Fracture Toughness of Two Types of All-ceramic Restoration after Repeated Firing

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Abstract
Background The ability to measure and describe changes in the mechanical properties of ceramics due to various thermal handling protocols will provide a better understanding of the mechanical performance and application of dental ceramic materials. **Objectives** To evaluate and compare the fracture toughness of two types of all-ceramic restoration (Zirconia and IPS e. max (Lithium disilicate) CAD) after repeated firing. **Materials and Methods** Sixty all ceramic samples (in final dimensions of 18 mm Length, 4 mm Width, and 3 mm Thickness) were prepared and divided into two main groups according to the used materials; Group (Z) for Zirconia and Group (E) for IPS e. max CAD. Each main group subdivided into three subgroups according to firing cycles. The (Z) group subdivided into ZF3, ZF5 and ZF7; And group (E) also subdivided into EF3, EF5 and EF7 for three, five and seven firing cycles respectively. Then, the samples were subjected to the indentation strength using a Micro-Vickers Hardness Tester device. After completing of the firing process, the samples were subjected to the fracture toughness test for each specimen. Scanning electron microscope (SEM) images were taken for one random specimen for each subgroup. **Results** The data showed the highest fracture toughness were obtained with EF5 and the lowest fracture toughness with ZF7 and the P-value of ($P \leq 0.05$). LSD test done and revealed a highly significant difference between the groups. **Conclusion** generally, E. Max. CAD material may verify best results of fracture toughness test compared with Zirconia material after repeated firing cycles.

Keywords Fracture toughness, E. max CAD, Zirconia, Repeated firing, Manual milling.

Introduction
With growing awareness of esthetics and biocompatibility, patients increasingly request metal-free solutions (Reich et al., 2005). Due to the successful use of all-ceramic crowns both in the anterior and posterior segments (Pröbster et al., 1997, Fradeani et al., 2005), and with the introduction of advanced dental technology and

high-strength ceramic materials, all-ceramic systems may become a viable treatment option even for extended fixed partial dentures (FPDs). Such restorative all-ceramic systems must fulfill biomechanical requirements and provide longevity similar to metal-ceramic restorations (Denry, 1996, Derand et al., 2005) while providing enhanced esthetics (Raigrodski and Chiche, 2001). Many ceramic systems, which may differ in composition or fabrication technique, are available. Two of the clinically well-spread materials are the lithium disilicate and zirconia-based systems. They can be fabricated from prefabricated blocks, which are milled with a computer-aided design/computer-aided manufacturing (CAD/CAM) system (Raigrodski, 2004). Zirconia ceramics have been extensively studied because of their excellent mechanical properties, which are much greater compared with those of other dental ceramics (Aboushelib et al., 2007). Nowadays, a high strength zirconia used in fixed partial denture even in load-bearing area (Komine et al., 2005, KARATASLI et al., 2011), which is present in either partially sintered or fully sintered zirconia and it seems stronger than other types of ceramic (Subasi et al., 2012). Zirconia came in different sizes and shapes according to the type of restoration and the number of units to be milled (Parker, 2007). Lithium disilicate is a glassy ceramic that has 70% crystalline phase, and consists of quartz, lithium dioxide, phosphor oxide, alumina, potassium oxide and other components. This claim to have optimum esthetics, natural light refraction, high fracture toughness and high flexural strength in the range of 360-400 MPa (Alqahtani, 2016). Surface flaws and defects which may develop as a result of thermal, chemical or mechanical processes (Albakry et al., 2003).
As mechanical failure is always associated with a crack-initiation/crack-propagation process, fracture toughness values could be useful in comparing different ceramics and possibly predicting clinical performance (Alkadi and Ruse, 2016). Fracture toughness, defined as the resistance to fast crack propagation at a critical stress level, is widely used for mechanical characterization of dental ceramics (Azevedo, 2011, Wang et al., 2014).It has been demonstrated that the microstructure, and consequently the mechanical properties of a glass ceramic can be modified by varying the thermal treatment to which it is submitted (Milleding et al., 2002). Thus the effects of firing times, temperatures and cooling rate during the fabrication process should not be ignored (Tang et al., 2012). To achieve improved contour, color, and aesthetics, multiple firing procedures are necessary for the fabrication of all-ceramic restorations, especially when using the standard layering technique to match the esthetics of natural dentition (Rayyan, 2014).

