FRACTURE TOUGHNESS OF VARIOUS CORE MATERIALS

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This investigation evaluated the fracture toughness(K_{IC}) of eight currently available core materials, and relate the fracture toughness value to fractography analysis and surface characteristics using a atomic force microscope (AFM).
Single-edge notched (SEN) test specimens (n=10) and compact tension (CT) test specimens (n=10) were prepared conforming to the ASTM Standard E-399 for a high copper amalgam, three composite core materials (Core-Max Ⅱ, Core Paste, Bisfil Core), two reinforced composite core materials (Ti-Core, Ti-Core Natural), a resin-modified glass ionomer core material (Vitremer), and a conventional glass ionomer core material (Ketac-Molar). The specimens were tested with an Instron Universal Testing Machine. The maximum loads were measured to calculate the fracture toughness (K_{IC}). Thereafter, fracture surfaces of SEN specimens of each material were investigated for fractography analysis using scanning electron microscope. And, disc-shaped specimens with 1mm thickness were fabricated for each material and were investigated under AFM for surface morphology analysis.

The results were as follows:
1. Bisfil Core showed the highest mean fracture toughness regardless of test methods.
2. For the tooth-colored materials, Ti-Core Natural exhibited the highest fracture toughness.
3. Ketac Molar showed a significantly low fracture toughness when compared with the amalgam and the composite resin core materials($p<0.05$).
4. The fracture toughness values obtained with the single-edge notched test, except Ketac Molar, were higher than those obtained in the compact tension test.
5. SEM revealed that the fracture surface of high fracture toughness material was rougher than that of low fracture toughness material.
6. AFM revealed that the surface particles of the composite resins were smaller in size, with a lower surface roughness than the glass ionomer core materials.

Key Words
Core material, Fracture toughness, Fractography analysis, Atomic force microscope

Core materials utilized in dentistry need to be resistant to mechanical fracturing from the forces encountered during mastication.[41] For selection of core build-up materials, evaluation of the hardness, tensile, and compressive strength should be evaluated. For materials to be used in an oral environments, resistance to corrosion, fatigue, and creep are also important.[24]

Strength and elastic modulus have long been taken as indicators for the usefulness of dental materials. Although tensile and compressive strength are important mechanical properties of a material, these parameters do not take into consideration the presence of flaws or other stress concentrators, and varies with specimen geometry, size, and lateral pressure.[56] The fracture toughness is a more realistic measure for the resistance to fracture.

In brittle materials such as dental resin composites or glass ionomer, microcracks which exist on the surface or inside the material decrease the strength. The stress intensity factor (K) specifies the stress distribution around a crack tip by an external force. K is expressed as K_I, K_{II}, and K_{III}, according to the loading mode, and K_I, the stress intensity factor for the tensile mode, is particularly important in brittle materials.[8,20] Crack extension will occur when stress and strain at the crack tip exceed a critical value of material, which is called the fracture toughness of materials. So, the definition of fracture toughness (K_{IC}) is a measure of the stress intensity at the tip of a crack or flaw which a crack propagates through a material in the unstable manner. This property has been related to the ability of a dental restorative material to resist both crack propagation and wear in the oral environment.[17]

Many researchers have examined the fracture toughness of dental materials using various testing methods, including the single-edge notched beam method,[11,14,23,24,36,53] the compact tension method,[27,29,51] the short rod with chevron notch method,[38,43,44] the double torsion method,[3,4,17,40,52] the indentation hardness method,[12,21] and the ring specimen method.[21,49] There is no standard method for the measurement of K_{IC} for brittle materials. Previous studies using different test methods have reported considerable scatter(10~50%) in the values for the K_{IC} of the same material.[28]

The atomic force microscope (AFM) probes the surface of a sample with a sharp tip, a couple of microns long and often less than 100 Å in diameter. The tip is located at the free end of a cantilever that is 100 to 200㎛ long. Forces between the tip and the sample surface cause the cantilever to bend or deflect. A detector measures the cantilever deflection as the tip scan over the sample. The measured cantilever deflections allow a computer to generate a map of surface topography.

Although amalgam, composite resin, and glass ionomer materials commonly have been used for core build-up, recent formulations have incorporated different combinations of materials. Evaluating the suitability of these new materials is necessary. Previous studies have determined the fracture toughness of amalgam,[35,36,46] composite resin,[11,17,32,33,34,36,45] and glass ionomer.[36] However, few statistical comparison was made between the different materials. And, few studies are available on the fracture toughness of the currently available core up materials.

The purpose of this study was to evaluate the fracture toughness of eight currently available core materials using two test methods, and relate the fracture toughness to fractography analysis using SEM and surface characteristics using AFM.

MATERIALS AND METHODS

Eight currently used core build-up materials were tested in this study. Products and manufacturers

Table Ⅰ. Materials tested in this study

Material	Type	Manufacturer
Bestaloy	High copper amalgam	DongMyung, Hwasung, Korea
Bisfil Core	Radiopaque light-cure composite with natural contrasting blue color	Bisco, Schaumburg, IL, USA
Core Max Ⅱ	Adhesive self cure composite	Sankin, Tokyo, Japan
Core Paste	Radiopaque self-curing core composite	DenMat, Santa Maria, CA, USA
Ti-Core	Titanium-reinforced hybrid-filled autocuring composite resin	Essential dental system, S. Hachensack, NJ, USA
Ti-Core Natural	Lanthanide-reinforced hybrid-filled autocuring composite resin	Essential dental system, S. Hachensack, NJ, USA
Vitremer	Resin modified glass ionomer	3M, St. Paul, MN, USA
Ketac-Molar	Glass ionomer cement in a capsule	ESPE, Seefeld, Germany

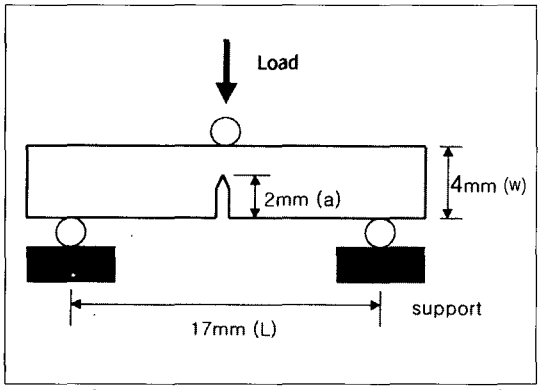

Fig. 1. Schematic representation of single-edge notched test.

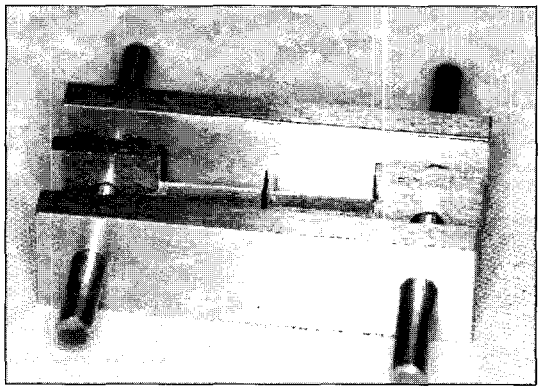

Fig. 2. Stainless steel mold for Single-edge notched specimen.

information is presented in Table Ⅰ.

