Fracture Toughness Comparison of Three Indirect Composite Resins Using 4-Point Flexural Strength Method

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Abstract

Objectives The advantages of indirect composite restorations such as less crack formation during their computer-aided design/computer-aided manufacturing process, compared with ceramic restorations, have resulted in their growing popularity. However, restoration failure is a major concern with regard to the long-term clinical success of restorations and may occur as the result of propagation of a crack originated from an internal flaw in the restoration. This study aimed to compare the fracture toughness of three indirect composite resins.

Materials and Methods In this *in vitro* experimental study, 10 specimens measuring 3 × 3 × 18 mm were fabricated of Gradia, Crios, and high impact polymer composite indirect composites. A single edge notch with a diameter < 0.3 mm and 0.3 mm length was created in the 9 mm longitudinal dimension of specimens using a no. 11 surgical scalpel. The specimens were then subjected to 4-point flexural strength test in a universal testing machine with a crosshead speed of 0.1 mm/s until failure.

Statistical Analysis Data were analyzed using IBM SPSS Statistics via one-way analysis of variance (ANOVA) and Tukey's HSD (honestly significant difference) test. The statistical power was set at $p < 0.05$.

Results One-way ANOVA showed a significant difference in fracture toughness of the three composite groups ($p = 0.000$). According to the Tukey HSD analysis, the fracture toughness of HIPC was significantly higher than that of the other two composites. The fracture toughness of Gradia was significantly lower among all.

Conclusions Within the limitations of this study, the results showed that high temperature-pressure polymerization can increase resistance to crack propagation and subsequently improve the clinical service of indirect composite restorations. Although we do not know the filler volume percentage of HIPC, it seems that filler volume percentage of the composite is inversely correlated with fracture toughness.

Keywords
- composite resins
- computer-aided design
- fracture toughness

Introduction

Composite resins are extensively used as a key material for tooth restoration. Optimal esthetics and biocompatibility, chemical stability, and easy clinical application have led to the increasing use of composite resins for restorative and esthetic dental treatments.[1] At present, use of indirect composite restorations has increased to overcome the problems of direct composite restorations such as inappropriate proximal and occlusal morphology, inadequate wear resistance, and suboptimal mechanical properties.[2,3]

Indirect dental composites are increasingly used in dental laboratories for indirect fabrication of inlays, onlays, and crowns.[4] The application of these restorative materials has greatly increased due to innovations in their fabrication and processing.[5] The polymerization process of indirect

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composite resins results in higher degree of conversion and higher rate of cross-links after polymerization, leading to improved mechanical properties.[6]

The main advantages of indirect composite restorations compared with ceramics include lower hardness and stiffness, lower antagonistic wear, lower brittleness, lower frequency of catastrophic failures, less chipping, and less crack formation during the fabrication process by the computer-aided design/computer-aided manufacturing (CAD/CAM) technique, and no need for crystallization or additional curing cycles after CAD/CAM milling.[7-10] Moreover, CAD/CAM composites have higher marginal adaptation than ceramics; thus, they are more suitable for cases requiring a thin margin or clinical conditions where tooth preparation is not required, because these composites have lower risk of chipping during their fabrication process.[11]

Restoration failure is a major concern with regard to long-term success and longevity of restorations.[1] Fracture toughness is a valuable parameter to determine the fracture resistance of materials.[12] Fracture toughness is an inherent property of a material that indicates resistance to crack propagation. It quantifies the energy required for the formation and propagation of a crack in a material that would lead to catastrophic failure. In general, the larger the defect, the lower the level of stress required for the fracture would be, because in this situation, stresses that are normally tolerated by a material accumulate at the defect margin. Fracture toughness is a suitable factor for prediction of clinical service of composite restorations. High rate of fracture toughness indicates a material with low susceptibility to chipping or fracture.[13]

Several techniques are commonly employed for the measurement of fracture toughness. The most commonly used techniques for the measurement of fracture toughness of dental materials include single edge notch and short rod chevron notch test using cylindrical, rectangular, and prismatic specimens.[14]

