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*Original Research Article*

# Hardness and Fracture Toughness of Heat Pressable and Machinable Dental All- Ceramics

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## ABSTRACT

All-ceramic restorations are one of the most esthetically pleasing prosthodontics restorations. Because there is no metal to block light transmission, they can resemble natural tooth structure better in terms of color and translucency. Their chief disadvantages are high hardness that adversely affects the natural teeth and brittleness that affect susceptibility to fracture.

The aim of this study was to evaluate the hardness and calculate the fracture toughness of the most commonly used All-ceramic materials fabricated by different techniques and compared them with conventional feldspathic porcelain.

Heat pressing and CAD/CAM All-ceramic materials are used to fabricate eight disc-shaped samples of each material. The hardness of the samples was determined and their fracture toughness was calculated. Feldspathic porcelain fabricated by powder compaction technique served as control.

The results of hardness showed that Control Group A (sintered) exhibited slightly higher significant VHN value when compared to Group B (heat pressed). Group C (CAD/CAM) recorded the highest significant VHN hardness value. Regarding fracture toughness, Groups A and B showed comparable values with no significant difference. Again, Group C recorded the highest significant $K_{C1}$ value when compared to A and B groups.

It could be concluded that the sintered dental porcelain has VHN and $K_{C1}$ values comparable to those of heat pressable ceramic and this attributed to quitting of veneering procedure during preparation of in-vitro lab specimens. Meanwhile, CAD/CAM ceramic has the highest significant VHN as well as $K_{C1}$ values in comparison to other groups.

***Keywords:*** Hardness, Fracture toughness, Heat pressable ceramics, Machinable ceramics.

## INTRODUCTION

All-ceramic veneers and crowns are one of the most esthetically pleasing prosthodontics restorations. Because there is no metal to block light transmission, they can resemble natural tooth structure better in terms of color and translucency than any other restorative option. Their chief disadvantages are high hardness that adversely affects the natural teeth and brittleness that endorse susceptibility to fracture. Clinically, all ceramic restorations are subjected to masticatory forces. These stresses act on the brittle materials and could ultimately compromise their durability. [1,2]

Hardness is a characteristic feature of a solid material expressing its resistance to permanent deformation. Meanwhile, toughness is the maximal amount of energy a material can absorb before fracture, which is different than the ultimate force magnitude that can be applied. In reality, fracture toughness is the elastic and plastic

deformations ability that allows materials to absorb large amounts of energy before failure. Dental porcelain is highly brittle material with low fracture toughness. [3]

The correlation between microstructural characteristics and fracture toughness supports theoretical predictions. Numerous strengthening conceptions are directed to expand fracture toughness of all-ceramic restorations via different modalities such that modify the chemical composition and/or alter the microstructure. Despite the chemistry of dental ceramics definitely plays a significant role the high percent crystallinity of dental ceramics and large grains could enhance fracture toughness (KIc); however, the microstructure alteration alone has a limited effect. [4]

Vickers indentation of ceramic produces cracks could be used to determine the fracture toughness of that ceramic. The obtained toughness value is within 10% of the typical values reported using standard fracture mechanics samples, demonstrating the viability of using such a method for toughness measurements; [5] These indentation techniques for assessing fracture toughness are attractive due to the simplicity and expediency of experiments, and because they potentially allow the characterization of both local and bulk fracture properties of biomaterials and biological hard tissues; [6] *In 2003 Albakry et al.*, [7] evaluated the fracture toughness and hardness of three pressable all-ceramic materials with two different techniques; indentation fracture and indentation strength. They proved the reliability of both methods. One year later, they extended their study to compare the strength and fracture toughness of the three hot-pressed glass-ceramics (IPS-Empress, Empress 2 and a new experimental ceramic) with alumina glass-infiltrated ceramics (In-Ceram Alumina), processed by both slip casting and dry pressing. This investigation provided the clinician with data regarding strength, fracture toughness of a broad range of the currently materials. [8]

Based on the previous review, the brittleness of dental porcelain paid the attention firstly to enhance the fracture toughness and secondly required to think about how to evaluate the fracture toughness of these materials. One of the most feasible techniques is through indentation measurements. Hence, this study was aimed to evaluate the hardness of heat pressable and machinable All-ceramic via Vicker's indentation and consequently calculate their fracture toughness; then, compare the resultant data with those of conventional feldspathic porcelain.

## MATERIALS

Two commercially available All-ceramic materials; heat preesable and CAD/CAM were investigated in this study. In addition, conventional feldspathic dental porcelain fabricated by sintering was employed as control. The materials used in the study are listed in the table below.

