Original Article

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Effect of resin volume fraction on fracture toughness of resin-infiltrated ceramic.

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Objective: The objective of this study was to investigate effect of resin volume fraction on fracture toughness of resin-infiltrated ceramic (IPS e-max® Ceram).

Materials and Methods: Forty bar specimens (2x4x24 mm) were divided into 4 groups. The control group was IPS e-max® Ceram specimens. They were prepared following the manufacturer's recommendation. The other groups were IPS e-max® Ceram infiltrated with 0.5%, 1% and 2% by volume of Silane/UDMA/TEGDMA resin mixture. They were prepared by mixing IPS e-max® Ceram glass powder with polymeric fiber (0.5%, 1% and 2 % by volume) until homogenous and added liquid to form slurry. Then, the mixture was packed into the mold and fired according to the firing schedule without vacuum. After that, resin mixture was infiltrated into the specimens under vacuum at 0.01 MPa for 2 hours then cured by dry heat in the oven 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 approach, and fracture toughness (K_{Ic}) was calculated. One-way ANOVA was used to determine and analyze a significant difference of the mean K_{Ic} between control group and 0.5%, 1% and 2% by volume of resin-infiltrated ceramic groups at $\alpha = 0.05$.

Results: The mean fracture toughness of control group, 0.5%, 1% and 2 % of resin-infiltrated ceramic were 0.69±0.05, 0.71±0.08, 0.77±0.11 and 0.80±0.11 $MPa \cdot m^{1/2}$ respectively. Only 2 % resin-infiltrated ceramic specimen showed the significant increasing on fracture toughness comparing with the control group, ($p < 0.05$).

Conclusions: The amount of resin volume fraction of resin infiltrated ceramic had an influence on its fracture toughness.

Key words: asymmetrical crack pattern, cracked deflection mechanism, fractography, fracture toughness, resin infiltrated ceramics, resin volume fraction

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Introduction

Nowadays, the trend to use all ceramic restorations increases due to their excellent biocompatibility, good mechanical property and higher esthetic compared with porcelain fused to metal crown [1]. However, the most common complication of all - ceramic restoration is chipping or cracking on veneering ceramic because of its brittleness, crack propagation and low tensile strength [2]. Recently, the application of translucent monolithic zirconia has increased. The advantage of this material is high strength that can solve the problem of cracking of veneering ceramic. On the other hand, it is still more opaque than conventional ceramic. In order to achieve the esthetic, substructure core is still made by zirconia and veneering with feldspathic ceramic [3].

The important mechanical property that defines the capability to prevent fracture or catastrophic failure of a material is fracture toughness [4]. In addition, it represents the serviceability of dental material in the oral cavity [5].

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Urethane dimethacrylate monomer (UDMA) is the most common resin monomer using in many dental products due to its relatively low viscosity and high flexibility of its structure linkage that may promote high fracture toughness [6,7]. UDMA can be used alone or mixed with the other low viscosity diluent monomer, for example, Triethyleneglycol dimethacrylate (TEGDMA) [6,7].

In resin composite, it consists mixture of ceramic filler particles to strengthen the soft and more flexible resin materials to improve its mechanical properties to be a long-lasting serviceable restorative material. It was found that ceramic filler can raise its strength, fracture toughness and can resist crack propagation [8,9].

Like composite resin materials, many researchers attempt to increase fracture toughness of dental ceramic by using the flexible resin infiltrated into the porous ceramic to increase its flexibility leading to increase its fracture toughness. Chaiyabutr et al [10] investigated effect of UDMA/TEGDMA resin-infused alumina. They found that the resin infusion technique can improve the strength and fracture toughness of alumina matrix [10]. Moreover, Coldea et al [11] attempted to create novel material called polymer-infiltrated-ceramic-network (PICN) by infiltration of UDMA/TEGDMA resin mixture into porous ceramic. They reported that the high volume of polymer fraction of polymer infiltrated ceramic group has greater flexural strength and stain resistance but lesser elastic modulus and hardness than these of the low volume of polymer fraction group [11, 12].