Although wear, surface roughness, and color stability are no longer considered as serious clinical challenges of composite restorations, restoration failure due to fracture is still a major concern with regard to composite restorations.[15] Some studies found no significant difference in fracture toughness of ceramic and indirect composite restorations fabricated by the CAD/CAM technology.[16,17] However, some others reported a higher fracture toughness for ceramic restorations compared with indirect composite restorations.[18,19] Considering the existing controversy in the results of relevant previous studies and introduction of new laboratory composites into the market, this study aimed to assess the fracture toughness of Gradia, Crios, and high impact polymer composite (HIPC) laboratory composite resins. The null hypothesis tested was that there are no differences in fracture toughness among the tested composite resins.

Materials and Methods

Preparation of Specimens

►**Table 1** presents the composite resins used in this in vitro experimental study. The CAD/CAM blocks of HIPC (Bredent, Germany) and Crios (Coltene, Germany) composite resins were sectioned by a Mecatome (201; Pressi, France) to fabricate 10 specimens of each composite measuring 3 × 3 × 18 mm. For the fabrication of Gradia (Indirect system; GC, Japan) composite specimens, a two-piece stainless steel mold with internal dimensions of 3 × 3 × 18 mm was used, and 9 increments of composite, each with 2 mm thickness, were packed in the mold. Each layer was cured for 40 seconds using a LED light curing unit (Woodpecker; Qudent, China) with 375 to 400 nm wavelength and 800 mW/cm^2 light intensity. The accuracy of light curing unit was first checked by a radiometer. Next, the specimens were removed from the molds and placed in a laboratory light curing unit (Eurolight, Taiwan) under halogen light for 15 minutes according to the manufacturer's instructions.

For the measurement of the fracture toughness of laboratory composites by the crack propagation technique, a single edge notch with less than 0.3 mm diameter and 0.3 mm length was created in the 9 mm longitudinal dimension of specimens using a #11 surgical scalpel.

Fracture Toughness Testing

To measure the inherent fracture toughness of laboratory composite specimens by the crack propagation technique, the notched specimens were subjected to four-point flexural strength test in a universal testing machine (ZwickRoell, Germany) based on ASTM STANDARD E399–83. The inner span was 10 mm and the outer span was 18 mm. The load was applied at a crosshead speed of 0.1 mm/s until fracture. The fracture toughness was calculated using the formula below:

$$K = \frac{P \sqrt{a}}{b \times d}$$

Where "P" is the load at fracture (MPa), "a" is the width of specimen in millimeters, "b" is the thickness of specimen

Table 1 Indirect composite resins used in this study

Material	Manufacturer	Resin matrix	Fillers
Brilliant Crios	Coltene	Cross-linked methacrylates (bis-GMA, bis-EMA, TEGDMA)	Glass and amorphous silica, 70%wt, 51% vol
Gradia	GC	UDMA, methacrylate copolymer	Microfine ceramic/prepolymerized filler, 75% wt, 65% vol
HIPC[a]	Bredent	Amorphous cross linked polymethyl methacrylate	Microceramic fillers

[a] High-impact polymer composite: The composition of this composite has not been clearly disclosed by the manufacturer.
Abbreviations: bis-EMA, bisphenol A diglycidyl methacrylate ethoxylated; TEGDMA, triethylene glycol dimethacrylate.

in millimeters, and “*d*” is the diameter of the notch also in millimeters.

Data were analyzed using IBM SPSS Statistics via one-way analysis of variance (ANOVA) and Tukey HSD. The statistical power was set at $p < 0.05$.

Results

►**Table 2** and ►**Fig. 1** present the results of the fracture toughness test in the three composite groups. One-way ANOVA revealed a significant difference in fracture toughness among the three composite groups ($p = 0.000$). Tukey HSD test showed that the fracture toughness of HIPC was significantly higher than that of other two composites ($p = 0.000$). The fracture toughness of Gradia was significantly the lowest among all (►**Table 3**).