Materials and Methods:

Samples preparation
Sixty bar all-ceramic sample of (18 mm length, 4 mm width, and 3 mm thickness) were prepared according to the ISO standardization 6872:2008 (ISO, 2008). Figure (1) shows the prepared all-ceramic specimen dimensions for fracture toughness test. Manual milling was accomplished in the fabrication of requested samples. The samples were grouped according to materials used, Zirconia (HT, VITA Zahnfabrik, Germany) for (Z) group and IPS e. max CAD (Ivoclar/Vivadent, Liechtenstein, Germany) for (E) group as shown in Figure 2 (A and B). After the preparation of the bar shaped sample, all specimens were polished with rubber disc burs (Renfert-GmbH, Germany) to the final dimensions. Finally, the specimens were cleaned completely from any

remaining dust and any trace of fingerprint with 70% ethyl alcohol, acetone and distilled water for (10) minutes by using a digital ultrasonic cleaner and then dried with a clean tissue (Tarib et al., 2016).

Samples grouping

The total sixty all-ceramic sample (zirconia and e. max CAD) that prepared for this study were grouped according to the used materials into two main groups, Zirconia and IPS e. max CAD. Then, the sample sub-grouped according to the number of firing cycles into three, five and seven firing cycles. Each main group composed of thirty specimens (n=10) and symbolized as (Z) for Zirconia and as (E) for IPS e. max CAD. According to the firing cycles, group (Z) is sub-divided into ZF3, ZF5 and ZF7, and group (E) sub-divided into EF3, EF5 and EF7 for three, five and seven firing cycles respectively.

Experimental procedure

Indentation strength test was performed using a Micro-Vickers Hardness Tester device (TH714, South Korean). Each specimen was placed on the stage of the device under the indenter at a distance of 2-3 mm. Following the instructions provided with the device by adjusting its stage horizontally. The Vickers indenter was then descended, at a low speed, till it touched the polished surface of (IPS e. max CAD and Zirconia) specimen. The fracture toughness was determined by indentation technique. One indentation was made on each sample with a load of 9.8 N for 20 sec. (Scherrer et al., 1998). After that time period, the load was automatically relived by the device. Crack resulting from the Vickers indentation were measured immediately to avoid slow crack growth, The images of the cracks were taken with a digital video camera model (TH714, South Korean) coupled to the hardness tester (Domingues et al., 2016). In firing cycles, all specimens were subjected to the heat treatments in a ceramic furnace (P3000, Ivoclar Vivadent, Schaan, Liechtenstein, Germany). The specimens were fired for 3, 5, and 7 times for each main group material (Milmine, 2014). For surface topography, a scanning electron microscopy (SEM) was used. One specimen was chosen randomly from each main group before heat treatment and one from each sub-divided groups after the firing process. All the chosen specimens were attached to the aluminum holder in a plasma gold-coating device for painting their surface with pure gold for imaging procedure, and the size of particles were of (20 nm). Finally, the fracture toughness test was accomplished using the Instron testing machine (China), and the applied test was performed with a crosshead speed of 0.5 mm/min. Due to the small size of the all-ceramic specimen, a holder for testing apparatus was fabricated to minimize the error raise during the testing procedure.
The bars of all-ceramic specimens (Zirconia and E. max CAD) subjected to fracture toughness test by exerting tensile stress on the indented surfaces until fracture. All the specimens were placed symmetrically on the fixtures where the loading pin in the center of the crack that made. The fracture toughness was calculated using the following formula adopted by Anstis et al, 1981 (Anstis et al., 1981) (Chantikul et al., 1981).

KIC= 0.016 (E/H)1/2 (P/C)3/2 were: -
KIC= Fracture toughness (MPa.m1/2).
0.016 is a material-independent constant.

E= Modulus of elasticity of the tested material (GPa).
H= Vickers Hardness (GPa).
P= Indentation load (N).
C= Crack length (m) (measured from the middle of the vickers indentation).
Finally, the mean KIC values were calculated for each experimental group and were tested for statistical significance.

