

TOUGHNESS MEASUREMENT IN DIRECT RESIN COMPOSITES USING QUANTITATIVE FRACTOGRAPHIC ANALYSIS

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ABSTRACT

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Objective: To outline a procedure to determine the fracture toughness of direct resin composites failing from “natural” flaws.

Methodology: Tensile (hour glass) tests ($n = 30$) of a conventional hybrid dental composite (Tetric EvoCeram, Ivoclar Vivadent) were fabricated and fractured in uniaxial tension loaded at a crosshead speed of 1 mm/min (≥ 10 MPa/s). The fracture toughness of the material was then calculated using the stress at failure and measurement of the crack size from fractographic analysis using SEM. Hardness (H) measurements were taken using a Vickers pyramidal diamond indenter. Elastic modulus (E) was calculated from the E/H ratio using a Knoop indenter.

Results: The values for fracture toughness found were similar to other Bis-GMA based dental composites 0.5 ± 0.2 MPa \sqrt{m} . The Vickers Hardness was 509 ± 27 MPa and the Knoop Hardness was 495 ± 14 MPa using 0.5 kg/30 s, while the elastic modulus was 9.5 ± 1.4 GPa.

Conclusion: The differences found in fracture toughness between this study and previous published studies are most likely due to variation in technique and material. Quantitative fractographic analysis offers a different method to evaluate the toughness of direct resin composites.

Keywords: fracture toughness, resin composites, fractography, dental materials.

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1. Introduction

Dental composites are a mixture of polymers and glass particles used in dental restorations to mimic the appearance and performance of teeth and are often used to repair damaged teeth [1]. Their mechanical properties have improved over the last years and consequently a lot of research has been performed to assess these properties and how they are affected by variations in particle size, polymerization depth, and viscosity [2-4].

In one longitudinal study it was found that out of 926 restorations investigated, 8% failed by fracture. This number increases to 18% when considering the failure of only resin composites restorations [5]. In a review of prospective studies, it was found that fracture of dental restorations is the most common cause of restoration failures in the first 5 years. [6] Therefore fracture toughness is a property that has received a lot of attention in dental composites. Fracture toughness is a property that represents the ability of some materials to resist crack propagation [7]. The load continuously applied in different directions with temperature and humidity variation leads to the progressive degradation and failure of the restoration, mainly due to the crack propagation. The introduction of imperfections in the material during processing, finishing, and/or in service has a high impact on the restoration failure probability. The organic matrix of dental composites has viscoelastic properties, which means that the response to stress will

be time dependent. Under a rapid stressing rate, the stress-strain response will be primarily linearly elastic. Under a slow stressing rate, the stress-strain response will be viscoelastic [8]. The presence of filler also has a high impact on the mechanical properties [9]. The greater the amount of fillers, the greater the modulus of elasticity. The combination of the viscous matrix and greater modulus reinforcement leads to a tougher composite [10]. The main concern with the increase in fracture toughness of the restorative material is maintaining or improving the lifetime service in the oral environment while maintaining the esthetic value. The mechanical properties of dental composites have been highly improved in the last few years [11]. Even though there are many tests done to correlate *in vitro* data with the clinical behavior of dental composites, there still is a need to improve these methods to be more realistic and comparable to material behavior while in service [12]. There are several ways to measure fracture toughness such as the single edge notch test, compact tension test, or double torsion test [13]. However, the tests for toughness, in almost all cases for resin composites, involve large crack techniques. Resin composites mostly fail from small cracks so it is important to develop small crack techniques to determine fracture toughness. The quantitative fractography method offers the advantage of using flaw sizes of those encountered in service, i.e., small cracks. There are two approaches when using fractographic procedures to determine

toughness: controlled crack techniques [14] and direct observation of “natural” flaws or cracks [15]. “Natural” here means cracks or processing defects caused by fabrication and handling of the material before testing. It was not possible to develop controlled cracks in the material so the controlled crack technique could not be used. This result of difficulty in forming controlled cracks agrees with a similar observation in a previous study by other authors [16]. Using the “natural” crack means that an assessment of the fracture toughness of the material as used in clinical practice can be found. Finishing operations will yield cracks of size on the order of “natural” cracks. The advantage of this technique over others is that it provides a tool for forensic analysis. Once the toughness is determined from flaws of the size considered in this work, any strength from field failures of the same material will be able to be determined.

There are limited studies in the field of dental composites using quantitative fractographic analysis. Therefore, the aim of the study was to outline a procedure to determine the fracture toughness of direct resin composites failing from “natural” flaws. The materials used in this study are compared to those in analogous studies using different materials and fabrication techniques.