SINGLE-EDGE NOTCHED (SEN) TEST

The single-edge notched test specimen configuration conformed to the American Society for Testing Materials (ASTM) guidelines for such a specimen (Standard E-399).

A mold was fabricated of stainless steel with a sharp stainless steel blade to form the centrally located notch. The dimension of the specimen obtained from the mold were 2.0mm × 4.0mm × 20mm with a 2mm central notch. (Fig. 1, 2)

Eight materials in current use for core build-up were studied. Manufacturer's instructions were carefully followed.

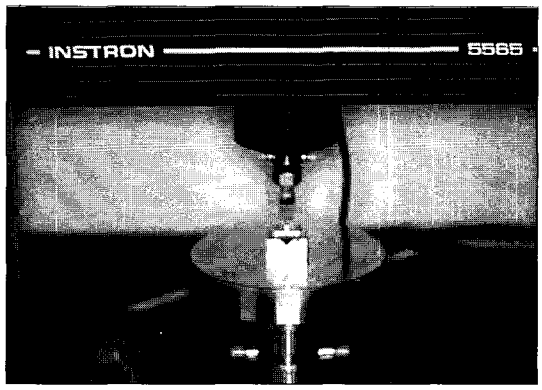

Fig. 3. Single-edge notched test.

The high copper amalgam (Bestaloy) was triturated according to the manufacturer's recom-

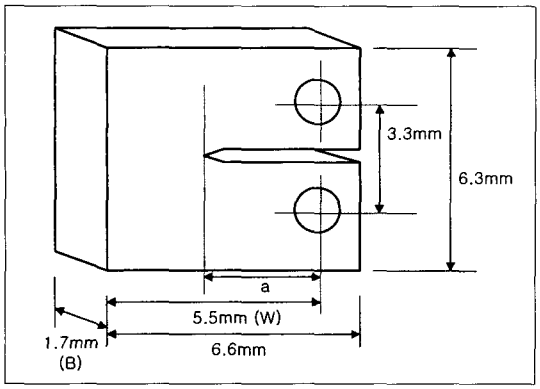

Fig. 4. Compact tension test specimen geometry.

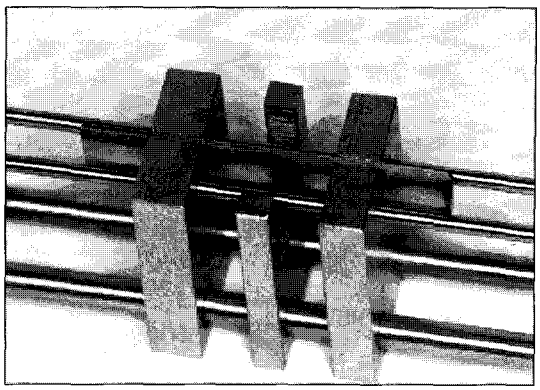

Fig. 5. Stainless steel mold for Compact tension specimen.

mendation and condensed into the mold in a conventional manner. The specimens were carved flush with the top of the mold and allowed to set for 30 minutes before removal from the mold. Bisfil Core was applied to the mold in increments, and light cured on the open part of the mold for 40 seconds. The mold was then opened, and the specimens were subjected to the curing light on the lateral surfaces for an additional 40 seconds on each side. Core Max II is supplied in a powder/liquid form, and the P/L ratio is 1:3. Core Paste, Ti-Core and Ti-Core Natural are supplied in a two-paste system. Equal portion of the base and catalyst were mixed until well-blended, then the mixture was applied to the mold and was allowed to set for 20minutes. Vitremer is a two part, powder/liquid composition. The mixtures of Vitremer were light cured using a 40-second exposure time for each surface. Ketac-molar supplied in a preencapsulated form was triturated in an amalgamator (ESPE Capmix, Germany) and injected into the mold.

Ten specimens of each material were prepared. The specimens were then lightly planed with abrasive paper to remove any irregularities from the mold. Measurements of the dimensional parameters for each specimen were recorded using a microscope interfaced with the Micro Vickers hardness tester (MVK-H2, Akashi Corp, Yokohama, Japan). A universal test machine (Instron model 5565, Instron Corp, Canton, MA) was used to apply a central load to the specimens in a three-point bending mode at a crosshead speed of 0.5mm/min. (Fig. 3) Visual examination of the fractured parts was performed to ensure that the fracture plane was through the notch and that it was perpendicular to the vertical and horizontal planes through the center of the specimens.

The fracture toughness was then calculated using the following equation :

$$K_{IC} = \left(\frac{PL}{b\,w^{1.5}}\right) f\left(\frac{a}{w}\right)$$

where

$$f\left(\frac{a}{w}\right) = \frac{3}{\alpha}\left(\frac{a}{w}\right)^{1/2} \cdot \left\{1.99 - \left(\frac{a}{w}\right)\left(1-\frac{a}{w}\right)\left[2.15 - 3.93\frac{a}{w} + 2.7\left(\frac{a}{w}\right)^2\right]\right\}$$

and

$$\alpha = 2\left(1+2\frac{a}{w}\right)\left(1-\frac{a}{w}\right)^{3/2}$$

where K_{IC}=fracture toughness; P=load at frac-

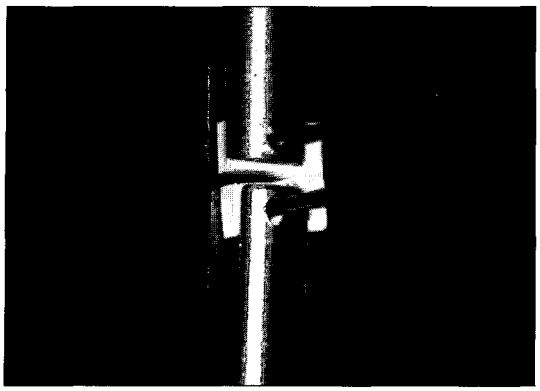

Fig. 6. Compact tension test.

ture; L=span distance between the support; w=width of the specimen; b=thickness of the specimen; and a=crack length.

Compact tension (CT) test

The compact tension specimen configuration and test fixture was taken from ASTM Standard E399-83. The specimen configuration is a rectangular type with dimensions 6.3mm × 6.6mm × 1.7mm.(Fig. 4. 5)

The materials were placed in a stainless steel mold. A sharp stainless steel blade was inserted into the mold to form a notch and two wires with 0.5mm diameter were inserted to form grip holes.