Discussion

In this study, the single edge notch technique was used to measure the fracture toughness of indirect composite specimens. This technique is commonly used for this purpose due to its simplicity and ease of use. The narrow notch created in the specimens in this technique provides the sharp tip required for fracture toughness test. The main advantage of this technique is simple creation of the notch and its accurate measurement.[12] Presence of an initial crack for simulation of an internal flaw is imperative for fracture toughness test. These defects are formed in the clinical setting as the result of fatigue due to mastication.[20]

The parameters related to the mechanics of fracture such as static fracture toughness are suitable for the detection of dental composite defects. Static fracture toughness refers to the resistance against propagation of defects and flaws in brittle materials, which can cause catastrophic failure under applied forces.[21]

The magnitude of fracture toughness depends on several factors such as the type of composite, the type of polymer matrix,[22,23] filler percentage,[1,24] size and distribution of filler particles,[12,21,22] shape of filler particles,[21] surface treatment of fillers,[25,26] and polymerization under pressure and heat.[27] Previous studies on fracture toughness of CAD/CAM materials revealed that the difference in fracture toughness was due to different composition and microstructure of materials. Difference in size and distribution of the crystalline phase is also responsible for different behaviors of materials.[28] In the present study, difference in distribution of filler particles, fabrication process, and rate of polymerization (degree of conversion) led to difference in fracture toughness of composite resins.

HIPC is a composite with an amorphous, cross-linked polymethyl methacrylate polymer matrix. Thus, it has physical properties superior to those of the conventional polymethyl

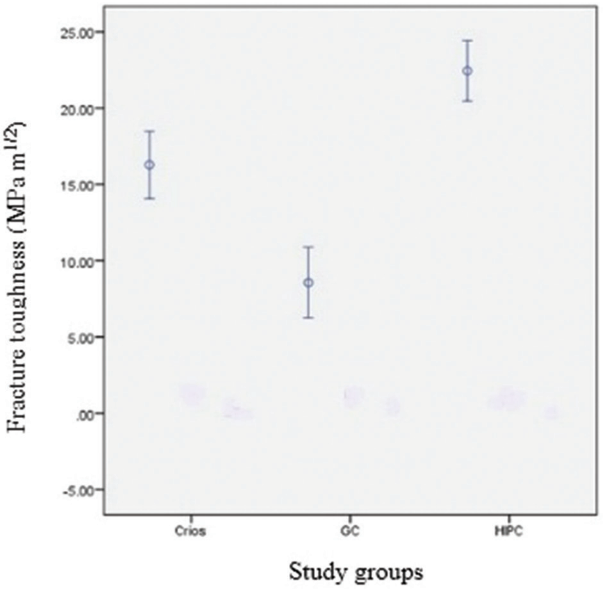

Fig. 1 Mean fracture toughness of the study groups with 95% confidence interval.

Table 2 Mean, standard deviation, minimum, and maximum fracture toughness of the study groups (MPa m1/2) (*n* = 10)

Composite	Minimum	Maximum	Mean	Standard deviation
Crios	9.66	21.40	16.2790	3.08478
Gradia	5.51	16.42	8.5630	3.25908
HIPC	19.04	27.80	22.4480	2.76390

Abbreviation: HIPC, high-impact polymer composite.

Table 3 Comparison of the mean fracture toughness of the study groups via Tukey HSD test

(I) composite		Mean difference (I-J)	Standard error	Significant
GC	Crios	7.71600[a]	1.36080	0.000
	HIPC	−6.16900[a]	1.36080	0.000
GC	Crios	−7.71600[a]	1.36080	0.000
	HIPC	−13.88500[a]	1.36080	0.000
HIPC	Crios	6.16900[a]	1.36080	0.000
	GC	13.88500[a]	1.36080	0.000

Abbreviation: HIPC, high-impact polymer composite.
[a]The mean difference is significant at the 0.05 level.

methacrylate. It is polymerized under 250 bar pressure at 120°C. The manufacturer claims that up to 99.9% of methyl methacrylate converts to polymethyl methacrylate. Thus, it seems that presence of strong, compact bonds and complete polymerization of composite block as well as the presence of micro-ceramic fillers have resulted in higher fracture toughness of HIPC (Kf = 22.44) compared with Crios and Gradia. Search of the scientific literature by the authors revealed no study regarding the fracture toughness of HIPC.

