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The Impact of Surface Treatments on Biaxial Flexural Strength of Some Dental Ceramics

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A B S T R A C T

The objective of this investigation was to observe the impact of altering surface topography on the biaxial flexural strength (BFS) of four dental ceramics namely Chameleon (CH), Mirage (MI), Optec-HSP (OP) and Flexoceram (FL).
A hundred and sixty samples (sixteen groups of ten discs 12 3mm) were fabricated and fired according to the suppliers' procedures. Ten samples of each material were exposed to three surface treatments, polished, grit blasted and etched with 10% hydrofluoric acid (HF) for 30 s and 2 min. Some samples were gold coated for investigation using Back Scattering Imaging (BSI) and Scanning Electron Microscope (SEM). BFS was evaluated using Lloyd M5K universal testing machine at a cross-head speed of 0.5 mm/min. The statistical results of differences between groups were determined using one-way ANOVA.
The results acquired exhibited that the only significant difference in the BFS was the 10% HF for CH, OP, and FL (One-way ANOVA, $P<0.05$). The decrease in BFS is most likely due to an increased surface flaw size, such that surface initiated crack growth dominates over the bulk internal flaw size of the dental porcelains.
The BFS of dental porcelain examined in this investigation may be administrated either by the internal or surface flaws depending on the manner of surface groundwork. Specimens etched with 10% HF (2 min) became weaker for CH, OP and FL ceramics.

Keywords: *Surface Topography, Biaxial Flexural Strength (BFS), Dental Ceramics/ Porcelain, Surface Treatments, Surface Flaw Size.*

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INTRODUCTION

Dental ceramics have developed increasingly and become popular because of their inimitable properties such as biocompatibility, chemical stability and superior aesthetic qualities. However, one of the inherent disadvantages of dental ceramic restorations is their brittle nature. This brittle behaviour combined with the presence of surface and internal flaws may result in a low strength and consequently has limited the clinical use of ceramics such as those based a leucite reinforcement [1].

The leucite reinforced ceramics often be unsuccessful at stresses below their reported strength values as results of either internal or processing weaknesses. The former could be due to residual stresses, large grains and micro cracks resulting from the differences in the thermal expansion between the glassy matrix and the crystalline phases of the leucite [2], whereas the latter could be machining scratches, impurity phases and porosity [3]. The strength for a certain material will, consequently, depend on the number and size of weaknesses that are incorporated within the material. These deficiencies in the materials are expected to be connected with sharp cracks which are not exposed by investigation of the fracture surface, since they form an indistinguishable part of the fracture surface [4].

Resent studies have shown that grinding process caused a significant reduction in the flexural strength of the feldspathic porcelains, whereas polishing and glazing have a significant increase of the flexural strength of the same porcelains [5] and [6]. Southan [7] found that scratches of a depth 30-40 μm in feldspathic porcelain specimens caused a reduction of about 75% in the biaxial flexural strength (BFS).

Hussain *et al.* [8] and Baez and Blackman [9] inspected the effect of chemical etching on the strength of some dental ceramics using either hydrofluoric acid (HF) or ammonium fluoride (NH_4F). They found a decrease in strength of 20 to 30% for aluminous porcelain and a cast-able glass ceramic. Jones [10] showed a reduction of 40% in strength of one brand of feldspatic dental porcelain rods and no weakening influenced of another brand after etching with HF. Levy [11]

found no significant difference in the flexural strength values of some dental ceramics when the surface was chemically etched. These suggest that the strength of dental porcelains is not always dependent on the features of the surface.

There is thus inconsistent information in the impact of surface treatments on the strength of dental ceramics. This is because the strength depends on the relationship between the fracture toughness (K_{ic}) and the flaw size [3], as shown in Equation 1:

$$\sigma_f = K_{ic}/Y\sqrt{C} \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots \quad (1)$$

Where σ_f is fracture strength, K_{ic} is fracture toughness, C is flaw size and Y is the geometric constant. The Kic values were obtained from results reported by Bieniek and Marx [12].

Therefore, it is vital to evaluate the impact of surface treatments on the strength of dental ceramics. The objective of this study was to evaluate the impact of three surface preparations namely polishing, grit blasting and etching on the biaxial flexural strength (BFS) of the fit surface of four dental ceramics and establish whether surface or internal flaws control the BFS.