2. Methodology

The material used in this study was a hybrid conventional dental composite (Tetric EvoCeram, Ivoclar Vivadent)¹. The Tetric EvoCeram composite is a light cured resin composite. The standard composition and physical properties of Tetric EvoCeram are listed in Table 1 as given by the manufacturer [17]. Tensile “hour glass” samples with average cross-sectional dimensions of 1.76 mm by 1.51 mm and a 3 mm gauge length were made by filling a mold with the resin and curing the samples for 10 seconds each. The mold was covered with a thin Mylar strip to ensure a flat surface. The curing process was done using an LED light curing unit (Bluephase Style, Ivoclar Vivadent) which emits light with an approximate intensity of 1000 mW/cm². The light cure unit was calibrated prior to use by means of a dental radiometer (BluePhase meter II, Ivoclar Vivadent). The tip of the light cure unit was positioned directly on top of the Mylar strip and stabilized with the plastic tip. Once the samples were cured they were polished with very light pressure to ensure that the corners were smooth. This was done using Sof-Lex² extra thin polishing discs of medium grit followed by fine grit at 6000-10000 rpm.

The polished samples were then broken in tension using a universal tensile testing machine³ loaded at a crosshead speed of 1 mm/min (≥ 10 MPa/s) using an anti-torsion parallel holder, and the load at failure, P , was recorded for each sample. The load-displacement graphs were linear until there was fracture with little or no non-linear behavior before fracture. The fracture stress, σ , was calculated from the load at failure and the dimensions of each specimen using equation 1:

$$\sigma = \frac{P}{A} \quad (1)$$

where A is the cross section within the narrow region (gauge section) of the specimen (3 mm). Any sample that did not break in the narrow cross section was discarded and not used for the data presented. Weibull parameters were calculated by maximum likelihood estimation according to ASTM C1239 – 13[18].

Table 1. Composition and physical properties of the Tetric EvoCeram Dental Composite.

Standard – Composition (in weight %)	
Bis-GMA, Urethane dimethacrylate, Ethoxylated Bis-EMA	16.8
Barium glass filler, Ytterbium trifluoride, Mixed oxide	48.5
Prepolymers	34.0
Aditives	0.4
Catalysts and Stabilizers	0.3
Pigments	<0.1
Physical Properties	
Flexural Strength (Mpa)	120
Flexural Modulus (Mpa)	10,000
Compressive Strength (Mpa)	250
Vickers Hardness HV 0.5/30 (Mpa)	580
Density (g/cm ³)	2.10

Fracture toughness was calculated using the quantitative fractographic analysis. The method uses optical and scanning electron microscopy to locate and measure the size of the origin of the fracture for each specimen [13]. Once the flaw, or crack, at the origin starts to propagate it travels with increasing speed spreading out in all directions. As the speed increases, the surface increases in roughness. The origin of the fracture can be determined by the observation of the characteristic markings surrounding the fracture origin on the fracture surface. Generally surrounding the fracture origin there is a relatively smooth region, sometimes called the “mirror” region, that transitions to a slightly rougher region, sometimes termed the “mist” region. These regions and other markings, such as twist hackle, can be used to identify the location of the failure origin [13, 15]. The fracture origin is situated approximately at the center of the surrounding topography. All surface cracks were treated as elliptical cracks for calculating the fracture toughness. Images were taken using a scanning electron micrograph SEM⁴. Once the crack sizes were obtained the fracture toughness was calculated using equation 2 where σ is the stress at failure, a the crack size, and Y is a geometric factor of loading, the crack shape, and location. Y was calculated using the solutions of Newman and Raju for locations at the surface of the crack or internal cracks [19]:

$$K_c = Y\sigma\sqrt{a} \quad (2)$$

The hardness, H , was determined in a conventional manner using a Vickers pyramidal diamond with an indentation load of 0.5 kg at a loading and unloading time of 30 s [20]. The Vickers diamond was used for hardness because it offers an equi-axed diamond

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⁴ Phenom Pro SEM, Phenom World, Eindhoven, Netherlands

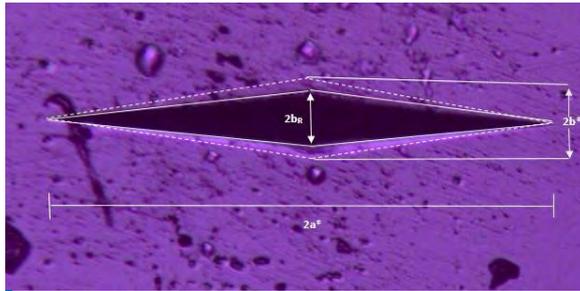


Figure 1. Optical Micrograph of Knoop Indentation Demonstrating the Measurement for Elastic Modulus.

