Original Article

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Effect of resin infusion on fracture toughness of dental veneering ceramic

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Objective: The objective of this study was to investigate effect of resin infusion on fracture toughness of dental veneering ceramic (IPS e.max Ceram®).

Materials and methods: Ten bars, size 2 x 4 x 24 mm of veneering ceramic as a control were prepared following the manufacturer's recommendation. The other ten experimental bars of resin-infused veneering ceramic were prepared by initially mixing the ceramic powder with polymeric fiber (0.1 % by mass), pressed into the mold and then firing according to the firing schedule without vacuum. After firing, a mixture of urethane resin was infused into the specimens under vacuum at $1x10^{-2}$ Torr for 6 hours and cured by dry heat at 100°C for 6 hours. Vickers hardness tester was used to create pre-crack indentation on the specimen. Four-point bending test was performed using a universal testing machine with a crosshead speed of 0.5 mm/min until fracture. Fracture surfaces of all specimens were examined under optical light microscope. Critical flaw sizes were measured using the fractographic analysis approach. Fracture toughness (K_{Ic}) was calculated. Independent sample *t*-test was used to determine significant difference of the mean K_{Ic} between control group and experimental resin-infused ceramic group at $\alpha = 0.05$.

Results: The mean K_{Ic} of resin-infused ceramic (0.75 ± 0.06 MPa•m1/2) was statistically greater than that of control group (0.69 ± 0.07 MPa•m1/2), ($p = 0.03$).

Conclusion: Resin-infusion had significant influence on fracture toughness of veneering ceramic.

Key words: Fracture Toughness, Resin-infused Ceramic, Fractography, Four-point bending, K_{Ic}, IPS e.max Ceram

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Introduction

Several all-ceramic systems have been developed in dentistry to meet the expectations of patients and dentists for high aesthetic, biocompatible, and long-lasting restorations. However, one drawback of all-ceramic restoration is chipping of veneering ceramic. Veneering ceramic is weaker than high-strength core material and possible to fail during masticatory function due to its low tensile strength and fracture toughness [1]. Microscopic surface flaws and defects may develop from mechanical, chemical or thermal during fabrication. These defects act as localized stress concentrators which can cause cracks to initiate from the defect sites and propagate, leading to devastating failure [2].

Fracture toughness is an important mechanical property of the material. It describes a material's resistance to resist rapid crack propagation and its consequent disastrous failure when a crack is present from the existing flaws [3]. Moreover, it indicates a material's serviceability in the oral cavity [4,5].

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Theoretically, composite technique is the easiest way to increase the fracture toughness to brittle material. Several researchers used the flexible resin as matrix that infiltrate into the porous ceramic to improve its fracture toughness. Chaiyabutr et al [6] investigated effect of resin-infused to alumina oxide ceramic core and found that the strength and fracture toughness of alumina were increased by resin infusion. Coldea et al [7] had attempted to develop novel material called as polymer-infiltrated-ceramic network (PICN) by infiltrating resin mixture into the industrial specially prepared porous ceramic. However, those researches were aiming to increase the fracture toughness of the core materials with a special industry preparing porous ceramic.

The present study was to develop a simple technique in the dental laboratory to infuse a resin into a veneering ceramic to increase its fracture toughness. The aim of this study was to investigate the effect of resin infusion on the fracture toughness of veneering ceramic.

Materials and methods

Veneering ceramic powder (IPS e.max Ceram®, Ivoclar-Vivadent, Schann, Liechtenstein) 1.5 g was initially mixed with 0.0015 g (0.1 wt %) chopped polymeric fiber, size 75 μm in diameter and 3.5 mm long, to prepare a space for resin infusion. The ceram liquid (Ivoclar-Vivadent, Schann, Liechtenstein) was added to the powder to form a slurry for easy fill and condensation into the mold. A slurry was filled into the bar shape mold, size 2x4x24 mm. The condensation of the powder was done under plugger and vibration. Excess liquid and powder was removed by paper and scalpel. The specimens were removed from the mold and placed on the firing tray until dry. The control specimen, veneering ceramic powder without polymeric fiber added was prepared as same as the experimental specimen. Ten bars for each were prepared.

After drying, the specimens were fired in a ceramic furnace according to the company recommendation firing schedule. However, the experimental specimens were fired without vacuum in order to relieve the polymeric vapor pressure during firing.

After firing and cool down, the experimental specimens were immersed in a mixture of urethane dimethacrylate (UDMA), triethylene glycol dimethacrylate (TEGDMA) and Benzoyl peroxide (BPO) on the ratio 75:24:1 wt%. The resin mixture was infused into the specimens under vacuum at $1x10^{-2}$ torr for 6 hours, and then cured the resin by dry heat in the oven at 100°C for 6 hours.

All specimens were grinded and polished with silicon carbide paper (# 400, 800, 1000 and 1500) then finished with 0.05 μm alumina particles.

The fractographic analysis method (FTA) was used for fracture toughness test. A well-defined controlled flaw for fractographic analysis was made by indentation on the surface using a Vickers indenter (FM-700, Future-Tech Corp., Tokyo, Japan) with load 9.8 N for 15 sec.