MATERIALS AND METHODS

Four commercial available dental ceramics were used in this investigation, namely Chameleon (CH), Mirage (MI), Optec-HSP (OP) and Flexoceram (FL) as detailed in Table 1. Four groups of ten specimens were fabricated for each material. A mixture of ceramic powder and condenser liquid (Myron, Inc., Kansas, USA) was cast into a silicon rubber mould 12mm in diameter × 3mm thick and vibrated to condense the particle and then fired according to the suppliers' procedures applicable to each ceramic. The fired discs were ground with 600 grit SiC paper (Buehler-Met, Metallographic Grinding Paper, UK), to produce flat parallel surfaces and their thickness was measured by a micrometer screw gauge (Mitutoyo, Japan). Ten specimens of each material were exposed to three surface treatments, namely: polished with diamond paste down to 1 µm, grit blasted (alumina 50 µm) and etched with 10% HF for periods of 30 s and 2 min. Some samples were gold coated for examination under BSI and SEM [2].

The biaxial flexural strength (BFS) values for ten discs of each material were evaluated by employing each specimen on an annular knife edge 9mm in diameter and loaded with a 3mm ball-ended indentor in a Lloyd M5K universal testing machine at a cross-head speed of 0.5 mm per min. The samples were loaded to failure and the maximum BFS values were calculated using the equation reported by other investigator [13]. Assuming a Poisson's ratio (ν) of porcelain of 0.25, the simple form of this equation is:

$$\sigma_f = P/h^2 \{0.606 \ln (a/h) + 1.13\} \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots \quad (2)$$

Where $\boldsymbol{\sigma_f}$ is the biaxial flexural strength (BFS), $\boldsymbol{P}$ is the load to rupture, $\boldsymbol{a}$ is the radius of the knife-edge support and $\boldsymbol{h}$ is the sample thickness.

Statistical assessments between groups were prepared using one-way analysis variance (ANOVA) significant difference test.

RESULTS

BFS data for each group of CH, MI, OP, and FL are sketched in Figure 1 and detailed in Table 2. Statistical analyses of these results are shown in Table 2 with analysis of variance (ANOVA) and Tukey HSD tests used to evaluate significance between groups. The results show no significant difference in the BFS for the polished and grit blasted. In contrast, the BFS reduced after etching with 10% HF (2 min), which was significant for CH (42 MPa), OP (64 MPa) and FL (42 MPa) (one-way ANOVA, $P<0.05$), but not for MI (64 MPa).

Values for the average flaw size (C) were calculated using Equation (1) and are shown in Figure 2 and detailed in Table 3. The fracture toughness (K_{ic}) values were acquired from data reported by Bieniek and Marx [12] and the BFS data of this investigation were used for σ_f.

The microstructural features were examined using Back Scattering Imaging (BSI) and Scanning Electron Microscope (SEM). The BSI images are shown in Figure 3a-d for CH, MI, OP and FL respectively. Also, Figure 4a-c shows example of SEM micrographs of CH, MI and FL respectively.

DISCUSSION

As shown in Fig. 1, the BFS decreased significantly for samples prepared from CH, OP and FL materials when etched with 10% HF for a period of 2 min, whereas polishing and grit blasting treatments have no impact on the BFS.

The reduction in the BFS values after etching with 10% HF is most likely due to an increase surface flaw size which would be sufficient to induce fracture initiation from the surfaces. Other investigators have also found that a decrease in the range of 20% to 40% in the strength values after acid etching of dental ceramics [8] and [10]. However, it should be noted that etching with HF does not always has impact on the BFS values.

Levy [11] reported that no significant difference in the flexural strength values of some dental ceramics between polishing with pumice and etching after air and vacuum glazing and over glazing. Also, Jones [10] reported that a reduction of 40% in strength of one brand of feldspatic dental porcelain rods and no weakening effect of another brand when etched with HF.