Ten specimens of each material were prepared. All materials were mixed and handled following the manufacturers' instructions. The rectangular specimen was then lightly planed with abrasive paper to remove any irregularities from the mold. The specimens were then tested in a tension mode in the universal testing machine (Instron Model 5565, Instron Corp, Canton, MA, USA) at a crosshead speed of 0.5mm/min with the direction of force perpendicular to the plane of the preformed notch in the specimen.(Fig. 6)

The fracture toughness was then calculated using the following equation :

$$K_{IC} = \left(\frac{PC}{B\,W^{0.5}}\right) \cdot f\left(\frac{a}{W}\right)$$

where

$$f\left(\frac{a}{W}\right) = \frac{2+\frac{a}{W}}{\left(1-\frac{a}{W}\right)^{3/2}}\left[0.866 + 4.64\left(\frac{a}{W}\right) - 13.32\left(\frac{a}{W}\right)^{2} + 14.72\left(\frac{a}{W}\right)^{3} - 5.60\left(\frac{a}{W}\right)^{4}\right]$$

where K_{IC}=fracture toughness; PC=maximum load prior to catastrophic fracture; B=average specimen thickness; W=average specimen width, dimension from the unnotched edge of the specimen to a plane centerline of the loading holes; and a=crack length.

Statistical analysis

The K_{IC} values of each of the fracture toughness test were statistically tested by a one way analysis of variance (ANOVA) using SPSS/PC+ software (SPSS, Chicago, IL, USA). LSD multiple comparison test was used to compare the correlations among the materials.

Fractography analysis

Each of the fractured SEN specimens were ground to 5mm in thickness and mounted with resin on SEM aluminum stubs. Thereafter, the fracture surfaces of the specimens were coated with a film of Au-Pd and examined in the scanning electron microscope (JSM-840A, JOEL, Japan).

Atomic Force Microscope

Disc-shaped specimen with 1mm thickness were fabricated for each material and were investigated under an AFM (Nanoscope Ⅱ, Digital Instrument, Inc, Santa Barbara, CA, USA) for surface morphology analysis. The scan rate was 2Hz, and the scan size was 5㎛ × 5㎛.

RESULTS

Fracture toughness

All specimens were fractured in a straight line from the preformed notch to the base, and the fracture surfaces were flat. The mean fracture toughness values and standard deviations for the two test methods are shown in Table Ⅱ, Ⅲ, and Fig. 7.

The One-Way ANOVA for single-edge notched test (Table Ⅳ) indicated significant differences among several of the core materials. The LSD test (Table Ⅵ) revealed that the mean fracture toughness of Ketac Molar was significantly lower than those of the other core materials($p<0.05$). There was no significant difference between the fracture toughness of Ti-Core and Vitremer core material($p>0.05$). The mean fracture toughness of these two materials were significantly lower than those of the other composites and high copper amalgam. And, there was no significant difference in the fracture toughness of Core Paste, Ti-

Table Ⅱ. Mean K_{IC}, SD of specimens from single-edge notched test

Material	n	Mean ($MN \cdot m^{-1.5}$)	SD ($MN \cdot m^{-1.5}$)
Bestaloy	10	1.497	0.174
Bisfil Core	10	1.547	0.208
Core Max Ⅱ	10	1.241	0.108
Core Paste	10	1.268	0.061
Ti-Core	10	0.918	0.140
Ti-Core Natural	10	1.325	0.131
Vitremer	10	0.787	0.206
Ketac Molar	10	0.441	0.091

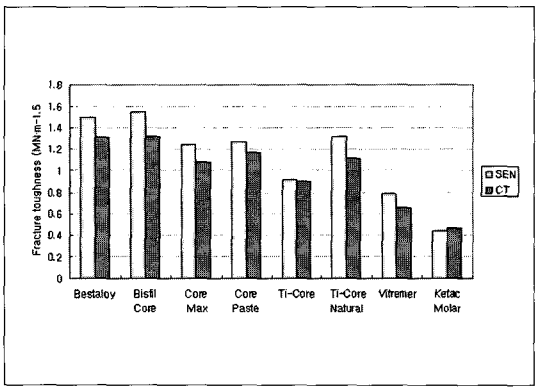

Fig. 7. Mean fracture toughness for core materials.

Table Ⅲ. Mean K_{IC}, SD of specimens from compact tension test

Material	n	Mean ($MN \cdot m^{-1.5}$)	SD ($MN \cdot m^{-1.5}$)
Bestaloy	10	1.312	0.098
Bisfil Core	10	1.326	0.118
Core Max Ⅱ	10	1.080	0.248
Core Paste	10	1.172	0.093
Ti-Core	10	0.899	0.146
Ti-Core Natural	10	1.113	0.224
Vitremer	10	0.654	0.144
Ketac Molar	10	0.459	0.061

Table Ⅴ. Analysis of variance of compact tension test

CT	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	6.744	7	.963	40.716	.000
Within Groups	1.704	72	2.366E-02		
Groups Total	8.448	79			

Table Ⅳ. Analysis of variance of single-edge notched test

SEN	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	10.153	7	1.450	65.811	.000
Within Groups	1.587	72	2.204E-02		
Groups Total	11.739	79			

Table Ⅵ. Multiple range test of single-edge notched test: LSD (P<0.05)

(I)Group	(J) Group	Mean Difference (I-J)	Std. Error	Sig.	95% Confidence Interval	
					Lower Bound	Upper Bound
Bestaloy	Bisfil Core	-4.97E-02	.066	.456	-.1821	8.262E-02
	Core Max Ⅱ	.2553*	.066	.000	.1230	.3877
	Core Paste	.2290*	.066	.001	9.667E-02	.3614
	Ti-Core	.5790*	.066	.000	.4467	.7114
	Ti-Core Natural	.1719*	.066	.012	3.956E-02	.3043
	Vitremer	.7102*	.066	.000	.5779	.8425
	Ketac Molar	1.0559*	.066	.000	.9236	1.1883
Bisfil Core	Amalgam	4.973E-02	.066	.456	-8.26E-02	.1821
	Core Max Ⅱ	.3051*	.066	.000	.1727	.4374
	Core Paste	.2787*	.066	.000	.1464	.4111
	Ti-Core	.6288*	.066	.000	.4964	.7611
	Ti-Core Natural	.2216*	.066	.001	8.929E-02	.3540
	Vitremer	.7599*	.066	.000	.6276	.8923
	Ketac Molar	1.1057*	.066	.000	.9733	1.2380
Core Max Ⅱ	Amalgam	-.2553*	.066	.000	-.3877	-.1230
	Bisfil Core	-.3051*	.066	.000	-.4374	-.1727
	Core Paste	-2.63E-02	.066	.693	-.1587	.1060
	Ti-Core	.3237*	.066	.000	.1913	.4560
	Ti-Core Natural	-8.34E-02	.066	.213	-.2158	4.892E-02
	Vitremer	.4549*	.066	.000	.3255	.5872
	Ketac Molar	.8086*	.066	.000	.6682	.9329
Core Paste	Amalgam	-.2290*	.066	.001	-.3614	-9.67E-02
	Bisfil Core	-.2787*	.066	.000	-.4111	-.1464
	Core Max Ⅱ	2.632E-02	.066	.693	-.1060	.1587
	Ti-Core	.3500*	.066	.000	.2177	.4824
	Ti-Core Natural	-5.71E-02	.066	.393	-.1895	7.524E-02
	Vitremer	.4812*	.066	.000	.3488	.6135
	Ketac Molar	.8269*	.066	.000	.6946	.9593
Ti-Core	Amalgam	-.5790*	.066	.000	-.7114	-.4467
	Bisfil Core	-.6288*	.066	.000	-.7611	-.4964
	Core Max Ⅱ	-.3237*	.066	.000	-.4560	-.1913
	Core Paste	-.3500*	.066	.000	-.4824	-.2177
	Ti-Core Natural	-.4071*	.066	.000	-.5395	-.2748
	Vitremer	.1312	.066	.052	-1.18E-03	.2645
	Ketac Molar	.4769*	.066	.000	.3446	.6092
Ti-Core Natural	Amalgam	-.1719*	.066	.012	-.3043	-3.96E-02
	Bisfil Core	-.2216*	.066	.001	-.3540	-8.93E-02
	Core Max Ⅱ	8.343E-02	.066	.213	-4.89E-02	.2158
	Core Paste	5.711E-02	.066	.393	-7.52E-02	.1895
	Ti-Core	.4071*	.066	.000	.2748	.5395
	Vitremer	.5388*	.066	.000	.4059	.6706
	Ketac Molar	.8840*	.066	.000	.7517	1.0164
Vitremer	Amalgam	-.7102*	.066	.000	-.8425	-.5779
	Bisfil Core	-.7599*	.066	.000	-.8923	-.6276
	Core Max Ⅱ	-.4549*	.066	.000	-.5872	-.3225
	Core Paste	-.4812*	.066	.000	-.6135	-.3448
	Ti-Core	-.1312	.066	.052	-.2635	1.177E-03
	Ti-Core Natural	-.5383*	.066	.000	-.6706	-.4059
	Ketac Molar	.3457*	.066	.000	.2134	.4781
Ketac Molar	Amalgam	-1.0559*	.066	.000	-1.1883	-.9236
	Bisfil Core	-1.1057*	.066	.000	-1.2380	-.9733
	Core Max Ⅱ	-.8006*	.066	.000	-.9329	-.6682
	Core Paste	-.8269*	.066	.000	-.9593	-.6946
	Ti-Core	-.4769*	.066	.000	-.6092	-.3446
	Ti-Core Natural	-.8840*	.066	.000	-1.0164	-.7517
	Vitremer	-.3457*	.066	.000	-.4781	-.2134