However, those researches were aiming to increase the fracture toughness of the core materials with a special industry preparing porous ceramic. From the initial study of in-laboratory technique to produce the resin infiltrated veneering ceramic by Urapepon et al [13]. They found the feasibility of this technique. Therefore, the objective of this study was to investigate the effect of resin volume fraction on fracture toughness of resin-infiltrated ceramic.

Materials and methods

Veneering glass ceramic powder (IPS e.max® Ceram, Ivoclar-Vivadent, Schann, Liechtenstein) 1.5 g, density at 3 g/ml was initially mixed with 0.00375g, 0.0075g and 0.015g (0.5, 1 and 2 vol %, respectively) of chopped polymeric fiber, density at 1.51 g/ml, 75 μm in diameter and 3.5 mm long, to prepare a space for resin infusion. The ceramic liquid (Ceram liquid, Ivoclar-Vivadent, Schann, Liechtenstein) was added to the powder to form a slurry. The ceramic slurry was filled and condensed into the bar shape mold, size 2x4x24 mm. The condensation of the powder was done using plugger and vibrator. The specimens were removed from the mold and placed on the firing tray until dry. The control specimen, veneering glass 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 for 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 74.5:24.5:1 wt%. After that 2% by weight of 3-acryloxypropyl trimethoxysilane were added. The resin mixture was infiltrated into the specimens under vacuum at 0.01 MPa for 2 hours, and subsequently cured 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) and finally finished with 0.05 μm alumina particles.

The fractographic analysis method (FTA) was used to identify the critical crack size for fracture toughness calculation. 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) at 9.8 N load for 15 sec.

All indented specimens were tested using four-point flexure on universal testing machine (LF Plus, LLoyd instruments, Ametek Inc., USA). The specimens were placed with the pre-cracked side under tension and located centrally on the bearers (20 mm supporting span, 10 mm loading span). The bars were loaded until fracture

at a crosshead speed of 0.5 mm/min. and the flexural strength was calculated.

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[13]

$$K_{Ic} = Y \sigma_f c^{1/2}$$

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 critical crack size (m) calculated from the equation $c = (ab)^{1/2}$ where a is the crack depth and b is the half crack width.

One- Way ANOVA and Tukey's test were performed to compare the mean fracture toughness of each group at 95% confidence level ($p < 0.05$).

The fracture surfaces of all specimens were examined under optical light microscope (Nikon Eclipse E400 Microscope, Fukuoka, Japan) at 25X, 50X magnification and under scanning electron microscopic (Quanta 250, FEI, Oregon, USA) at 60X, 500X magnification.

Results

The mean fracture toughness of control group, 0.5%, 1% and 2 % resin-infiltrated ceramic were 0.69 ± 0.05, 0.71 ±0.08, 0.77±0.11 and 0.80 ± 0.11 MPa·$m^{1/2}$, respectively.

It was found that only fracture toughness of 2% resin-infiltrated ceramic (0.80 ± 0.11 MPa·$m^{1/2}$) was significantly higher than that of control (0.69 ± 0.05 MPa·$m^{1/2}$) while the other were not significant ($p>0.05$).

The optical light micrograph of fracture surfaces of specimens at magnification of 25x and 50x were presented in figure 1. The crack patterns of the control group are symmetry while the other resin-infused ceramic groups, the cracks have asymmetrical pattern and greater deflection than that of control group.

The scanning electron micrographs of fracture surfaces of specimens at magnification of 60x and 500x are presented in figure 2.

Resin-infiltrated ceramic groups have porosities distribution greater than the control group and the high resin volume fraction in ceramic group has porosities more than the low resin volume fraction group.