**Table1: The investigated materials used in this study**

| Materials ( Trade Name) | Fabrication Technique | Manufacturing |
|---|---|---|
| VITAVM®13 | Sintered feldspathic porcelain | Vita Zahnfabrik H Rauter, GmbH & Co. KG, Germany |
| VITAPM®9<br>IPS e.max Press Ingots | Heat Pressable Ceramic | |
| Cercon® | CAD/CAM Ceramic | DeguDent, GmbH, Rodenbacher, Chaussee. 463457 Hanau-Wolfgang, Germany |

## Methods:

### *Fabrication of conventional feldspathic dental porcelain discs*

A circular copper mold of dimensions 15 mm diameter and 3 mm thickness was machined. The mold was sectioned into two halves and reassembled by two screws for easy and safe removal of the specimens after processing.

Five disc-shaped glass-ceramics specimens were prepared using the copper

mold. The powder and liquid of the conventional feldspathic dental porcelain (Vita VM13; Vita Zahnfabrik) were manipulated according to the recommendation of the manufacture. Using a brush, sufficient distilled water was added to wet the powder. The wet powder was compacted into the mold against a glass slab then compressed with an acrylic resin plunger with light tapping forces. Later, excess moisture was removed with an absorbent tissue.

After condensation, the porcelain specimens were dried in front of a heated furnace to extract the remaining moisture. Preheating step was accomplished by putting the packed porcelain into the furnace (Vacumat 40T; Vita Zahnfabrik), under 480°C for 2 min. till it reached the leathery state. A firing cycle with a heat rise of 25°C/min to 890°C under vacuum for 17 minutes was preformed, firing temperature was held for 2 minutes, and then allowed to cool to 350°C with muffle mostly opened, then held for 10 minutes. Then, another layer was then applied over the first layer and the specimen was placed again in the porcelain furnace and re-fired similarly. Before the subsequent glaze firing, all specimens were cleaned to remove any dirt or grease using ultra sonic cleaning in distilled water for 2-3 min. (Sweep Zone Technology, L & R Ultrasonic, Kearny, NJ), and the specimens were then allowed to dry in air . A self-glazing was performed in the porcelain furnace at 500°C for 16 min.

***Fabrication of IPS e.max heat pressable specimens:***

Five wax disks, 15 mm in diameter and 5 mm thick, were cut from modeling wax sheets. All wax patterns disks were trimmed and adjusted with the copper mold to be in the same thickness and radius as the conventional porcelain specimens and then sprayed with Sure Take surfactant (Ivoclar Vivadent) to reduce surface tension. The phosphate-bonded investment (Sure Vest Quick High Heat Investment, Ivoclar Vivadent) powder (100g.) was mixed with the provided special liquid and distilled water under vacuum at high speed for 1 min. to remove the air bubbles (Vacum Mixer, Ivoclar Vivadent). For spruing, 8-gauge wax with a diameter of 3 mm was angled in order to insure non-turbulent flow of the viscous ceramics during the subsequent pressing, and the attachment point was rounded with no angels and edges. The IPS Silicon Ring was sprayed with a lubricant to be ready for receiving the unset investment. As recommended by the manufacture, the wax patterns discs were weighed to determine the size of the porcelain ingots to be used during the pressing. The IPS Silicone ring was positioned such that surrounding the sprued wax specimens. Careful attention was taken not to damage or deform any part of the wax patterns disks.

After complete setting of the investment; 30 minutes, IPS Silicone ring was pushed out carefully. The investment ring was placed into the burnout oven to eliminate the wax rapidly where the temperature was raised upto 850°C in 1 min.

For pressing, the cold IPS e.max plunger was first dipped into the IPS e.max Alox Plunger Separator, and then a cold IPS ingot was inserted into the hot investment ring with ingot face upward ready to be pressed by the plunger. Then, the ring was placed at the center of furnace, and the selected furnace program (EP500) was started. After the hot pressing was completed, the ring was removed from the furnace, and placed for one hour on a wide-mesh cooling grid till reaching room temperature. Afterward, the Alox Plunger was removed from the investment by marking the length on the outside of the investment and using a separating disk to remove the investment-encased plunger. The investment material around the pressed specimen was removed using a polishing jet at approximately 4 bar pressure such that the pressed specimen was not visible. The blasting pressure was then reduced to approximately 1-1.5 bar, to blast the

invested specimens carefully without damaging them.

The pressed specimen were finished using a dental rotary instrument (Alumina stones: Brown, pink, white, Brasseler, USA) with low pressure and without overheating. All specimens were then carefully sandblasted, cleaned under running water and dried thoroughly using oil-free air.