* : The mean difference is significant at the .05 level.

Table Ⅶ. Multiple range test of compact tension test: LSD (P<0.05)

(I)Group	(J) Group	Mean Difference (I-J)	Std. Error	Sig.	95% Confidence Interval	
					Lower Bound	Upper Bound
Bestaloy	Bisfil Core	-1.39E-02	.069	.840	-.1511	.1232
	Core Max Ⅱ	.2318*	.069	.001	9.464E-02	.3689
	Core Paste	.1407*	.069	.045	3.542E-02	.2778
	Ti-Core	.4128*	.069	.000	.2756	.5499
	Ti-Core Natural	.1995*	.069	.005	6.235E-02	.3366
	Vitremer	.6579*	.069	.000	.5208	.7950
	Ketac Molar	.8529*	.069	.000	.7158	.9901
Bisfil Core	Amalgam	1.394E-02	.069	.840	-.1232	.1511
	Core Max Ⅱ	.2457*	.069	.001	.1086	.3829
	Core Paste	.1546*	.069	.028	1.748E-02	.2918
	Ti-Core	.4267*	.069	.000	.2896	.5639
	Ti-Core Natural	.2134*	.069	.003	7.629E-02	.3506
	Vitremer	.6719*	.069	.000	.5347	.8090
	Ketac Molar	.8669*	.069	.000	.7297	1.0040
Core Max II	Amalgam	-.2318*	.069	.001	-.3689	-9.46E-02
	Bisfil Core	-.2457*	.069	.001	-.3829	-.1086
	Core Paste	-9.11E-02	.069	.190	-.2282	4.604E-02
	Ti-Core	.1810*	.069	.010	4.386E-02	.3181
	Ti-Core Natural	-3.23E-02	.069	.640	-.1694	.1048
	Vitremer	.4261*	.069	.000	.2890	.5633
	Ketac Molar	.6212*	.069	.000	.4840	.7583
Core Paste	Amalgam	-.1407*	.069	.045	-.2778	-3.54E-02
	Bisfil Core	-.1546*	.069	.028	-.2918	-1.75E-02
	Core Max Ⅱ	9.110E-02	.069	.190	-4.60E-02	.2282
	Ti-Core	.2721*	.069	.000	.1350	.4092
	Ti-Core Natural	5.881E-02	.069	.395	-7.83E-02	.1959
	Vitremer	.5172*	.069	.000	.3801	.6544
	Ketac Molar	.7123*	.069	.000	.5751	.8944
Ti-Core	Amalgam	-.4128*	.069	.000	-.5499	-.2756
	Bisfi Core	-.4267*	.069	.000	-5.639	-.2896
	Core Max Ⅱ	-.1810*	.069	.010	-.3181	-4.39E-02
	Core Paste	-.2721*	.069	.000	-.4092	-.1350
	Ti-Core Natural	-.2133*	.069	.003	-.3504	-7.62E-02
	Vitremer	.2451*	.069	.001	.1080	.3823
	Ketac Molar	.4401*	.069	.000	.3030	.5773
Ti-Core Natural	Amalgam	-.1995*	.069	.005	-.3366	-6.24E-02
	Bisfil Core	-.2134*	.069	.003	-.3506	-7.63E-02
	Core Max Ⅱ	3.229E-02	.069	.640	-.1048	.1694
	Core Paste	-5.88E-02	.069	.395	-.1959	7.833E-02
	Ti-Core	.2133*	.069	.003	7.615E-02	.3504
	Vitremer	.4584*	.069	.000	.3213	.5956
	Ketac Molar	.6534*	.069	.000	.5163	.7906
Vitremer	Amalgam	-.6579*	.069	.000	-.7950	-.5208
	Bisfil Core	-.6719*	.069	.000	-.8090	-.5347
	Core Max Ⅱ	-.4261*	.069	.000	-.5633	-.2890
	Core Paste	-.5172*	.069	.000	-.6544	-.3801
	Ti-Core	-.3451*	.069	.001	-.3823	-.1080
	Ti-Core Natural	-.4584*	.069	.000	-.5956	-.3213
	Ketac Molar	.1950*	.069	.006	5.788E-02	.3322
Ketac Molar	Amalgam	-.8529*	.069	.000	-.9901	-.7158
	Bisfil Core	-.8669*	.069	.000	-1.0040	-.7297
	Core Max Ⅱ	-.6212*	.069	.000	-.7583	-.4840
	Core Paste	-.7123*	.069	.000	-.8494	-.5751
	Ti-Core	-.4401*	.069	.000	-.5773	-.3030
	Ti-Core Natural	-.6534*	.069	.000	-.7906	-.5163
	Vitremer	-.1950*	.069	.006	-.3322	-5.79E-02

* : The mean difference is significant at the .05 level.

Core Natural, and Core Max Ⅱ ($p>0.05$). These three materials had significantly lower fracture toughness than Bisfil Core and amalgam($p<0.05$).