Four-point bending test was performed again on the pre-cracked specimen using a universal testing machine (Instron 5566, Instron LTD., Buckinghamshire, England). The specimens were placed the pre-cracked side down for tension and located centrally on the bearers (20 mm supporting span, 10 mm loading span). The bars were loaded until fracture with a crosshead speed of 0.5 mm/min. Flexural strength (σ_f, MPa) was calculated according to the equation (A):

$$\sigma_f = \frac{3PL}{4wb^2} \quad \text{(A)}$$

Where P is the load at fracture (N); L is the center-to-center distance between the support rollers (mm); w is the width of the specimen (mm); and b is the thickness of the specimen (mm).

The critical flaw sizes on fracture surfaces of all specimens were measured under optical light microscope (Nikon Eclipse E400 Microscope, Fukuoka, Japan) at 100X magnification. Fracture toughness, K_{Ic}, is calculated using the equation (B) [8,9]:

$$K_{Ic} = Y \sigma_f c^{1/2} \quad \text{(B)}$$

Where *Y* is the geometric factor for sharp cracks that are induced by Vickers indentation (1.65), σ_f is the flexural strength (MPa), c is the crack size (m) calculated from the equation $c=(ab)^{1/2}$ while a is the crack depth and b is the half crack width. The schematic diagram of the critical flaw size was shown in Figure 1.

Independent *t*-test was used to compare the mean fracture toughness (n=10) of both groups at 95% confidence level (σ=0.05, two-tailed).

Results

The mean fracture toughness of control group and resin-infused ceramic were 0.69 ± 0.07 and 0.75 ± 0.06 MPa·$m^{1/2}$, respectively.

The result of independent t-test shows a significant difference of the mean fracture toughness between both groups. The mean fracture toughness of resin-infused ceramic was significantly higher than that of the control (P = 0.03).

The fracture surface of specimens for both groups is presented in figure 2. The crack pattern of control group showed symmetrical pattern, while those of the resin infused ceramic group showed asymmetrical pattern and greater deflection.

Discussion

From the result of this study, resin infusion had effect on fracture toughness of veneering ceramic. Enhanced fracture toughness is attributed to the porous structure's ability to anchor infused resin matrix. The ultimate advantage of the interconnectivity of materials having an interpenetrating phase is an improved resistance to crack propagation [10]. In this study, symmetrical crack patterns were presented in non-infused ceramic group whereas asymmetrical crack pattern with greater deflection were presented in resin-infused ceramic group. These findings are in agreement with the findings of Coldea et al [7]. They reported that polymer network caused greater crack deflection in polymer-ceramic composites and the deflections occurred at polymer-ceramic interfaces. Crack deflection may be the possible

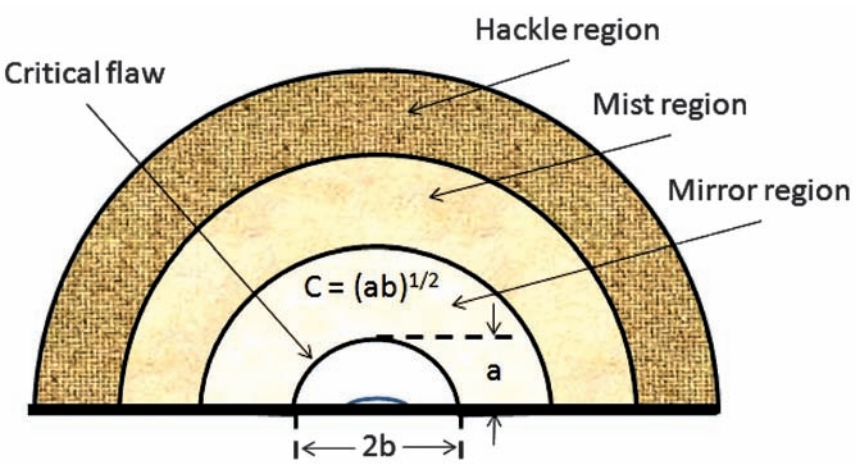

Figure 1. Schematic diagram of the critical flaw size; a the crack dept; 2b the crack width[8]