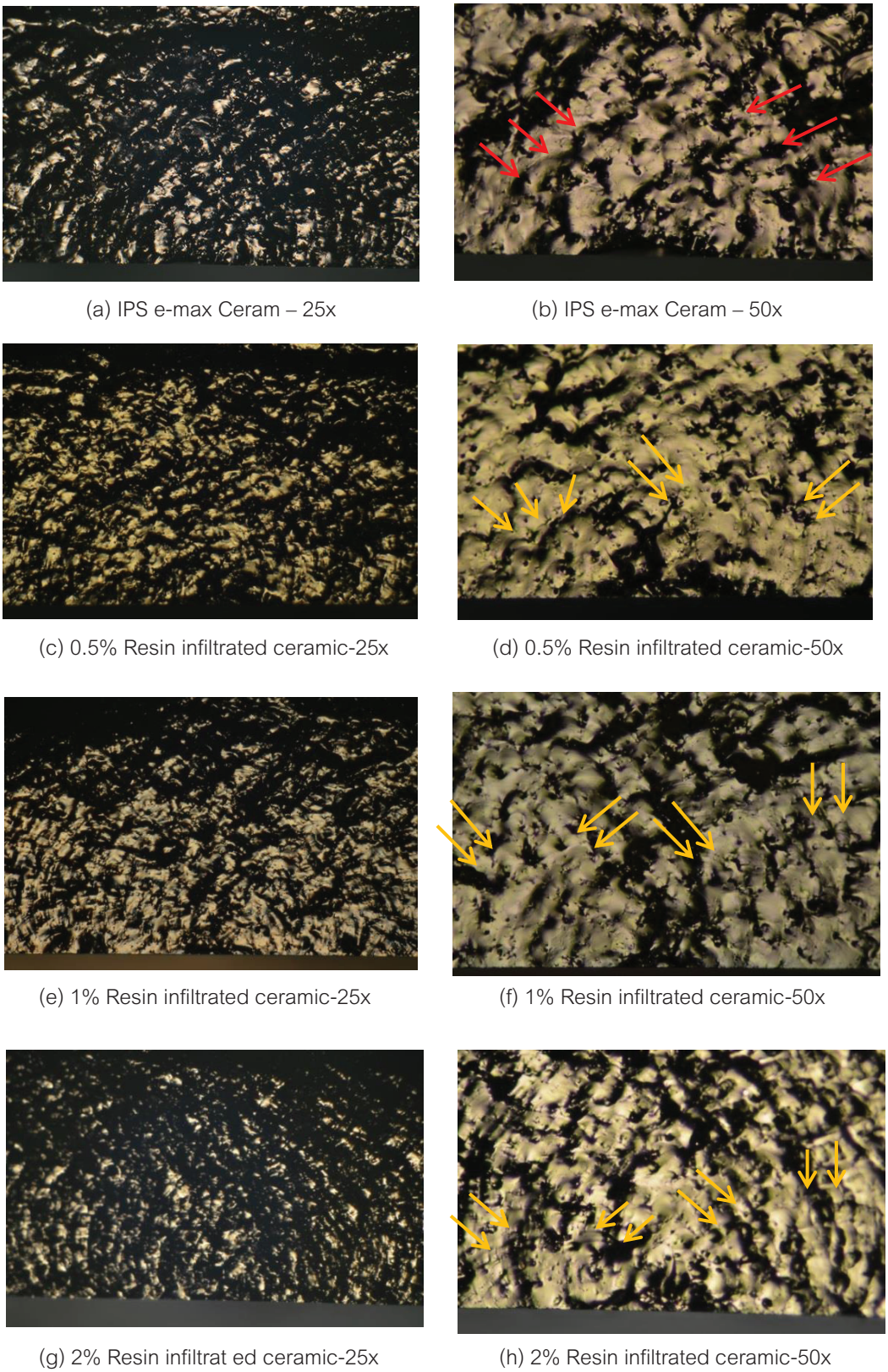
Discussion

This study intended to create the method of resin-infiltration into veneering ceramic during the process in dental laboratory. The dental technician can add the polymer fiber into veneering ceramic to produce the space for resin infusion. Then they can perform a conventional technique to veneer the glass on its substructure. After the firing, the polymeric fiber will burn out and left the replica space in ceramics [13]. This space can be filled by resin mixture to create interpenetrating phase [14] by using vacuum method and this process can be used as a grazing process to smoothen the glass ceramic surface.

From the result of this study, the resin infiltration had effect on the fracture toughness of the glass ceramic. Although only the highest load of resin infiltration (2%) group had fracture toughness greater than the control group.

From the optical light microscope examination at Vickers' indentation point on resin-infiltrated ceramic specimen, the crack extensions from indentation diagonals crack run though ceramic parts had the deflection at polymer-ceramic interfaces (Figure 3) and found greater asymmetrical pattern than control group.