The BFS was unaffected for the samples polished with diamond paste down to 1 µm and grit blasted (alumina 50 µm). This suggests that the BFS is ruled primarily by the internal flaw size. However, Sano *et al.* [14] reported that feldspathic porcelain samples showed a higher four-point flexural strength when polished with 0.3 µm alumina. Sherrill and O'Brien [15] found that no difference between the strength of feldspathic porcelain samples when their surfaces were fine polished or auto-glazed. Fairhurst *et al.* [16] and Giordona *et al.* [17], on the other hand showed that polishing surfaces with 1 µm and 15 µm diamond pastes produced significant stronger specimens than auto-glazing. Also, it has been reported that no statistically significant differences in the load at failure of glazed porcelain and polished auto-glazed porcelain [18]. However, polishing the glazed specimens showed higher strength values [11].

These suggested that the characteristic properties of dental porcelains are not always dependent on the characteristics of the surface.

A measure of the severity of fracture-initiating flaws can be acquired from the measured values of strength and fracture toughness by assuming that the flaws had a particular simple geometry (Equation 1). Therefore, usable strength is a function of flaw size.

Based on the present data the calculated flaw size was around 100 µm (Figs. 2, 3 &4), which corresponds well to the observed internal cracks size that have been reported in the earlier investigation by Shareef *et al.*, [2].

CONCLUSIONS

1) The BFS for the polished and grit blasted surface finishes of dental porcelain used in this investigation is control primarily by the internal flaw size.
2) Etched surfaces (10% HF for 2min) caused a reduction in the BFS values because the large surface flaws created control the fracture process.
3) The calculated flaw size values were confirmed by the BSI and SEM micrographs.

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Table 1: Details of dental ceramics used in this investigation.

Materials' Name	Designated Code	Shade No.	Batch No.	Supplier
Chameleon	CH	Body D-4	059020	Myron Int. Inc. Kansas, USA
Mirage	MI	Body D-4	8130	Myron Int. Inc. Kansas, USA
Optec-HSP	OP	A2	B1637E	Jeneric, Conn., USA
Flexoceram	FL	D-EB1	442	Elephant Ceramics, Hoorn, Netherlands

Table 2: The mean values of flexural strength and standard deviation as a function of surface treatments of dental ceramics used in this investigation.

Materials' Code	Biaxial Flexural Strength/ MPa(±SD)*			
	Surface Treatments**			
	PS	GBS	LES	HES
CH	63.6(±8.7)	60.5(±7.2)	64.8(±3.7)	41.7(±5.7)
MI	78.0(±15.5)	77.7(±16.9)	73.8(±16.2)	63.7(±19.2)
OP	102.8(±17.5)	101.1(±12.8)	89.1(±14.8)	63.1(±17.8)
FL	60.9(±6.8)	61.7(±5.7)	59.3(±8.4)	42.3(±15.4)

*SD = Standard Deviation

** Surface Treatments: Polished Surface (PS), Grit Blasted Surface (GBS), Light Etched Surface (LES-30 s), Heavy Etched Surface (HES-2min)

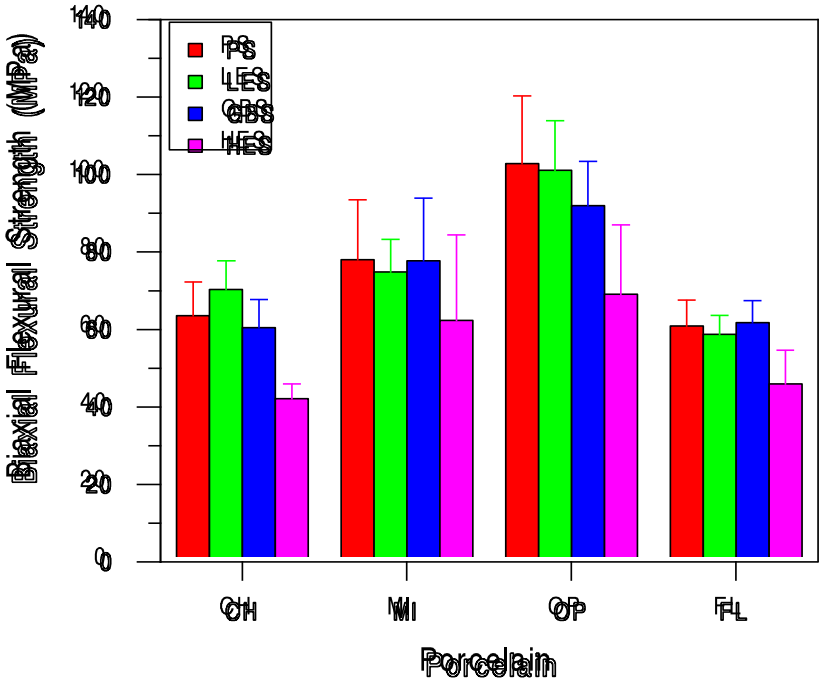


Figure 1: The mean values of biaxial flexural strength sketched in contrast to surface treatments for CH, MI, OP and FL materials.