Statistical Analysis

The study data were analysed using SPSS (V-22). The statistics of descriptive data analysis was used with a significant P-value of ($P \leq 0.05$) and a confidence level of (95%). Also, inferential data analysis of LSD test was used.

Results

In the table (1) and Figure (3), results show that in the zirconia group a ZF3 subgroup accounted the highly level of fracture toughness, followed by ZF5, and finally the ZF7 which recorded the lowest mean value comparing with the other groups. While for e-max group, the subgroup of EF5 recorded the highest value in fracture toughness, followed by EF3, and then EF7 which recorded the lowest mean value of fracture toughness in e-max group. Also the results show that e-max group recorded the highest mean value compared with Zirconia group after repeated firing cycles.

Table 1: Probable Pairs wised comparisons using (LSD) test among studied groups for (Fracture Toughness test).

Dependent Variable: Toughness				
(I) Group	(J) Group	Mean Diff. (I-J)	P-Value	Sig.
ZF3	ZF5	0.903	0.014	S
	ZF7	1.550	0.000	HS
	EF3	-0.727	0.046	S
	EF5	-1.293	0.001	HS
	EF7	-0.210	0.558	NS
ZF5	ZF7	0.647	0.075	NS
	EF3	-1.630	0.000	HS
	EF5	-2.197	0.000	HS
	EF7	-1.113	0.003	HS
ZF7	EF3	-2.277	0.000	HS
	EF5	-2.843	0.000	HS
	EF7	-1.760	0.000	HS
EF3	EF5	-0.566	0.118	NS
	EF7	0.517	0.152	NS
EF5	EF7	1.083	0.004	HS

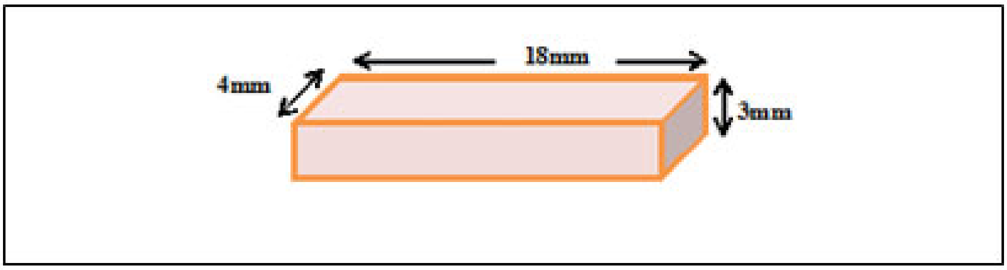

Figure 1: The prepared all-ceramic sample dimensions.

Figure 2: Material used in this study: A- Zirconia block, B- IPS e. max CAD blocks.

