the aid of centralization device then they were kept in distilled water at the room temperature during the study period. An injectable heavy indices were made for each tooth prior preparation to assist the amount of reduction. To achieve standardization of preparation, the dental surveyor (Marathon 103 surveyor, Saeyang company, Daegu, South Korea) was used. The forty premolars were prepared to receive chamfer finishline of 0.5-mm with 1-mm occlusal reduction and 10° tapering of axial walls.<sup>13</sup>

The specimens were randomly divided according to the type of zirconia into two groups (n=20): K and Z where K is the group restored with KATANA HTML zirconia (ECLCN, Noritake, Japan) and Z is the group restored with Zolid fx ML (2002000, AMANN GIRRBACH Germany). Then, each group was subdivided according to sintering protocol into two subgroups (n=10): C and S where C subgroup included specimens sintered by conventional protocol (KC and ZC) and S subgroup included specimens sintered by speed protocol (KS and ZS). Then all specimens were scanned individually by an extra-oral scanner (Medit T300 blue light digital scanner, South Korea) and forty monolithic crowns were designed with aid of CAD software (Exocad DentalCAD, Exocad GmbH, Darmstadt, Germany) where the occlusal thickness was adjusted to be 2.5-mm and 2-mm on the

Mean vascular density (MVD) is a quantitative analysis of angiogenesis, which has been evaluated by using various molecules including: CD31, CD34 and CD105 (endoglin)<sup>(20,21)</sup>. CD105 (endoglein) is a homodimeric cell membrane glycoprotein and is a component of TGF-β receptor complex. This marker is an indicator of endothelial cell proliferation and is up-regulated during angiogenesis<sup>(22,23)</sup>. Moreover, the expression of CD105 is one of the most conspicuous characteristics of newly formed blood vessels; Hence, it is more appropriate to determine MVD<sup>(24)</sup>. Several types of cells are associated with the development of cysts and tumors<sup>(16)</sup>. Among inflammatory cells, mast cells have been considered in growth and expansion of cysts. Mast cells are one of the defense cells of immune system with metachromatic cytoplasmic granules<sup>(25,26)</sup>.

Recently, mast cells were recognized in the pathogenesis of more aggressive pathologic lesions<sup>(27)</sup>. Mast cells have an inhibitory role on the development of pathological lesions. However, stimulatory role of mast cells in the growth of pathological lesions is more prevalent and obvious than their inhibitory effect<sup>(28)</sup>. With respect to several roles of mast cells such as participation in inflammation, degradation of extracellular matrix and bone resorption<sup>(29)</sup>, previous studies have identified mast cells in odontogenic cysts, but there were limited studies about the role of mast cells in the pathogenesis of odontogenic cysts<sup>(30)</sup>. There is a hypothesis that the more aggressive behavior of odontogenickeratocysts is related at least, partly, to distribution of mast cells. However, their pathogenesis and mechanism of expansion and enlargement have not been evaluated<sup>(31)</sup>.

The aim of this study was to determine the density of microvessels and MCs in odontogenic cysts. Correlate the microvessel density with their corresponding mast cells density in the three types of cysts, in order to detect their possible role in the variable behavior of these odontogenic cysts.

functional and non-functional cusp, respectively. Additionally, the restoration thickness was adjusted to be 0.8-mm with cement space 0.06-mm.<sup>14</sup> The design data were converted into processing data and sent to the milling machine (Roland DWX-52D DGSHAPE milling machine, Osaka, Japan) then milling of the restorations was made following manufacturer's instructions.

The restorations were loaded in the furnace tray (inFire HTC speed, Sirona, Germany) and sintered according to the proposed protocols following the manufacturer's instructions of each zirconia type. The restorations were left till complete cooling to avoid cracking. Afterward, the intaglio surface of each restoration was air-borne abraded regarding manufacturer's guidelines using aluminum oxide particles of mean particle size 50-70 μm at 0.2 MPa pressure with 10mm and 45° between nozzle and crown surface for obtaining a rougher surface to enhance durable cementation followed by glazing of restorations. Afterward, a coat of zirconia primer (Z-Prime Plus, Schaumburg, IL, USA) was applied to fitting surface of the restoration and dried for 3-5 seconds. In this *in vitro* study, the cementation was done using adhesive resin cement (DUO-LINK UNIVERSAL adhesive cementation system, Bisco, Schaumburg, IL USA). After application of the cement in the fitting surface, the restoration was gently seated with finger pressure during curing for 2-3 seconds per quarter surface for removing the excess material. The final curing was done after seating each specimen in special loading device in order to apply a load of approximately 950 gm weight on each crown for 10 minutes to achieve seating load of 10 N<sup>15</sup> followed by curing for 40 seconds on each aspect of the crown based on manufacture instructions.