The One-Way ANOVA for compact tension test (Table Ⅴ) also indicated significant differences among several of the core materials. The LSD test (Table Ⅶ) revealed the similar result to that of single-edge notched test without the significantly different fracture toughness of Ti-Core and Vitremer($p<0.05$).

The fracture toughness value generated by the single-edge notched test were higher than the value generated by the compact tension test, except for the Ketac Molar.

Fractography analysis

The fracture surface of Bisfil Core (Fig. 9) was rougher than those of other core materials. Many filler particles with sharp edges were found in the fracture surface. For Core Max Ⅱ (Fig. 10) and Core Paste(Fig. 11), the fracture surfaces were smoother than that of Bisfil Core, and some porosities were observed. The filler particle size of Core Max Ⅱ was smaller than that of other composite resin core materials. In addition, smooth surfaces due to debonding between filler and matrix were also observed. In case of Ti Core Natural(Fig. 13), the size of the filler particle was smaller than that of Bisfil Core, and the filler volume fraction was larger than that of Bisfil Core. The fillers were angular shape, and a few filler particles were coated with matrix resin. In the fracture surface of Ti Core(Fig. 12), most of the particles of angular shape which may be the titanium particles were seen to be debonded from the matrix. In the fracture surface of Vitremer(Fig. 14) and Ketac Molar(Fig. 15), cracks formed by desiccation on the surfaces exposed to air were typical. Extensive porosity was also a feature of this material. The sizes of the particles were small, and the particles were debonded from the matrix.

Atomic Force Microscope (AFM)

In the AFM scan, each material exhibited a different surface morphology. In the case of amalgam(Fig. 16), the triangular shaped particles were of regular size, and a number of particles were observed. The surface particles of Bisfil Core (Fig. 17) were very small and large in number, and the surface roughness relatively low. In the case of Core Max Ⅱ (Fig. 18), the number of small particles were lesser than the other composite materials and the surface roughness was also low. The particles of Core Paste(Fig. 19) were relatively small and regularly arranged, although the particles were somewhat larger than those of Bisfil Core and Core Max Ⅱ. In the case of Ti-Core(Fig. 20) and Ti-Core Natural(Fig. 21), although the arrangement of the small particles were similar to those of the other composite resins, a few large size particles were also observed. Finally, on the surface of Vitremer(Fig. 22) and Ketac Molar(Fig. 23), the particles were irregularly shaped and larger in size, and the surface roughness was higher than those of the composite resins and the amalgam.

DISCUSSION

Most of the commonly used techniques of toughness measurements were developed to test metals, but they can be applied, with a few modifications, to brittle materials in general. According to Fujishima and Ferracane,[15] the double torsion test which loads a notched and grooved plate in four-point bending was the most difficult to conduct successfully due to the more demanding requirements of specimen preparation and specimen alignment during testing. In their study, a few of the short rod tests which loads in tension a cylinder containing a chevron-shaped notch were also unsuccessful because the fracture plane deviated substantially from the plane of the notch, however, no such problems were encountered in the single-edge notched or compact tension experi-

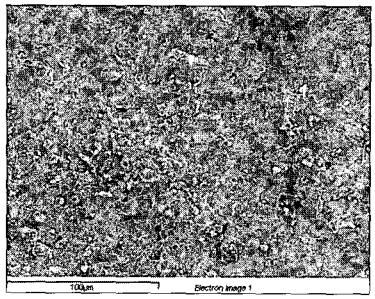

Fig. 8. Fracture surface of Amalgam. SEM ×500.

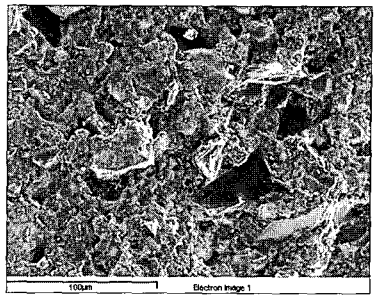

Fig. 9. Fracture surface of Bisfil Core. SEM ×500.

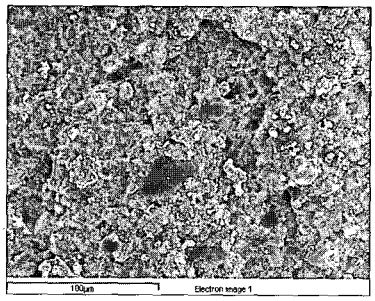

Fig. 10. Fracture surface of Core Max Ⅱ. SEM ×500.

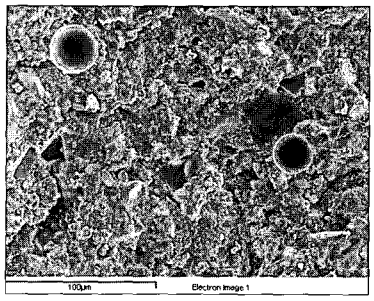

Fig. 11. Fracture surface of Core Paste. SEM ×500.

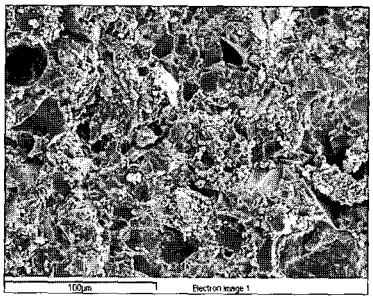

Fig. 12. Fracture surface of Ti-Core. SEM ×500.

Fig. 13. Fracture surface of Ti-Core Natural. SEM ×500.

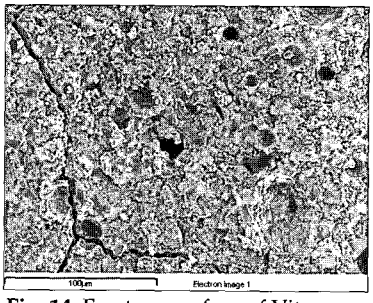

Fig. 14. Fracture surface of Vitremer. SEM ×500.

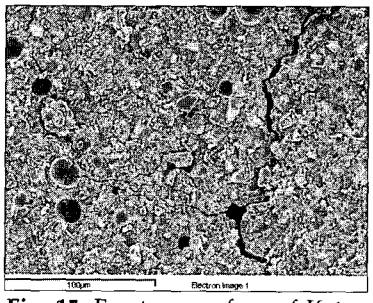

Fig. 15. Fracture surface of Ketac Molar. SEM ×500.

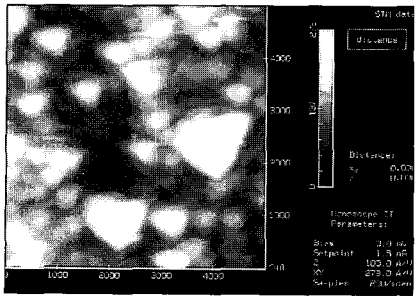

Fig. 16. AFM scan of amalgam.

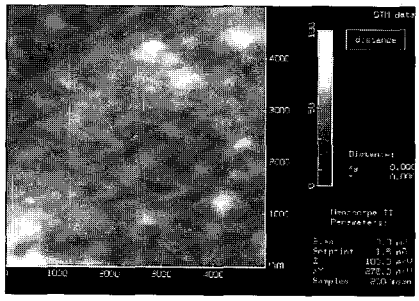

Fig. 17. AFM scan of Bisfil Core.