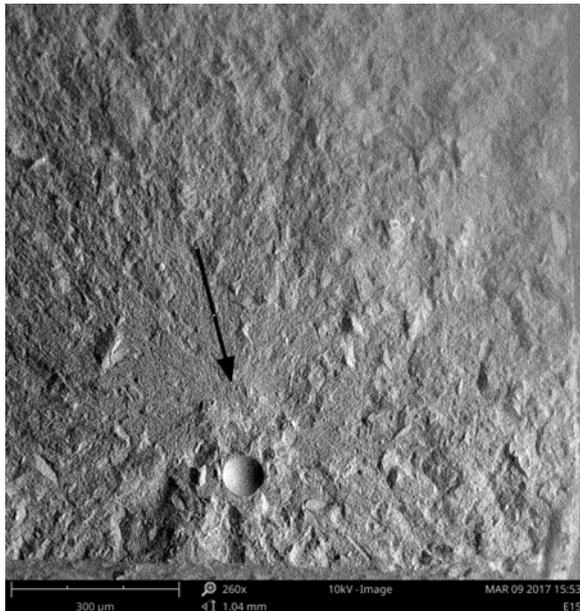


Figure 2. Example of an internal crack. The arrow points to fracture origin.

and thus increases the number of measurements and increases precision. For completeness, the Knoop hardness value was also determined at 0.5 kg at a loading and unloading time of 30 s.

The elastic modulus was determined using a technique developed by Marshall [21] and Conway [22]. The method used the geometry of the Knoop indenter, i.e., due to the asymmetrical shape of the indenter, one direction of the impression is elongated and the transverse direction is considerably shortened. Upon loading, the geometry of the impression will be determined by the shape of the diamond (cf. Figure 1). Upon unloading, the shorter direction ($2b^*$) will contract ($2b_R$) due to the elasticity of the material. The elongated end ($2a^*$) will not be measurably changed because of the length. Thus, the difference between the original measurement of the diagonals (from the shape of the diamond) and the impression on the material will provide a measure of the elasticity of the material. The E/H ratio can be calculated from the measurement of the diagonals as shown in equation 3 [22]:

$$\frac{E}{H} = 1 - 2[\tan \gamma(1 - \nu^2)] \left(\frac{b^*}{b_R}\right)^2 \quad (3)$$

where H is the Knoop hardness, ν is Poisson's ratio (0.3), $\gamma = 75$ (the average half angle of the Knoop indenter), b^* is half the minor diagonal at maximum load, and b_R is half the residual minor diagonal that is measured. The value of b^* can be calculated for the Knoop indenter because it is related to the major diameter, i.e., $b^* =$

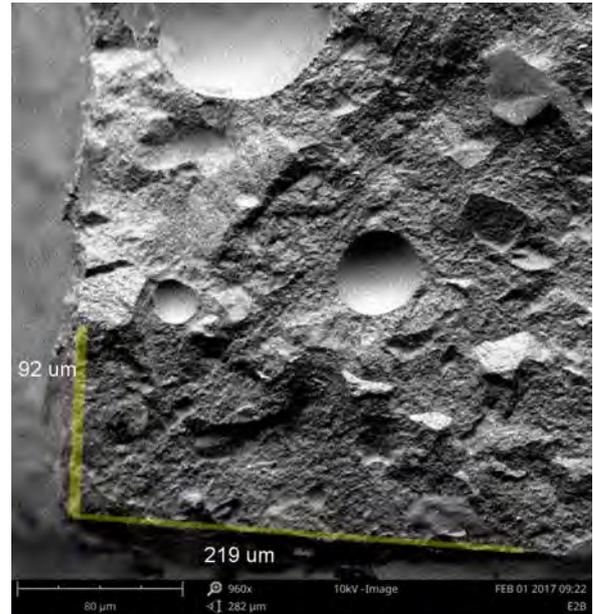


Figure 3. Example of a corner crack. The lines indicate the dimensions of the crack at the fracture origin.