All specimens were subjected to thermo-cycling treatment which was performed for 5000 cycles (1 minute each cycle) in 5 °C and

55 °C water with dwell time 30 seconds<sup>16</sup> using thermocycler (Mechatronic thermo-cycler, JULABO FT200 GmbH, Seelbach, Germany) followed by mechanical loading treatment was performed using a programmable logic controlled equipment (multimodal ROBOTA chewing simulator, Model ACH-09075DC-T, AD-TECH TECHNOLOGY CO., LTD., Germany) where each one was subjected to a 5 kg weight comparable to 49 N of chewing force. The test was repeated for 120,000 cycles which was clinically equivalent to one year of chewing condition.<sup>17</sup> The fracture test was performed through universal testing machine (Instron Universal testing machine Model 3345, USA). Each specimen underwent a load applied on the center of the occlusal surface at the central fossa with a stainless steel ball of diameter 5 mm with a crosshead speed of 1 mm/min until fracture occurred.<sup>2</sup> The fracture load data were recorded and represented on stress-strain curve through computer software (BlueHill Universal software, Instron, USA).

Mode of fracture was classified according to Bruke's classification<sup>18</sup>; Class I where less than half of crown affected, Class II where half of crown affected, Class III where more than half of crown affected and Class IV Severe fracture of the tooth and/or crown. For assisting mode of fracture, stereoscopy and fractographic analysis using scanning electron microscope (JEOL JSM 6510 Iv, Japan) to two representative specimens from each subgroup to detect origin and direction of crack (Figure 1). The

## Results

### Quantitative Data

T student test revealed no statistical difference in mean values of fracture resistance of KATANA HTML zirconia specimens regarding sintering protocol ( $P=0.533$ ) while a significant difference was found in those of Zolid fx ML zirconia specimens ( $P=0.006$ ). The fracture resistance values of conventional sintering protocol between different zirconia types showed a statistical significant difference ( $P=0.024$ ) as well as those of speed sintering protocols ( $P<0.001$ ) as shown in. Two-way ANOVA test revealed

### Discussion:

The present results demonstrate that the speed sintering protocol had a negative impact on the fracture resistance of translucent zirconia specimens regardless the material type (KATANA HTML and Zolid fx ML). For this reason, the null hypothesis, stated that no significant influence of speed sintering on the fracture strength of different zirconia fixed restorations can be partially rejected.

In order to eliminate the risk of veneered zirconia chipping, two types of monolithic zirconia were used for manufacturing the restorations: KATANA HTML and Zolid fx ML. Previous studies revealed that the fracture load values of monolithic zirconia were 3250 to 5600 N.<sup>19</sup> Bankoglu et al (2018)<sup>16</sup> concluded that veneered zirconia restorations showed lower fracture resistance than that of CAD CAM monolithic zirconia restorations. In contrary to the results of Choi et al (2017)<sup>21</sup> whom confirmed that the fracture resistance of the monolithic zirconia restorations (4948.02 ±974.51 N) and the lithium disilicate glass ceramics pressed on zirconia-based restorations (4943.87 ±1243.70 N) did not show a statistically significant difference which was explained that the comparable level of fracture resistance accounts that a higher fracture resistance of FDPs with ceramic core can be obtained from the core material.

data were tabulated and statistically analyzed through t student and Two-way ANOVA tests. Distribution of failure modes was detected using Chi square and Fischer exact tests. Humidifying chamber, followed by incubation with secondary biotinylated antibodies and streptavidin for 15 min each. Di-aminobenzidine was applied to produce brown staining followed by counterstaining with Mayer's hematoxylin. After each step, the slides were put in phosphate-buffered solution (PBS). For the negative control, the primary antibody was eliminated and replaced with PBS.