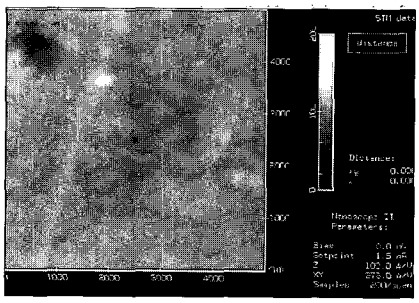

Fig. 18. AFM scan of Core Max Ⅱ.

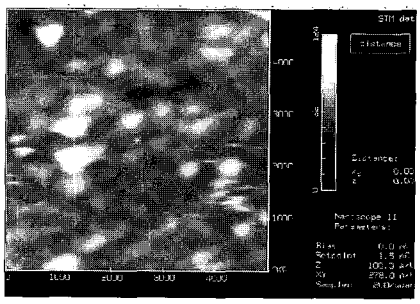

Fig 19. AFM scan of Core Paste.

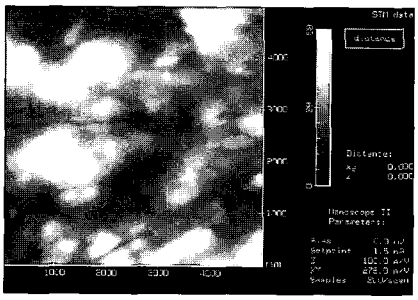

Fig. 20. AFM scan of Ti-Core.

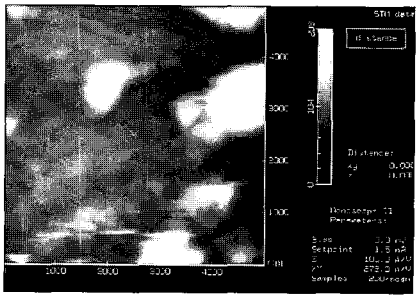

Fig. 21. AFM scan of Ti-Core Natural.

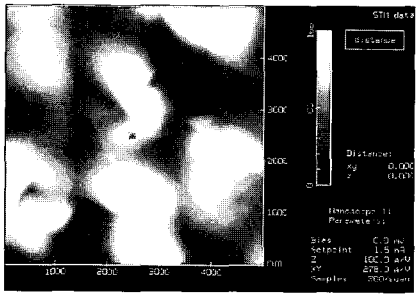

Fig. 22. AFM scan of Vitremer.

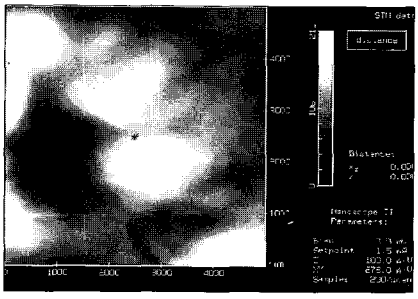

Fig. 23. AFM scan of Ketac Molar.

ment. So, in this study, the test methods included the single-edge notched design which loads a notched bar in three point bending and the compact tension design which loads a notched plate in uniaxial tension.

In order to obtain valid and reproducible fracture toughness parameters, it is essential that a fracture toughness test satisfies three requirements: 1) The specimen geometry must be such that K_{IC} can be estimated with sufficient accuracy, 2) The values of the load and the crack length at the onset of cracking must be measured accurately, and 3) Pre-cracking must ensure that the crack introduced is a sharp one.[22]

The effect of the specimen size is important because fracture mechanics theory is based on assumption of specimen geometry. To obtain a true measure of the fracture toughness, it is essential that the testing method meet the criteria for plane strain conditions. To satisfy plane strain conditions, the thickness of the specimen would have to be infinitely thick.[27] However, when a restorative material is to be evaluated, it is important to use test specimens of a similar size to those used in clinical situation. The main reason for this is so that the setting reaction will occur under similar conditions to those in the mouth. Furthermore, for light-cured products, where the position and duration of irradiation is critical, realistically sized specimens are essential.[54] The research design of the compact tension study followed the recommendations of Kovarik et al,[27] who studied the effects of specimen geometry on the measurement of fracture toughness, and recommended specimen thickness of 1.7mm. The specimen geometry of single-edge notched test was in accordance with the study of Johnson et al[19] and Ziebert et al.[56]

The standard for measuring fracture toughness require the preexisting crack within the material to be a crack of molecular dimensions like that assumed by Griffith.[11] Such a flaw may be produced in vitro by fatigue, e.g., during chewing. It was believed that the most accurate evaluation of fracture toughness for dental materials would be achieved by testing specimens with an extremely sharp flaw, i.e., one made by propagating a crack from a sharp notch. However, these cracks are very difficult to introduce into specimens without causing catastrophic failure. As a result, investigators have used a variety of alternative methods for precracking the specimen. This has been by sawing a notch in the sample using a Bard Parker blade under hand pressure,[16,47,48] or curing the material around the sharp edge of a razor blade.[32,34,36] Other investigators have run the test without any attempt to form a crack or flaw at the tip of the notch. Previously, Ferracane et al.[11] ascertained that there was no difference between fracture toughness measured on precracked specimens and that determined from notches made with a sharp razor blade. According to the study of Kovarik and Fairhurst,[29] the measured fracture toughness of the group with a crack formed around a razor edge during polymerization was significantly lower than the precrack carved using a Bard Parker blade under hand pressure and similar to the result of the stress-induced crack. Therefore, in this experiment, the notch tip was produced by a sharp stainless steel blade inserted into the mold.

Toughening of a resin composite system is possible by crack pinning, crack branching by use of a large filler, and plastic deformation of the matrix around the filler.[9,55] In addition to the above mentioned toughening mechanism, for high filler contents, numerous microcracks reduces the stress concentration at the crack tip and increases the fracture toughness. This phenomenon is called a microcrack-induced toughening effect.[9,13] When these microcracks are distributed broadly around the main crack, the crack tip extends by deflection from one direction to another and coalesces with microcracks, resulting in a very

rough fracture surface. This phenomenon is called crack-deflection-induced toughening effect.[10,42]

Among the experimental core materials, Bisfil Core, a light-cure composite exhibited the highest fracture toughness value. Bisfil Core comes with its own dentin bonding system, which may reduce microleakage. Bisfil Core's distinctive blue color is designed to aid in distinguishing the material from natural tooth structure and is recommended for metal-based, fixed restorations.

Ti-Core and Ti-Core Natural are patented, titanium and lanthanide metal reinforced, fluoride releasing, self curing composite core build up materials. Ti-Core's grey color is easy to distinguish from tooth structure, and Ti-Core Natural is available in natural shade Vita A3. These materials are Bis-GMA based hybrid resin composites that provide compressive and tensile strength comparable to dentin according to the manufacturer.[6] However, in this study, the fracture toughness value of Ti-Core was significantly lower than those of the other composite resins. Although the reason is not clear, but may be explained by the bonding between titanium used for reinforcement and resin matrix, because Ti-Core Natural, the analogous material which is reinforced with lanthanide mixture, exhibited similar fracture toughness value with other composite materials.