***Cercon® CAD/CAM Specimens' Preparation:***

Cercon art is a CAD/CAM hardware and software system developed for the virtual design of dental crowns and bridges in the dental laboratory. It allows production of crown and bridge frameworks using one of two different methods: the familiar "classical" method or the virtual design method using the CAD module.

However, in this investigation, the Cercon ingots were trued to a diameter ≃ 10.5 mm. A block was sectioned with a slow speed saw (Isomet; Buehler, Germany) to make five specimens with a thickness ranging from 3.5-4.5 mm. The thickness of each individual specimen was measured with a dial caliper (Mitutoyo, Japan) to the nearest 0.01 mm.

***Measurements:***

Vicker's Hardness Number (VHN) of each group' samples; conventional porcelain, IPS e.max and Cercon were determined using Vicker's Hardness Tester (Tukon 1102 Buehler, Germany). A load of 500 g. for duration of 15 s. was applied to Vickers diamond pyramid indenter (a square pyramid with opposite faces at an angle of 136° and edges at 148° and face angle 68°). The obtained *VHN* was following equation [1]:

$VH = 1.8544 \times 2P / d^2$

Where *d* is the length of the diagonal in mm measured from corner to corner and *P* is the load in kg; Figure (1).

Then, the applied load was raised to 1000 g. to make a crack originates from the corners of the indentation. The originating crack should be more than double length of the diagonal; Figure (2). The characteristic length (c) of the indentation diagonal in micron (2a) was measured. The initiated crack originated from the corner of the Vickers indentation (2c) due to greater applied load was also measured in micron such that 2c were more than double 2a; Figure (3). Then, the fracture toughness was calculated by the following formula: [9]

$K_{IC} = (1/\pi^{2/3} \tan \Psi) P/c^{2/3}$

Where $K_{IC}$ is fracture toughness, $\Psi$ is the half of the angle of Vickers indenter ($68^o$), P is the load and c is the crack length.

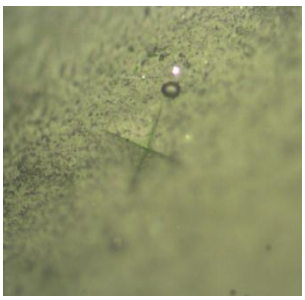

**Figure (1): The indentation of Vicker's Hardness Tester**

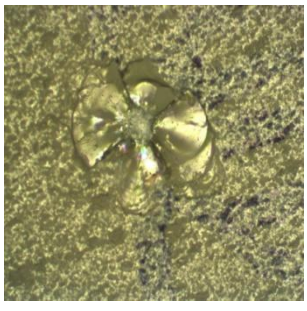

**Figure (2): The originated cracks from the corner of the indentation mark having double length of the diagonal**

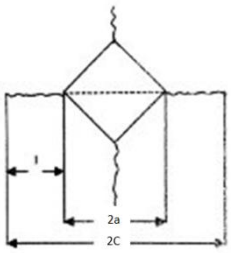


**Figure (3): A diagrammatic representation of the characteristic length of the crack**

**Statistical Analysis:**

Data of hardness and then the calculated fracture toughness results were collected for each investigated group; tabulated and statistically analyzed. One way ANOVA test was used at P level equal

to ≤0.01. The comparison among groups was measured by the independent T-test using SPSS program

## RESULTS

Table (2) shows the results of the present investigation. VHN of machinable ceramic (Group C) recorded the highest significant value compared to both control (feldspathic porcelain) and heat pressable ceramic; Groups A and B respectively. Also, VHN of control group (A) was significantly higher than that of heat pressable ceramic group (B), Figure (4).

Regarding fracture toughness, Groups A and B exhibited comparable values with no significant difference, while Group C recorded the highest significant value compared to groups A and B; Figure (5).

**Table (2): The descriptive statistical analysis data of (VHN) and ($K_{IC}$) of the different investigated groups**

| Groups<br><br>Property | A<br>(feldspathic porcelain)<br>Mean ± S.D. | B<br>(Heat pressable)<br>Mean ± S.D. | C<br>(Machinable)<br>Mean ± S.D. |
|---|---|---|---|
| Hardness (VHN) | 757.44±117.47<br>b | 656.37±39.74<br>c | 1185.69±63.48<br>a |
| Fracture Toughness | 0.4858±0.118<br>B | 0.4627±0.0588<br>B | 0.7065±0.0842<br>A |