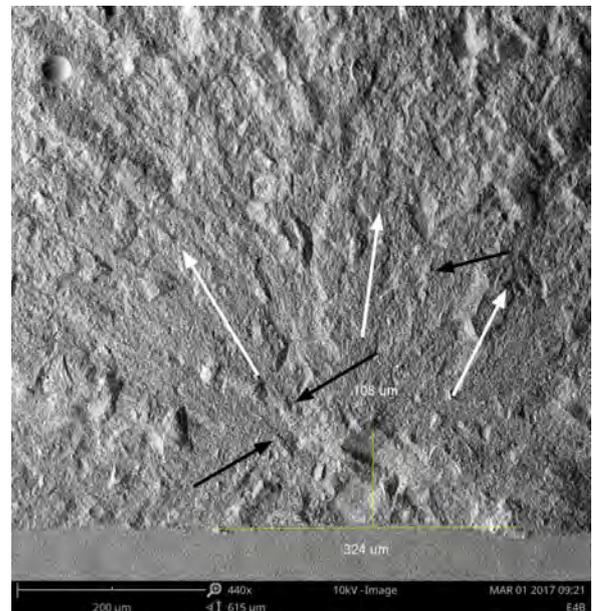


Figure 4. Example of a surface crack. Yellow lines indicate the width and depth of the crack at the fracture origin. Black arrows indicate direction of crack propagation away from the crack origin. White arrows indicate twist hackle marks on the fracture surface.

Table 2. Average values of obtained data.

Physical Property	Standard Deviation	
Fracture toughness ($\text{MPa}\sqrt{\text{m}}$)	0.5	0.2
Vickers Hardness (MPa)	509	27
Y (geometric constant)	1.28	0.04
Elastic Modulus (GPa)	10	1.7
90% Confidence Interval		
Unbiased Weibull Modulus	4.4	3.4 - 5.6
Characteristic Strength (MPa)	26	24 - 28

$7.11a^*$, where a^* is half the major diagonal, assumed to be the same before and after indentation.

3. Results

The average values of toughness, hardness, Y , elastic modulus, Weibull modulus, and characteristic strength can be found in Table 2. The detailed strength and toughness data obtained are presented in Table A-1 in the [Appendix](#). The average fracture toughness for this material was found to be $0.5 \pm 0.2 \text{ MPa}\sqrt{m}$. Of the 30 samples investigated, most cracks originated at the surface of the material, often at a corner that was polished. Only 3 of the 30 samples had internal crack origins. An example of an internal origin is shown in Figure 2. Figures 3 and 4 show the more common edge and corner cracks at the surface of the samples. They are also representative of the measurement technique. Figure 3 also illustrates the presence of voids in various samples often found near crack origins. The Vickers Hardness was $509 \pm 27 \text{ MPa}$ using $0.5 \text{ kg}/30 \text{ s}$, while the elastic modulus was $10 \pm 1.7 \text{ GPa}$.

A Weibull graph for the data is presented in Figure 5. The unbiased Weibull modulus and characteristic strength were calculated using MATLAB and found to be 4.4 (90% confidence intervals as per [16]: 3.4 - 5.6) and 26 MPa (90% confidence intervals as per [16]: 24 MPa - 28 MPa) respectively. The locations of the fracture origins are also depicted on the Weibull graph. All origins appear to be uniformly distributed.

4. Discussion

While the values for fracture toughness could not be found from the manufacturer, the value obtained agrees with other Bis-GMA based dental resin composites [23]. Our value is less than the value of $1.11 \text{ MPa}\sqrt{m}$ found by Cho et al. for the same material [24] and less than the value of $1.1 \text{ MPa}\sqrt{m}$ found by Quinn et al. for materials that are resin based, but manufactured in a different way [16]. Note that a different technique was used by Cho et al. to measure the fracture toughness. The notched bend test is noted for producing increased values of fracture toughness unless the notch is artificially sharpened [13]. In the present study, as well as the one in Quinn et al., we were not able to produce a sharp crack artificially due to the viscoelastic nature of the material [16]. The condition at the crack tip can explain the difference in the numerical values between the notched bend test and the "natural" flaws. The material used in Quinn et al. [16] is an indirect resin composite block (Paradigm, 3M ESPE, MN) used for indirect restorations. The composition of the indirect material used in their research contains a high fraction of filler particles (85 wt% ultrafine zirconia-silica ceramic to reinforce a highly crosslinked polymeric matrix). Thus, as the authors state, this material is closer to ceramic behavior. The materials used in this research is a direct dental composite material which contains 40-48 wt% Baria-aluminosilicate glass filler as well as 34.0% pre-polymer fillers. In addition, the sample preparation was different in the two studies. The present study used a prefabricated mold followed by a light cure and then shaped for tensile specimens, while in the Quinn et al. article, a hard block was used and it was sectioned to get the desired shape for flexural tests. Thus, we should not expect the values to be comparable. While there are many fractographic studies of resin composites [25,