Glass ionomer cements are still popular as buildup materials because they are easy to manipulate, bond to tooth structure, and release fluoride in vitro.[39] However, Ketac Molar had the lowest mean fracture toughness in this study. The use of Ketac Molar may be further restricted by an unattractive feature observed during this investigation: extensive cracking upon drying, which is more clearly visible on the surfaces of recently set material. Such cracks could be flaws from which a fracture initiates.

The most recent and significant change in glass ionomer formulation has been the development of resin modified light-cured glass ionomers.[30] Manufacturers have developed the capability to attach mehylmethacrylate groups on polyacrylic acid chains used in glass ionomers. This unique chemistry allows photo-initiation of the cross-linking of methacrylate groups forming the same covalent bonds as developed in resins, while at the same time allowing the cross linking of carboxylate groups via multivalent cations. These materials are reported by manufacturers to have greatly increased strength and fracture resistance over traditional glass ionomers while still possessing the advantages of fluoride release.[30] Vitremer is a resin-modified glass ionomer system that can be light cured or self cured and displays long term fluoride release and adhesion to tooth structure.

The tooth-colored shade is one of the important requirement in the area where all ceramic restoration is indicated. Among the materials used in this study, Ketac Molar, Vitremer, and Ti-Core Natural are a tooth-colored shade. According to the result of this study, Ti-Core Natural is the recommended core material for all ceramic restoration.

SEM analysis of the fracture surfaces has characterized the initiation site and propagation features that occurred during the fracturing process of various dental materials.[28] A fracture surface represented the result of the material's resistance to fracture, and materials having high fracture toughness showed a rougher fracture surface.

For glass ionomer material, filler debonding without any crack inhibiting process was related to materials with low K_{IC} values. Numerous cracks occurred through the matrix, a characteristic feature of the glass ionomer materials.[41]

The main mode of crack propagation in all composites investigated was through the matrix and along the interface of the filler and the matrix.[11,34,41] In another report,[34] however, distinction of the filler and matrix was often times dif-

ficult because of a layer of resin adhering to the filler. A resin coating on the particles would suggest that the adhesion between the filler and matrix was stronger than the actual resin matrix itself. This implies that the cracking process has been mainly in the matrix at short distances from the filler particles. Although crack propagation in composite resin occurs mainly through the matrix, occasional cleavage or debonding of the larger filler particles occur in the composite as well. For each composite system, filler/matrix dedonding was more apparent for the larger particles in the system, implying that the stress concentration around these particles was greater than that around the smaller particles. In particulate composite material, the crack growth rate depends not only on the structure of the matrix but on the strength of the interfacial bond, the strength of the particles, and on the volume fraction of these particles.[41] As a result of this study, the composite resin having large filler particle size and high filler volume fraction showed high fracture toughness, except Ti-Core.

For high copper amalgam(Fig. 8), the presence of an oxide or other contaminant on the particle surfaces would hinder the amalgamation with mercury to create isolated surfaces of weakness along which cracks can propagate preferentially at a lower energy requirement. Weak interfaces created by contaminants on alloy particle surfaces are most likely to be the inherent flaws by angular contraction porosity.[36]

Atomic force microscopy was used to analyze the surface morphology of core materials. Generally, composite resin having high fracture toughness showed lower surface roughness than glass ionomer having low fracture toughness. In the Bis-GMA based composite resins, although the size and number of the particles were different with one another, for the most part, relatively small filler particles were regularly arranged, and the surface roughness was low. Ketac Molar and Vitremer had wider particle surfaces and deeper dents. And it seems to be related to the crack initiation along the interface of the particle and the matrix. The regular packing of triangular shaped particles were found in amalgam, and it seems that the arrangement and the shape of particles were related to the high fracture toughness of amalgam.

The fracture value generated by the single-edge notched test were higher than the value generated by the compact tension test, except for Ketac Molar. It is possible that the propagation of a crack from the single-edge notched specimen is more sensitive to the sharpness of the crack tip than that for the compact tension test.[15]

As a result of the statistical analysis, the compact tension test had higher sensitivity than the single-edge notched test, because there was significant difference between the fracture toughness of Ti-Core and Vitremer in the compact tension test while there was no significant difference between the value of both materials in single-edge notched test.

In any case, different test methods were in general agreement concerning the relative fracture toughness of the eight materials. This suggests that both test methods can be used to provide a reasonable comparison of the fracture toughness of a group of materials, even if the absolute values may differ.

Based on the results of this study, amalgam and composite resins except for Ti-Core seem to be appropriate for core build-up. Fracture toughness of the newly developed core build-up materials should be evaluated routinely so that the clinician determine whether the material will be able to withstand the occlusal force, while resisting fracture in the presence of surface flaws. In addition, standard test conditions, such as specimen type, crosshead speed, and storage conditions, must also be established so that data from different studies may be compared and analyzed.

CONCLUSION

The single-edge notched test and compact tension test were performed to evaluate the fracture toughness of currently used eight core materials, and the fracture surface of each material was observed under SEM. And, the surface morphology of each material was analyzed with the AFM.

Within the limits of this study, the following conclusions were drawn;

1. Bisfil Core showed the highest mean fracture toughness regardless of test methods.
2. For the tooth-colored materials, Ti-Core Natural exhibited the highest fracture toughness.
3. Ketac Molar showed a significantly low fracture toughness when compared with the amalgam and the composite resin core materials($p<0.05$).
4. The fracture toughness values obtained with the single-edge notched test, except Ketac Molar, were higher than those obtained in the compact tension test.
5. SEM revealed that the fracture surface of high fracture toughness material was rougher than that of low fracture toughness material.
6. AFM revealed that the surface particles of the composite resins were smaller in size, with a lower surface roughness than the glass ionomer core materials.