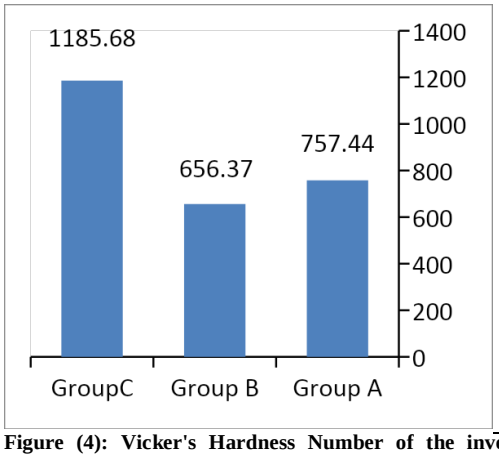


**Figure (4): Vicker's Hardness Number of the investigated groups**

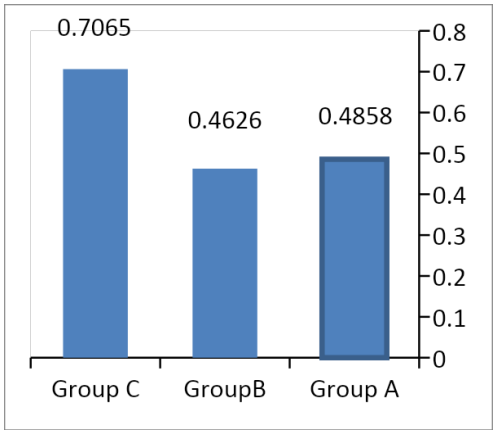


**Figure (5): Fracture Toughness of the investigated groups**

## DISCUSSION

Despite major laboratory tests are performed to investigate materials based on the bulk features, surface characteristics are also a determinant factor. For example, hardness is an intrinsic material property used to evaluate surface resistance to scratching and defined by units of mass and surface area. From empirical relationships, this surface property; hardness, could be used to estimate bulk properties such as fracture toughness of ceramics and glasses; ($K_{1c}$). [10]

In reality, accurately measuring the fracture toughness of brittle materials can often be challenging. Creating sharp pre-cracks is usually difficult without catastrophic failure of the specimen, while fracture toughness data using notched specimens can give erroneously high values. [11,12] For those reasons, assessing fracture toughness by making direct measurements of cracks created using a sharp diamond indenter, such as Vickers, Knoop or Berkovich appear an attractive alternative to more traditional fracture toughness testing techniques. [13,14] Such tests can be relatively quick and easy to perform, require little specialized equipment, and can allow probing of localized microstructural features. Accordingly, such techniques represented considerable usage in studying the fracture behavior of biomaterials and hard tissues. [15,16]

By far, the most widely used technique in the literature for assessing the fracture toughness directly from indent cracks utilizes the Vickers indenter. This technique was firstly developed in the late

1970's to estimate the fracture toughness of ceramic materials by measuring the lengths of cracks emanating from Vickers indents. [17,18] Later, this method has subsequently received much attention for measuring fracture toughness of bioceramics. [19,20]

The ultimate goal of all fracture toughness testing techniques is to quantify the fracture toughness accurately in a way that can be universally compared with the results generated using other techniques employed by other studies. Unfortunately, techniques involving direct measurements from indent cracks are often unsatisfactory in this regard. [5,21] A secondary goal may be to provide a quick semi-quantitative way to rank the toughness of different materials. In this case, this less responsive indentation technique can have some merit in comparison with other techniques as it has the advantages of less cost effectiveness and ease of set-up, and it is one of the simplest and least time-consuming.

Thus, the aim of the present work was to determine the hardness of some commonly used dental glass-ceramics using Vicker's indentations and then, calculate the fracture toughness through crack-length measurements of cracks emerged into the sample surface.

Using the crack opening displacements due to Vickers indentation to assess the fracture toughness of ceramics resulted in value of Ko equal to -2.3 MPa-$m^{1/2}$. This value is within 10% of the typical values reported using standard fracture mechanics samples, demonstrating the viability of using such a method for toughness measurements. Indeed, measured crack openings are smaller, and the deduced toughness is lower for one crack where significant secondary radial cracking is evident. These secondary radial cracks are believed to relief some of the residual stresses affecting the crack opening profile and, correspondingly, the computed toughness values. Although this method holds promise, it is apparent that there are remained unresolved issues that must be addressed before this can be considered as a reliable test method. [6] Also, Ćurković L, et al; in 2007 [22] proposed that this test could be unreliable due to subcritical crack growth and the difficulty in determining the exact length of the cracks.

*Lawn et al., (1980)* [23] modeled the elastic- plastic behavior under the indent, assuming that a median/radial crack system is created due to tensile stresses that form during unloading. Therefore, the observed subsurface lateral cracking; Figure (3) would be possible explanations for the bargain fracture toughness values. They relieved some residual stresses and affect the crack openings or cracking during the loading of the indentonr.