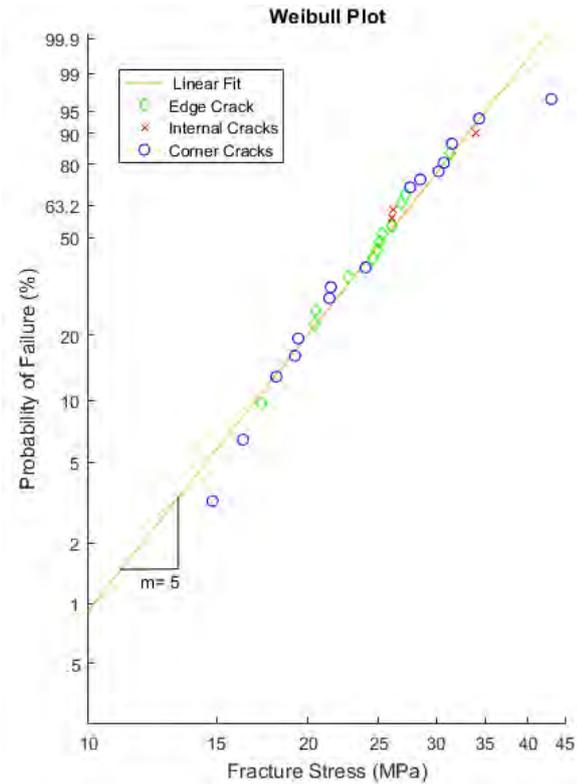


Figure 5. Weibull graph of the composite strengths.

26] and determination of toughness values for resin composites [27, 28], to our knowledge there is no record of toughness values for direct resin composites measured using the quantitative fractographic technique used here. Thus, we provide useful information for use in *in vitro* analysis because the size of the cracks are those expected in clinical failures. The results here and from Quinn et al. suggest that the fractographic technique may be used to determine differences in manufacturing techniques as well as differences in particle loading. Further research in this area should be pursued.

The unbiased Weibull modulus was 4 for this specific material, which is less than the value of 8 found by Quinn et al. for their material [16]. Of course, the Weibull modulus is just an indication of the distribution of the values of strength obtained. This distribution is related to the uniformity of the flaws in the material which, in turn, is related to manufacturing procedures and handling. Thus, both values found in the two studies are relatively low, indicating a wide spread of flaw sizes and locations. As observed in the Weibull graph, there does not appear to be an effect of the location of flaws as to the strength of the material. The characteristic strength was $26 \pm 2 \text{ MPa}$. Since the fracture initiating flaws were "natural", they were not controlled except by the fabrication and finishing procedure. The sizes should be comparable to those observed in clinical procedures. Better control of the fabrication procedures could result in greater toughness values, but most likely not greater than $\sim 1 \text{ MPa}\sqrt{m}$, and thus, in greater strengths for the same size flaws.

The method used to determine the elastic modulus in this work is relatively straightforward and unique for resin composites. Since the value agrees with the value provided by the manufacturer for flexural modulus, we think this is encouraging in that this presents

an easy method to obtain values for elastic moduli measurements in resin composites.

5. Conclusions

Quantitative fractographic analysis offers a different method to evaluate the toughness of direct resin composites. The advantage of this technique is that it occurs with the strength measurements. No additional testing is necessary. The other advantage of this technique to measure fracture toughness is that the flaws causing failure are of the size expected with the handling and finishing procedure used in clinical practice. More research is needed using the quantitative fractographic technique with resin composites to determine the effect of particle size and volume fraction as well as manufacturing techniques on the mechanical properties. The Knoop hardness technique to measure the elastic modulus offers a relatively easy technique to use for resin composites.

Author contributions

Equal contribution to the paper.

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Questions

1. The fracture toughness of the resin composites was measured using:

- a. Single edge notch test;
- b. Compact tension test;
- c. Quantitative fractography;
- d. Double torsion test.

2. The elastic modulus was calculated using:

- a. The slope of the stress-strain curve;
- b. The sonic modulus technique;
- c. Knoop indentation;
- d. Vickers indentation.

3. The value for the fracture toughness found was:

- a. In agreement with comparable studies;
- b. Lower than values found in comparable studies;
- c. Greater than values found in comparable studies;
- d. Not compared to values found in other studies.

4. Quantitative fractography uses what measurements to calculate fracture toughness?

- a. Crack size and stress at failure;
- b. Crack size and elastic modulus;
- c. Elastic modulus and stress at failure;
- d. Stress at failure and Vickers hardness.



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