For immunohistochemical and histochemical counting using the microvascular count technique, according to the method suggested by Weidner et al. (33). MVD and MCD was assessed as the mean number of microvessels and MCs per high power field. The field size for 400 magnification (40 objectives and 10 ocular) was approximately 0.18 mm<sup>2</sup>. Scores of overall CD105 and Toulidine blue expression were represented as mean density/mm<sup>2</sup> ± SD for quantitative variables using SPSS (Statistical package for Social Sciences) software. Comparisons among the experimental groups were done using One-way analysis of variance test (ANOVA) ( $p<0.05$ ). To compare the number of microvessels and MCs between inflamed and non-inflamed DCs and OKCs, independent t-test was used. Pearson correlation coefficient test was used to determine the correlation between MVD and MCD. A p-value of  $<0.05$  was considered statistically significant

that The interaction between the variables of the present study has no effect on the fracture resistance as illustrated in Table 1.

### Qualitative Data

Chi Square test was performed to detect distribution of mode of fracture between the subgroups of the present study and there was no significant difference recorded between the subgroups. Further tests, which are Chi-square test and Fischer exact test, were performed to detect distribution of mode of fracture between: conventional and sintering protocols with the used zirconia types and zirconia type with the sintering protocols.

Although, the used translucent zirconia in the present study showed relatively lower fracture resistance values following the speed sintering compared to that of the conventional protocol, it is still acceptable for clinical use since its strength exceeds the occlusal forces at the maxillary premolar areas which is 222-445 N and increases to 520-800 N during clenching.<sup>22</sup> This is in accordance with the results of a previous study, which concluded that all the tested crowns at the premolar area, whether they were fabricated of traditional monolithic or translucent monolithic zirconia, fractured at higher levels exceeding the maximum occlusal forces.<sup>23</sup>

The results of the current study revealed no significance difference between the fracture resistance of KC and KS subgroups. This was in agreement with the study of Michailova et al (2020)<sup>7</sup> whom analyzed the mechanical properties of conventionally sintered and speed sintered katana STML and found that both groups showed comparable fracture load values after aging, which was related to the identical lattice structure (4Y-TZP). Also, the conventional sintered 4Y-TZP showed decrease in fracture load values following the artificial aging compared to non-aged group which was explained by longer sintering times, especially the dwell time on final temperature and thus a larger area under the sinter curve, the structure had more time to form the grains. While Lawson et al (2020)<sup>10</sup>

concluded that the strength of katana STML was not affected by the speed sintering with mostly unaffected grain size as any of the present tetragonal grains may be over-stabilized leading to no or poor transformation. Additionally, the current results showed a significant statistical difference in fracture resistance of ZC and ZS subgroups. This was in agreement with Jerman et al (2020)<sup>8</sup> investigation which revealed the influence of speed sintering regimen on the flexural strength of Zolid HT<sup>+</sup> restorations and found that they presented the lowest flexural strength compared with the other groups. Their explanation was linked with the tetragonal phase of Zolid HT<sup>+</sup> is supersaturated in yttria with less transformation. On the other hand, Wiedenmann et al (2020)<sup>24</sup> who investigated the impact of different sintering parameters on fracture load of Ceramill Zolid HT<sup>+</sup> crowns and claimed that high speed sintering resulted in a comparable or even higher fracture loads results than that was found in the control group of conventional sintering protocol.

A significant statistical difference was found between fracture resistance results of the zirconia materials used in the present study regarding the two sintering protocols, where KATANA HTML recorded higher fracture resistance values than that of Zolid fx ML in both of conventional and speed sintering. This might be attributed to the different composition and flexural strength of the zirconia material.<sup>6</sup> This was in agreement with Jassim et al (2018)<sup>25</sup> who concluded that the microstructure and composition of the material used in manufacturing CAD CAM monolithic crown significantly influence the fracture strength of the fabricated crowns. The yttria amount present in KATANA HTML is about 5% (flexural strength= 1125 MPa) while it is about 8.5-9.5% in Zolid fx ML (flexural strength= 700 ±150 MPa) as recorded in the manufacture's reports. Although elevation in yttrium oxide percentage provides better translucency, it may result in decreasing in mechanical strength.<sup>12</sup> However, even the high-translucency materials demonstrate fracture strength acceptable for clinical application exceeding 3000 N.<sup>12</sup> El Saka et al (2019)<sup>26</sup> studied the mechanical properties of different zirconia brands including Zolid fx ML and recorded decreased values of flexural strength and fracture toughness compared to 5% yttria-containing zirconia.