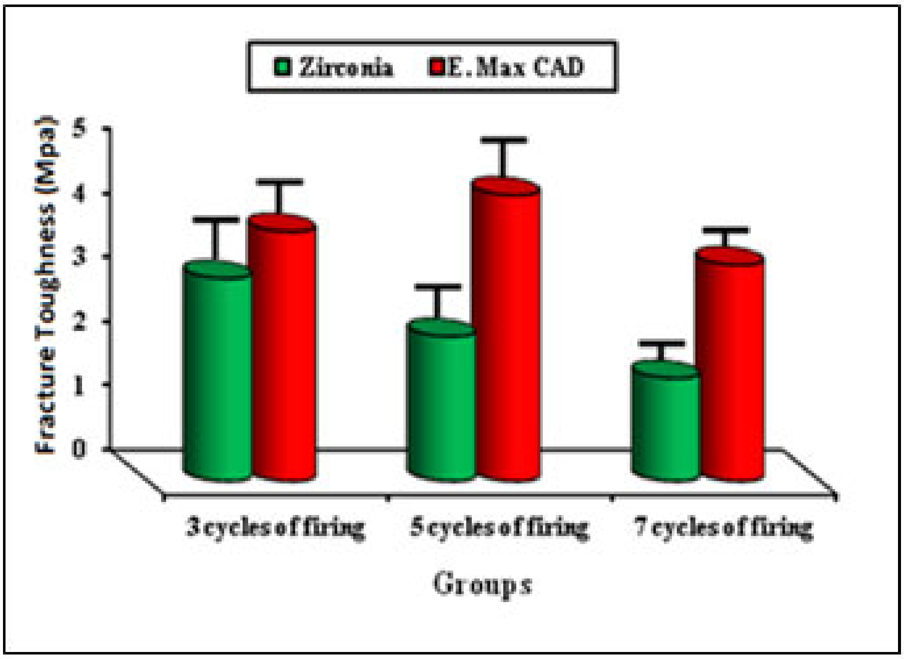

Figure 3: Mean values for Fracture Toughness test distributed in different groups.

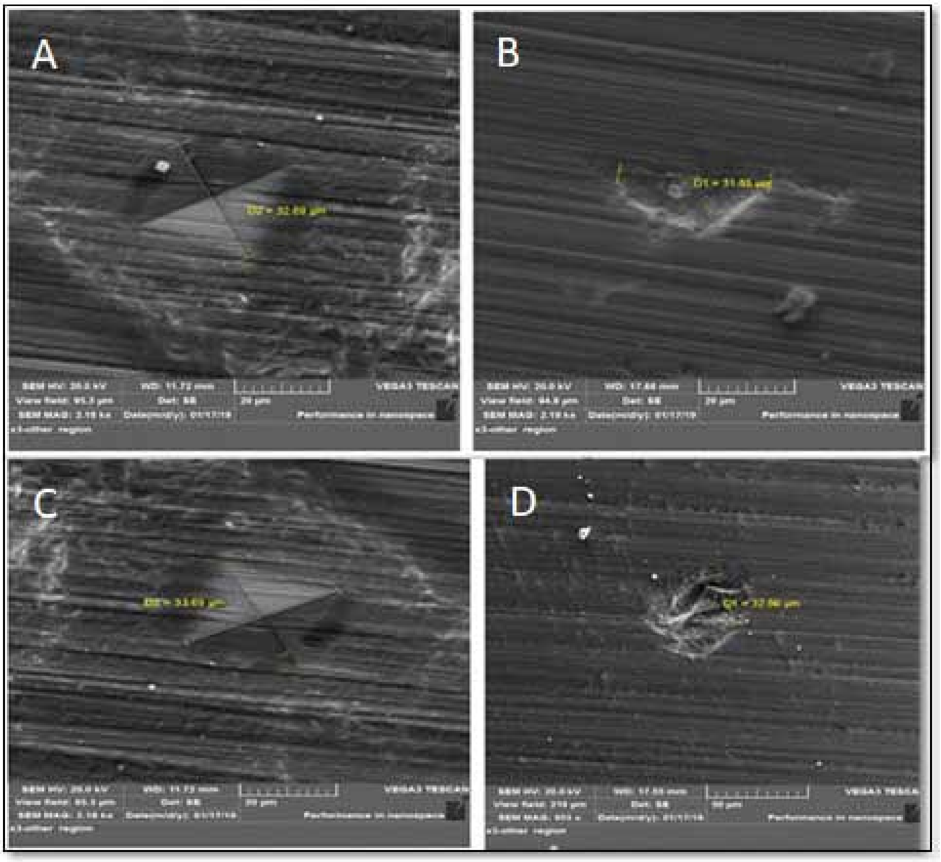

Figure 4: SEM image for both materials: A- Zirconia without firing. B- Zirconia with 3 cycles of firing C- IPS e. max CAD without firing. D- IPS e. max CAD with 3 cycles of firing.

Discussion

The purpose of our study was to evaluate the effect of multiple firings on the fracture toughness of the zirconia and e. max CAD restoration. The results of the present study support the null hypothesis that there are significant differences in the fracture toughness between the zirconia and e. max CAD restoration after repeated firings. All ceramic materials have proven to be the fastest growing product category. In the early 1990s, the introduction of zirconia as a dental material has generated considerable interest in the dental community. Since then, zirconia has been widely used to build restorations because of its good chemical properties, dimensional stability, high mechanical strength, toughness, and young›s modulus which similar to that of stainless steel alloy (Guazzato et al., 2004, Chevalier and Gremillard, 2009).