Table 3: The mean values of flaw size as function of surface treatment for MI, OP and FL materials.

Materials' Code	Surface Treatment	Calculated Flaw Size (±SD) (μm)*
MI	PS	102.9 (47.3)
	GBS	102.8 (41.1)
	ES	190.5 (111.5)
OP	PS	134.4 (65.4)
	GBS	156.9 (40.4)
	ES	314.0 (148.2)
FL	PS	115.2 (23.2)
	GBS	111.0 (18.8)
	ES	216.1 (79.8)

* K_{1c} values was taken from Bieniek and Marx (1994).

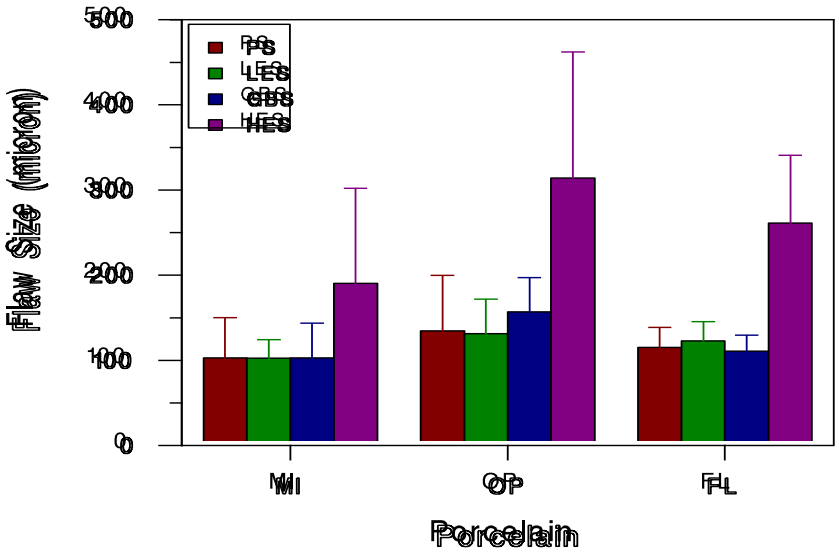
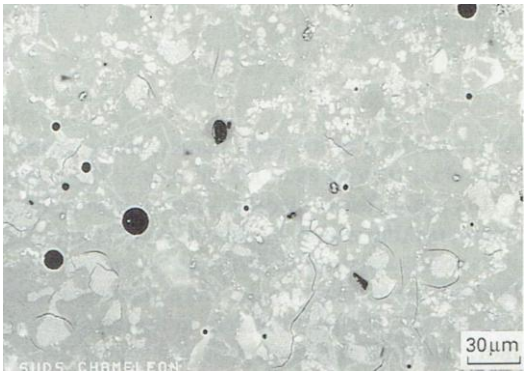
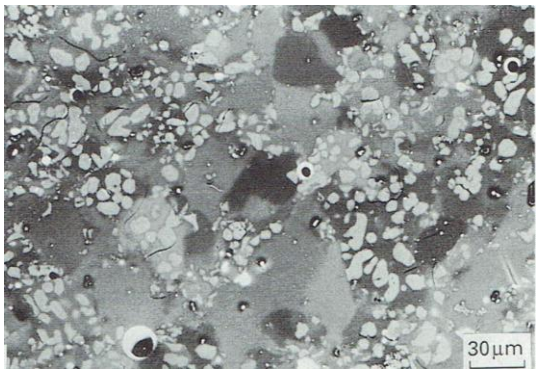


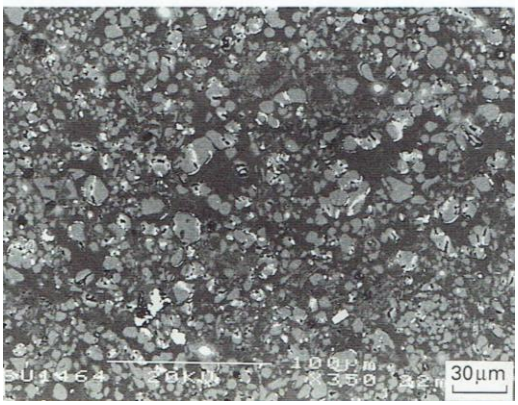
Figure 2: The mean values of flaw size sketched in contrast to surface treatments for MI, OP, and FL materials.