REFERENCES

1. ASTM Standard E-399-83. Standard test method for plane-strain fracture toughness of metallic materials. In:1992 Annual book of ASTM standards. Philadelphia: ASTM; 1992. p. 676-706.
2. Bonilla ED, Mardirossian G, Caputo AA. Fracture toughness of various core build-up materials. J Prosthod 2000;9:14-8.
3. Cook WD, Johnson M. The influence of postcuring on the fracture properties of photo-cured dimethacrylate based dental composite resin. J Biomed Mater 1987;21:979-89.
4. Davis DM, Waters NE. An investigation into the fracture behavior of a particulate-filled BisGMA resin. J Dent Res 1987;66:1128-33.
5. Davis DM, Waters NE. Fractography of a bis-GMA resin. J Dent Res 1989;68:1194-8.
6. Deutsch AS, Cohen BI, Condos S, Musikant BL. Compressive and diametral tensile strength of titanium reinforced composites (Abstract 246). J Dent Res 1992;71:136.
7. Dieter GE. Mechanical Metallurgy. 2nd Ed. New York:McGraw-Hill, 1976, 247-91.
8. Edwald HL, Wanhill RJH. Fracture mechanics. Edwald Arnold Co., London.(1989)
9. Evans AG, Faber KT. Crack-growth resistance of microcracking brittle materials. J Am Ceram Soc 1984;67:255-60.
10. Faber KT, Evans AG. Crack deflection processes. Acta Metall 1983;31:565-76.
11. Ferracane JL, Antonio RC, Matsumoto H. Variables affecting the fracture toughness of dental composites. J Dent Res 1987;66:1140-5.
12. Ferracane JL. Indentation fracture toughness testing of and crack propagation mode in dental composites. Mater Res Symp Proc 1989;110:619-24.
13. Fu Y, Evans AG. Some effects of microcracks on the mechanical properties of brittle solid. Acta Metall 1985;33:1515-23.
14. Fujishima A, Miyazaki T, Aoyama M, Suzuki E, Miyaji T. Fracture toughness of composite resin inlay. J Jpn Dent Mater 1991;10:137-45.
15. Fujishima A, Ferracane JL. Comparison of four modes of fracture toughness testing for dental composites. Dent Mater 1996;12:38-43.
16. Gegauff AG, Pryor HG. Fracture toughness of provisional resins for fixed prosthodontics. J Prosthet Dent 1987;58:23-9
17. Goldman M. Fracture properties of composite and glass-ionomer dental restorative materials. J Biomed Mater Res 1985;19:771-83.
18. Griffith AA. The phenomena of rupture and flow in solids. Philos Trans R Soc 1920;221A:163-198.
19. Hashemi S, Williams JG. Crack sharpness effects in fracture testing of polymers. J Mater Sci 1985;20:922-8
20. Hertzberg RW. Deformation and fracture mechanics of engineering materials. John Wiley and Sons Inc., London. (1976)
21. Higo Y, Damri D, Nunomura T. The fracture toughness characteristics of three dental composite resins. Biomed Mater Eng 1991;1:223-31.
22. Jayatilka AS. Fracture toughness engineering brittle materials. Applied Science Publishers Ltd., London. (1979)
23. Johnson WW, Dhuru VB, Brantley WA. Composite microfiller content and its effect on fracture toughness and diametral tensile strength. Dent Mater 1993;9:95-8.
24. Kim KH, Park JH, Imai Y, Kishi T. Microfracture mechanics of dental resin composites containing spherically-shaped filler particles. J Dent Res

1994;73:499-504
25. Knobloch LA, Kerby RE, Seghi R. Fracture toughness of resin-based luting cements. J Prosthet Dent 2000;83:204-9.
26. Knott JF. Fundamentals of fracture mechanics. Butteworths, London. (1973)
27. Kovarik RE, Ergle JW, Fairhurst CW. Effects of specimen geometry on the measurement of fracture toughness. Dent Mater 1991;7:166-9.
28. Kovarik RE, Ergle JW, Fairhurst CW. A comparison of methods used to measure fracture toughness of resin composites. J Dent Res 1991;70:345 (Abstract No. 637).
29. Kovarik RE, Fairhurst CW. Effect of Griffith precracks on measurement of composite fracture toughness. Dent Mater 1993;9:222-8.
30. Kovarik RE, Muncy MV. Fracture toughness of resin-modified glass ionomers. Am J Dent 1995;8:145-8.
31. Lange FF, Radford KC. Fracture energy of epoxy composite system. J Mater Sci 1971;6:1197-1203.
32. Lloyd CH, Iannetta RV. The fracture toughness of dental composites. I. The development of strength and fracture toughness. J Oral Rehabil 1982;9:55-66.
33. Lloyd CH. The fracture toughness of dental composites. II. The environmental and temperature dependence of the stress intensification factor (KIC). J Oral Rehabil 1982;9:133-8.
34. Lloyd CH, Mitchell L. The fracture toughness of tooth coloured restorative materials. J Oral Rehab 1984;11:257-72.
35. Lloyd CH, Adamson M. Fracture toughness of amalgam. J Oral Rehabil 1985;12:59-68.
36. Lloyd CH, Adamson M. The development of fracture toughness and fracture strength in posterior restorative materials. Dent Mater 1987;3:225-31.
37. Lloyd CH, Butchart DGM. The retention of core composites, glass ionomers and cermets by a self-threading dentin pin: The influence of fracture toughness upon failure. Dent Mater 1990;6: 185-188.
38. Mair LH, Vowles R. The effect of thermal cycling on the fracture toughness of seven composite restorative materials. Dent Mater 1989;5:23-6.
39. McLean JW. Glass ionomer cements. Br Dent J 1988;164:293-300.
40. Montes-G GM, Draughn RA. Slow crack propagation on composite restorative materials. J Biomed Mater Res 1987;21:629-642.
41. Mueller HJ. Fracture toughness and fractography of dental cements, lining, build-up, and filling materials. Scanning Microscopy 1990;4:297-307.
42. Pezzotti G, Tanaka I, Okamoto T. Si3N4/SiC-whisker composite without sintering aids. J Am Ceram Soc 1990;73:3039-45.
43. Pilliar RM, Smith DG, Maric B. Fracture toughness of dental composites determined using the short-rod fracture toughness test. J Dent Res 1986;65:1308-14.
44. Pilliar RM, Vowles R, Williams DF. The effect of environmental aging on the fracture toughness of dental composites. J Dent Res 1987;66:722-6.
45. Roberts JC, Powers JM, Craig RG. Fracture toughness of composite and unfilled restorative resins. J Dent Res 1977;56:748-53.
46. Roberts JC, Powers JM, Craig RG. The fracture toughness and critical strain release rate of dental amalgam. J Mater SCI 1978;13:965-71.
47. Sands MP, Morena R, Fairhurst CW. Fracture toughness of posterior composite resins. J Dent Res 1987;66:183, Abstr. No. 613.
48. Sands MP, Morena R, Fairhurst CW. Fracture toughness of posterior composite resins. J Dent Res 1988;67:220, Abstr. No. 857.
49. Sawa N. The effect of filler shape and size of resin composite on fracture toughness. J Jpn Conserv Dent 1993;36:507-18.
50. Spanoudakis J, Young R. Crack propagation in a glass particle-filled epoxy resin, Part I: Effect of particle volume fraction and size. J Mater Sci 1984;19:473-86.
51. Takeshige F. Fatigue crack propagation in composite resins. J Jpn Conserv Dent 1994;37:31-49.
52. Truong VT, Tyas MJ. Prediction of in vivo wear in posterior composite resins: a fracture mechanics approach. Dent Mater 1988;4:318-27.
53. Uctasli S, Wilson HG, Zaimoglu L. Variables affecting the fracture toughness of resin-based inlay/onlay systems. J Oral Rehab 1993;20:423-31.
54. Uctasli S, Harrington E, Wilson HJ. The fracture resistance of dental materials. J Oral Rehabil 1995;22:877-86.
55. Young RJE, Beaumont PWR. Failure of brittle polymers by slow crack growth. J Mater Sci 1975;10:1343-59.
56. Ziebert AJ, Dhuru VB. The fracture toughness of various core materials. J Prosthod 1995;4:33-7.

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