This conformed to the study by He and Swan [12], which reported that the polymer infiltrated ceramic had higher fracture toughness as a result of a crack deflection mechanism. In dense ceramic, the crack propagation occurred around of ceramic crystalline but in polymer infiltrated ceramic, the crack propagation occurred pass into the polymer phase. Moreover, from the investigation of Coldea et al [11], they reported that the crack extension form indentation diagonals of dense ceramic was greater than polymer infiltrated ceramic and the crack run through ceramic part but the deflect occurred at polymer-ceramic interface. It can imply that the interpenetrating



(a) IPS e-max Ceram – 25x

(b) IPS e-max Ceram – 50x

(c) 0.5% Resin infiltrated ceramic-25x

(d) 0.5% Resin infiltrated ceramic-50x

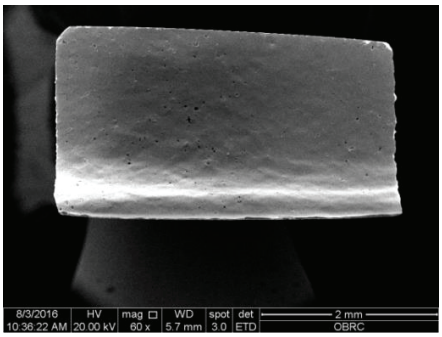
(e) 1% Resin infiltrated ceramic-25x

(f) 1% Resin infiltrated ceramic-50x

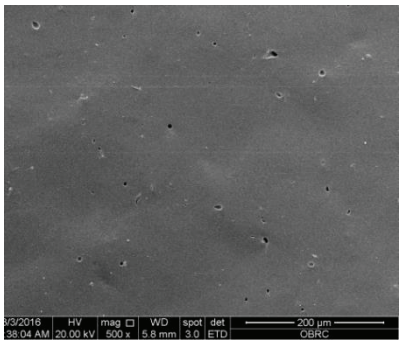
(g) 2% Resin infiltrat ed ceramic-25x

(h) 2% Resin infiltrated ceramic-50x

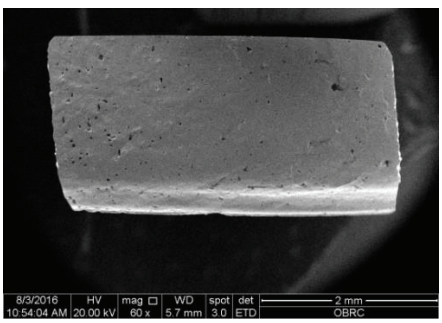
Figure 1. Fracture surface at the crack initiation point of specimens at magnification of 25x and 50x. (Images a-b) IPS e-max Ceram (control), symmetrical crack patterns (red arrows) are presented. (Images c-h) 0.5%, 1% and 2% resin-infiltrated ceramic, asymmetrical crack patterns with deflection (yellow arrows) are presented.