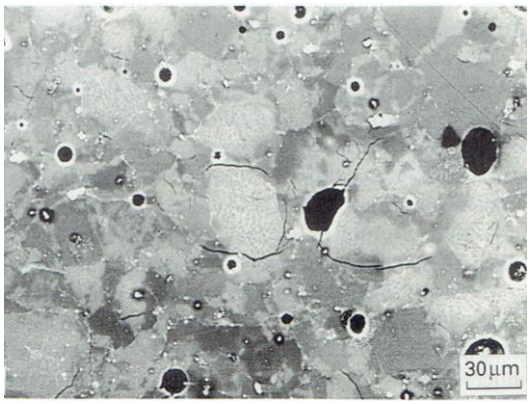
a) CH micrograph



b) MI micrograph



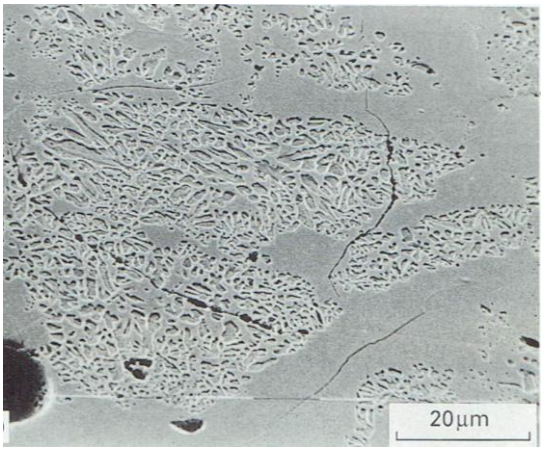
c) OP micrograph



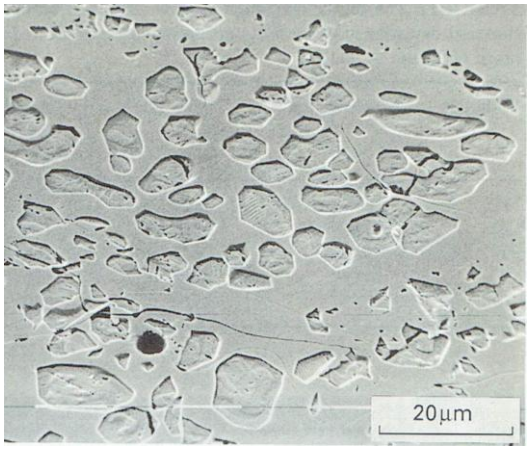
d) FL micrograph

Figure 3: Back scattered micrographs [2] of:

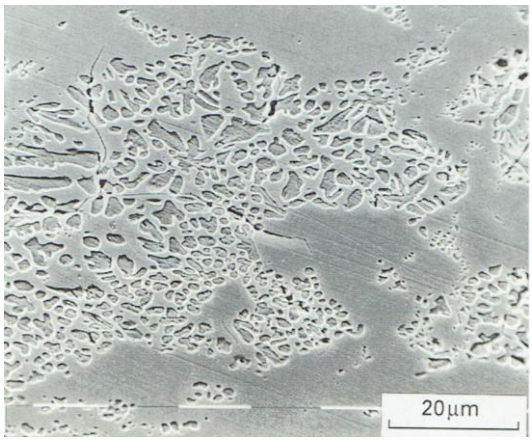
a) CH showing extensive cracking around the crystalline phase.
b) MI showing phase separation in the glassy matrix as indicated by the different shade of the grey.
c) OP showing a uniform distribution of leucite crystals and
d) FL showing clear indication of microcracking around the crystalline phase.



a) CH micrograph



b) MI micrograph



c) FL micrograph

Figure 4: SEM micrographs of the appearance of clusters of leucite crystals exposed by light etching with 1% HF for 30 s [2] for:

a) CH
b) MI and
c) FL