However, a previous study did not show a substantial difference between translucent zirconia and traditional Y-TZP in the respect of fracture resistance.<sup>27</sup> Such results might indicate that different types of translucent zirconia differently perform compared to each other, possibly because translucency is gained by using a variety of compositions and production methods, which results in different properties.<sup>28</sup> This leads to contradictory results and makes it difficult to compare different studies as they were testing different brands of translucent zirconia.<sup>28</sup>

The fractographic analysis had illustrated the fracture origin and direction of crack propagation which originated from the occlusal surface at the site of contact with the indenter in the most of specimens. This is supposed to be related to the complex occlusal morphology of the restoration design such as cusp angle and radius of fissure areas.<sup>23</sup> Furthermore, upper premolars have steeper cusps which make them more susceptible to fracture under occlusal

loads.<sup>23</sup> Bergamo et al (2016)<sup>29</sup> study showed a fracture pattern resembling that of the current study. Also, it had recorded an increase in the monoclinic phase following the chewing stimulation with damaging of glaze material and exposing the underlying the Y-TZP surface.<sup>29</sup> The elevation of monoclinic level might be related to the mechanical stresses generated by the contact between the crown surface with the indenter of the chewing stimulator.<sup>29</sup>

However, one of SEM micrographs revealed different fracture origin where it started from the intaglio surface. This might be related to the occlusal thickness as described in a previous study.<sup>13</sup> A second possible cause could be the thickness of the restoration as explained by an investigation of Shahmoradi et al (2020),<sup>30</sup> who claimed that the decreased load of crack initiation in thinner specimens of thickness 0.7 mm may be related to increased flexural and tensile stress on the intaglio surface of the crown which can potentiate the crack initiation.<sup>30</sup> A previous study showed that the calculated tensile stresses at the internal surface of the loaded and bonded ceramic crowns increased proportionally to cement thickness (8 times, from 50 to 500 µm).<sup>31</sup> The cement effect could be attributed to the lower elastic modulus compared to that of the ceramics (6.3 GPa and 64 GPa) and the resin cement shrinkage stresses could be more influential by increasing the cement thickness.<sup>31</sup> In conclusion, Polymerization shrinkage might cause tensile stresses in the internal axio-occlusal line angles of ceramic crowns, that would be enough to cause the fracture of the crowns with thicker cement.<sup>31</sup>

**Conclusion:** Within the limitations of this in vitro study, the following conclusions could be drawn:

1. The fracture resistance values of the tested zirconia exceed the biting forces values, Thus, they can be used safely in the premolar region.
2. The fracture resistance of 8 mol% Y-TZP (Zolid fx ML) is lower than that of 5 mol% Y-TZP (KATANA HTML).
3. Even though the sintering protocols did not influence the fracture resistance of the tested zirconia, the 8 mol% Y-TZP (Zolid fx ML) was negatively affected by the speed sintering protocol.

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**Table 1. Two-way ANOVA test**

Material (KATANA HTML and Zolid fx ML),  
Sintering (Conventional sintering and Speed sintering).

Source	Type III Sum of Squares	df	Mean Square	F	P-value
Corrected Model	9.447E6 <sup>a</sup>	3	3149000.56	8.563	.001*
Intercept	2.560E8	1	2.560E8	696.290	.001*
Material	7887038.481	1	7887038.41	21.448	.001*
Sintering	1343449.740	1	1343449.740	3.653	.064
Combined material * sintering	216513.567	1	216513.567	0.589	.448

a. R Squared = 0.416 (Adjusted R Squared = 0.368)

**Figure 1.** Micrograph×1000 crack origin and propagation direction in Class I fracture mode specimen.