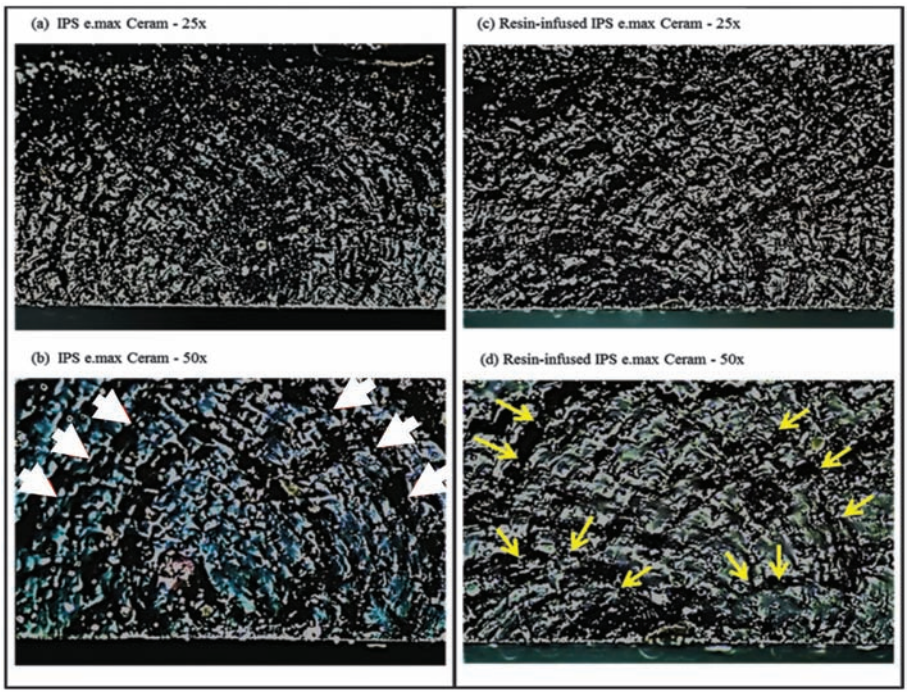

Figure 2. Representative optical light microscopic images of fracture surfaces of specimens at magnification of 25x and 50x. (Images a-b) glass ceramic, symmetrical crack patterns (arrows indicated) are presented. (Images c-d) UDMA/TEGDMA resin mixture-infused glass ceramic, asymmetrical crack patterns with deflection (arrows indicated) are presented.

toughening mechanism for crack-tip shielding in polymer-ceramic composites.

There are various methods can be used to determine K_{Ic} and the obtained values can be varied depending on technique used. In this study, K_{Ic} of glass ceramic and resin-infused glass ceramic were determined using the quantitative fractography and controlled surface flaws were created from Vickers indentation. The main advantage of Vickers indentation is that their flaw size and shape can be controlled and these sharp cracks are easy to detect on the fracture surface [11]. Fracture surface analysis of ceramics can be combined with fracture mechanics relationships as a quantitative tool to determine the location, size of the critical flaw and fracture toughness of the material. In this study, the means K_{Ic} of both control veneering ceramic and resin-infused veneering ceramic are in the normal range value of veneering glass ceramic (0.7 - 0.9 MPa·$m^{1/2}$) [11,12]. Several studies used this method to determine K_{Ic} values of dental ceramics and the results showed that the fractographic analysis could provide comparable results to other testing methods. No significant differences were found between numeric and fractographic K_{Ic} [11,13].

Several studies investigated the clinical failures of all-ceramic restorations [14-18]. Cohesive fractures within low fracture toughness veneering ceramics were described as the most frequent reason for failures, irrespective of substructure ceramic systems. There were many attempts to develop purposeful materials having an interpenetrating phase microstructure in order to improve material's properties [6,7,19]. In this study, the polymeric fiber were acted as a space creator and burn out during firing the ceramic, created the replica spaces of polymeric fiber in

ceramics. Although, the inevitable pore formation during powder condensation was presented in both groups, the greater amount of pore formation in experimental group can be seen on the surface under microscope. Moreover, the results from EDS elemental mapping showed homogeneous lower density of silicon distribution in experimental group than the control group. These confirm the well prepared porous in the ceramic before resin infusion.

From study of Ruddell et al [20], fused-fiber filler block networks were infiltrated with resin by two different methods: vacuum method or immersion method. They reported that vacuum method provided more resin infiltration than immersion method. Without the aid of vacuum, the resin was not completely infiltrated into the network and leaving the voids so the vacuum method was used for resin infiltration in this study. From the pilot study, methylene blue was mixed with resin mixture and infiltrated into prepared porous ceramic in order to evaluate capability of resin infiltration technique and the existence of resin. The prepared ceramic pores are fully filled by the resin.

However in this study, the mean K_{Ic} of resin infused glass ceramic was not as high as expectation, only slightly greater than that of non-infused glass ceramic. This might be the reasons as follows. First, ceramic and resin parts did not have any chemical bond on each other, they joined together with mechanical interlocking only. The chemical bond on the interface between ceramic and resin can enhance the crack deflection in polymer-ceramic composites leading to higher fracture toughness of the composite material[7]. Application of coupling agent may be beneficial for improving the mechanical strength and enhance the fracture toughness of composite materials. Second, the volume fraction of resin in this study was probably too low. If the volume fraction of resin increases, this may affect to fracture toughness of the material. Coldea et al [7] reported that elastic modulus of polymer-ceramic composite material decreased and the strain at failure increased significantly when polymer fraction increased. The higher strain at failure implies the higher fracture resistance of material.

From this study, resin infusion had effect on fracture toughness of glass ceramic. However, the actual balance of properties of a composite system depends on the combination of the material used. Improving or adjusting the component or fabrication method of dental ceramics is quite complicate and may affect to color, translucency, opacity and other properties of ceramics, further studies are required.

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