Zirconia Restoration Group (Z)

In the current study findings, the mean of the fracture toughness after three firing cycles of zirconia sub-group (ZF3) is about 3.148 Mpa, with five firing cycles (ZF5) of 2.245 Mpa. On the other hand, that of the seven firing cycles (ZF7) was 1.598 Mpa. The fracture toughness of zirconia with three firing cycles was higher than that of zirconia with five and seven firing cycles. This may be due to the density of tested materials that increased may be due to the number of crystals increased, and the porosity decreased may be due to the high crystallinity lead to interlocking of microstructure

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of these ceramics so the porosity will be decreased. There for the crack decreased as shown in SEM images Figure 4 (A and B). The results of this study findings may in agreement with (Tang et al., 2012) that concluded that there were no major effects found in fracture toughness after repeated firing.

IPS e. max CAD Restoration Group (E)

In these group findings, the mean of the fracture toughness after five firing cycles (EF5) was of 4.441 Mpa, and after three firing cycles (EF3) was of 3.875 Mpa. While after seven firing cycles was of (EF7) 3.358 Mpa. A highly significant difference in fracture toughness was noticed between the IPS e. max CAD subgroups. The fracture toughness of e. max CAD with five firing cycles was higher than that of three and seven firing cycles. This increased variability of fracture toughness results at EF5 may be related to the capacity of some material for being disturbed by repeated firing cycles. A possible cause is the depletion of the glass phase by crystallite growth, as it is a crystal of a very similar stoichiometry, depleting the glass phase (Milmine, 2014). As the glass phase has CTE higher than that of crystal phase, this may put the glass in tension (Watts and Satterthwaite, 2008), which along with a possible increase in CTE of glass phase may set up a critical residual stress in glass phase resulting in the observed increased variation of mechanical testing results (Fracture toughness) as shown in SEM images Figure (4)C and D. The current findings may show an agreement with (Milmine, 2014), they found that there was no change in measured fracture toughness of IPS e. max CAD ceramic under experimental conditions, however, the coefficient of variation increase for fracture toughness more variability after five cycles possibly increasing the likelihood of premature failures.

Comparing between Zirconia (Z) and IPS e. max CAD (E) restorations

According to the results of this study, it could be concluded that E. Max. CAD material has recorded the highest fracture toughness value compared with that of Zirconia material. This is probably due to the IPS e. max CAD porosities which smaller than in a competitor's products. The effect of material porosity could cause stress concentration and strength-limiting fractures, and this also appear to be minimal since there are almost no porosities when viewed under SEM at the 100 μm level (Albakry et al., 2003). In addition, during the manufacturer procedures of IPS e. max CAD, the ceramic may undergo a number of phase changes. Initially, the material is a transparent glass that consequently undergoes crystallization into a blue coloured metasilicate phase before the final firing cycle transforms the crystals into a tooth colored disilicate ceramic phase. Following the final crystallization firing, it is approximately 70% crystal and 30% glass phase (Watts and Satterthwaite, 2008).

To counter this, they achieved a coarser and stronger microstructure by increasing the nucleation temperature from 650 ^{o}C to 700
C for the precursor metasilicate crystal. Thus the microstructure appears to affect the strength. This findings could in agreement with (Milmine, 2014) who reported that the high strength and toughness is a result of the high crystallinity and interlocking microstructure of these ceramics. However, the Zirconia restoration material has lower fracture toughness due to larger porosities than IPS e. max CAD, and the increase in the hardness. Also with the increase in the number of firings, although no observable difference was found in crystallization in the zirconia material. This might

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agree with (Tang et al., 2012) that concluded that there were no major effects found in fracture toughness after repeated firing.

Conclusion

Within the limitations of this study, it could be concluded that
1-E. Max. CAD material has higher fracture toughness compared with Zirconia material after repeated firing cycles.
2-The IPS e. max CAD with five firing cycles has the best fracture toughness than that of three and seven firing cycles.
3-The Zirconia with three firing cycles has the best fracture toughness than that of five and seven firing cycles.

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