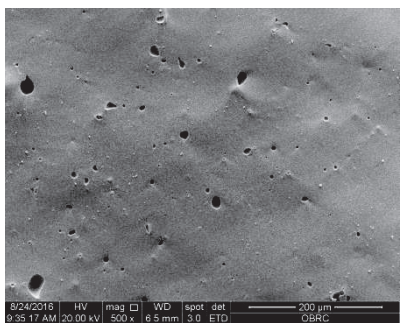
(a) IPS e-max Ceram – 60x



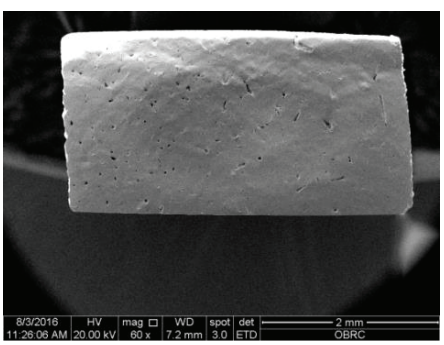
(b) IPS e-max Ceram – 500x



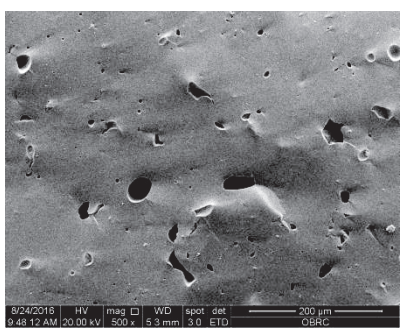
(c) 0.5% Resin infiltrated ceramic-60x



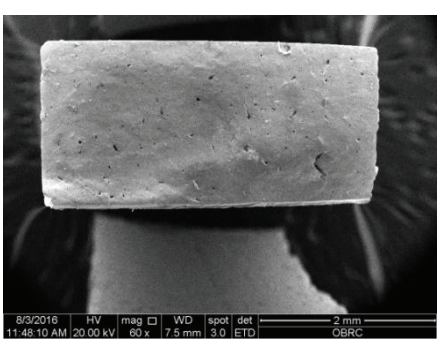
(d) 0.5% Resin infiltrated ceramic-500x



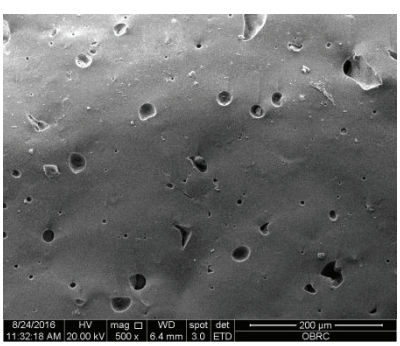
(e) 1% Resin infiltrated ceramic-60x



(f) 1% Resin infiltrated ceramic-500x



(g) 2% Resin infiltrated ceramic-60x



(h) 2% Resin infiltrated ceramic-500x

Figure 2. SEM micrograph of fracture surface of specimens at magnification of 60x and 500x. (Images a-b) IPS e-max Ceram (control), small round porosities distribute. (Images c-h) 0.5%, 1% and 2% resin-infiltrated ceramic, larger porosities and tube-like porosities are presented.

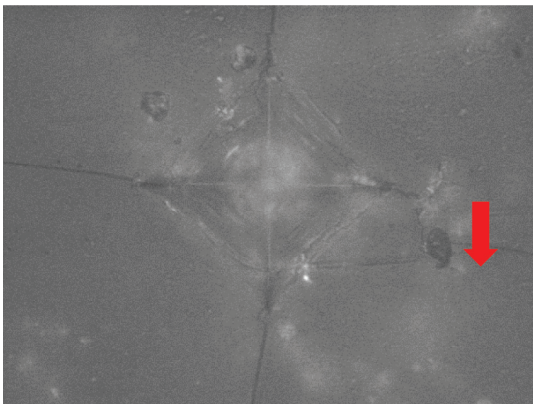


Figure 3. Illustration of optical light micrograph of Vickers' indentation on resin-infiltrated ceramic specimen that shown the crack deflection at polymer-ceramic interfaces.(red arrow)

phase of resin infiltrated ceramic can increase crack resistance [14]. These effects can improve the durability of the material and decrease chipping of veneering ceramic [14].

Although the polymer infiltration has an effect to the fracture toughness of veneering ceramic, the effect also depends upon the amount of volume fraction. This study, the effect was statistically significant when the volume fraction was up to 2%. This result corresponds with the findings by Coldea et al [11], they reported that the high volume of polymer fraction in polymer infiltrated ceramic group had high flexural strength and stain resistance but lower elastic modulus and hardness.

In this study, the maximum volume of resin infiltrated ceramic was end up at 2% although the fracture toughness result showed promising higher when the volume fraction increase more than 2%. The preliminary study found that the specimen was dimension, after firing difficult to hold their dimension. Moreover, the color of the resin infiltrated ceramic seemed to change a little bit grayish. This may be due to the residual acrylic fiber that was unable to burn out completely or burn out vapor was incorporated in the ceramic and also the higher amount of resin infiltrated might showed some affected on the original color of ceramic. Therefore, the highest volume fraction of this study was end up at 2% by volume. There should be further studies to increase more volume fraction and improve the properties of this material, including color are required.

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Competing interests : None

Ethical approval : No requirement

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