Gradia is a hybrid microfill reinforced composite resin. In this composite, a reinforced bond exists between the organic and mineral fillers. Also, it has a highly filled resin matrix that results in high mechanical properties due to its interactions with the filler particles. Since polymerization of this composite was not performed under pressure and heat in the present study, it seems that the internal defects of Gradia are higher than those of the other two composites.[9] This factor along with its different chemical and molecular structure and filler volume percentage resulted in significantly lower fracture toughness of this composite compared with the other two composites (Kf = 8.56).

Crios is a reinforced submicron hybrid composite with a modulus of elasticity similar to that of tooth structure. The internal stresses of this composite have been controlled by thermal methods for production of composite blocks. It is composed of amorphous silica and glass particles in combination with a resin matrix. In the present study, the fracture toughness of Crios composite (Kf = 16.27) with a low filler load was significantly higher than that of Gradia with a high filler load. This finding is in agreement with the results of previous studies showing that increasing the filler volume percentage to a certain level enhances the mechanical properties of composite resins.[29-31] Ikejima et al reported the optimal filler volume percentage to be 60% and added that filler load higher than 60v% is associated with a reduction in strength of composites.[29] Kim et al reported maximum fracture toughness in presence of 55v% filler load. Further increase in filler load decreased the fracture toughness. They attributed this reduction to the superimposition of cracks in high filler load, which would weaken the crack pinning by the fillers.[21] Similarly, in the present study, Gradia composite with 65v% filler load showed lower fracture toughness than Crios with 51v% filler load. However, Ilie et al demonstrated that the fracture toughness increased by an increase in filler load by up to 57v% and remained stable by further increase in filler volume to 65%. Further increase in filler load over 65% decreased the fracture toughness and this reduction was attributed to an increase in internal flaws following increased viscosity of the material.[12] Difference between their results and ours may be due to differences in the structure, chemical composition, and method of polymerization of composite resins since they only evaluated direct composite resins polymerized in normal conditions while we studied indirect composite blocks polymerized under pressure and heat. Sarabi et al indicated lower fracture toughness of Gradia (with 65% volume filler) compared with Z250 (with 60% volume filler). They attributed the lower fracture toughness of Gradia to its higher modulus of elasticity due to its higher filler load.[32] Thus, it seems that higher filler load of Gradia compared with Crios results in higher modulus of elasticity and higher risk of fracture. Plastic deformation of material increases the load required for crack propagation and occurrence of fracture.

On the other hand, Nguyen et al evaluated the mechanical properties of Paradigm MZ100 (3M ESPE) blocks with a polymer matrix made of bis-GMA (bisphenol A-glycidyl methacrylate) and experimental blocks with UDMA matrix and observed that the mechanical properties of blocks with UDMA matrix were superior to those of conventional blocks with bis-GMA matrix.[23] They attributed these superior mechanical properties to polymerization under pressure and heat and consequently fewer internal flaws in UDMA blocks.[9] The current results showed that the fracture toughness of Crios composite with bis-GMA matrix was significantly higher than that of Gradia composite with UDMA (urethane dimethacrylate) matrix. Difference between our findings and those of Nguyen et al may be due to the difference in filler loads of the two composites and lack of polymerization of Gradia samples under pressure and heat in the present study, which resulted in presence of higher number of internal flaws and defects in these composite specimens.

Conclusions

Within the limitations of this study, the results showed that high temperature-pressure polymerization can increase resistance to crack propagation and subsequently improve the clinical service of indirect composite restorations. Although we do not know the filler volume percentage of HIPC, it seems that filler volume percentage of the composite is inversely correlated with fracture toughness.

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Conflict of Interest
None declared.

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