The mechanism responsible for this loss of strength in dental ceramics is the mechanical degradation. [24] It should be pointed out that cyclic loading in humid environment permits crack propagation at stress levels in some cases of less than 50% of the initial material strength. [25] Dental ceramics, particularly porcelains, are vulnerable to slow crack growth. At ambient conditions, a crack slowly but continuously grows in length, degrading the strength of the ceramic which might be endorsed by low continuous cyclic loads in a humid environment. [26]

Failures of ceramic restorations can be initiated from several different sites on the surface, at interfaces, or within the material. In laboratory studies, the first crack to appear in nearly all dental ceramics is an outer cone crack, developing on the outer surface of the material due to the stress field created by a loaded indenter. On subjecting glasses (like feldspathic porcelains) to subcritical cyclic loads, the failure mode is usually a radial fracture, initiating from tensile stresses and propagating through the entire interior of the material, leading to the bulk fracture. [27,28]

Remarkably, in dental all-ceramics, the first crack to initiate seems not to be the one that propagates and ultimately causes the material to fail. Zirconia is rarely indexed failure by radial crack, instead; the secondary crack that develops could be an

inner cone crack beneath the indenter. Inner cone cracks develop during loading which are the cracks created by the expanding compressive stresses beneath the indenter that concentrated into one large crack, oriented perpendicular to the direction of the sliding indentor and penetrating deep into the ceramic. These inner cone cracks develop, initiate and, propagate till failure. [29]

In agreement with *Queinn, et al; in 2003* [4] the obtained results supported the theoretical basis predictions of the microstructure/toughness relationship in the literature. From a practical standpoint, the chemistry of dental ceramics definitely plays a crucial role. On the other hand, microstructural is influential but only within a limited range suggesting that the fracture toughness is unlikely to be attained by changes in microstructure alone. Generally, in multi-phases ceramic microstructure, the glass phase is the dominant factor controlling slow crack growth. [30] Lithium disilicate (IPS Empress 2) and glass-ceramics are the most susceptible materials. [31] As well, zirconia is also vulnerable to this slow crack growth but in slower rate of crack propagation. [25] Cracks are initiating from the contact area, became evident long before fracture. [32]

The conventional dental porcelain is 75-85 % feldspathic with primarily glass microstructure with crystalline silica and metal oxides inclusions. Owing to the glassy amorphous structure of conventional porcelain, the cracks propagate trans-granularly and thus interpret for the recorded low fracture toughness value; Table (2).

*Cesar et al., in 2008* [33] reported that materials designed to slow down fast crack propagation by crystalline inclusions and even resist the slow crack growth might be susceptible to fast cracking as crack propagates at supersonic speeds. Lithium disilicate content has been thought to reduce slow crack growth. However; in this study, Lithium disilicate failed to hinder the crack growth. This might be related to presence of tensile stresses in the glass matrix around the crystals that increased the matrix interatomic spacing and weakened the inter-atomic bonding making the region more sensitive to crack propagation. [34] The microstructure of IPS e.max consists of approx. 70% lithium disilicate ($Li_2Si_2O_5$) needle-like crystals of 3 to 6 µm in length embedded in a glassy matrix. The purpose of adding crystals is the reinforcement of the glass-ceramics;[35] however, the obtained results showed that this all-ceramic material recorded comparable hardness and fracture toughness to those of conventional feldspathic porcelain. This could be attributed to the segregation of the crystal content in the central bulk of the specimens during the processing. Meanwhile the glassy phase constitutes the outer most layers of the specimens. According to manufacture recommendations, the IPS glaze has to be applied by firing twice at 800°C for 6 min. It was supposed that glazing serves for uniform distribution of lithium disilicate crystals. Unfortunately, the glazing procedure was quitted during the preparation of the investigated specimens. IPS Empress 2 also recorded the lowest significant hardness value; table (2), which is beneficial for clinical applications.

Regarding the highest fracture toughness value recorded by zirconia, it is attributable to the toughness of zirconia. It is considered that toughening of zirconia is due to stress-induced phase transition. Crystal phase transformation of tetragonal phase stabilized at room temperature to monoclinic phase by loading prevent crack progression due to volume expansion of 4 % during phase transformation which is accompanied by compression stress at the leading end i.e. crack tip. [1,2]

## CONCLUSION

From the present investigation, the employed Vicker's indentation technique to determine the fracture toughness is easy and quick test. As well, it is semi-quantitative to evaluate and compare the fracture toughness of the different available dental ceramics

and semi-qualitative to predict their mechanism of failure.

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