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치과용 복합레진의 기계적 성질과 AE특성

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MECHANICAL PROPERTIES AND ACOUSTIC EMISSION CHARACTERISTICS OF DENTAL COMPOSITE RESINS

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ABSTRACT

Bending strength ,bending elastic modulus and fracture toughness of different types of dental composite resins were determined . The relationship between bending strength ,fracture toughness and filler volume fraction of dental composite resin was understood. In Acoustic Emission(AE) behavior during fracture toughness test, characteristic generation patterns of each type were understood. The fracture toughness values ,AE generation patterns, and the nature of fracture surface were analyzed to understand fracture behavior of dental composite resin.

INTRODUCTION

Dental composite resin was developed for use of restorations on anterior teeth with the advantage of its close color match with natural teeth and ease of manipulation. There have been many experimental and clinical reports on the surface hardness , compressive strength, wear-resistance of dental composite resins [1-4]. Recently, newly developed resin matrix and filler surface treatment technique increase the mechanical properties of composite resins ,but low resistance to marginal fracture and wear still remains in an unsolved problem. Composite resins are inherently brittle and apt to produce microscopic surface flaws on surface. Therefore, short service life caused by these flaws is a particularly severe problem for these materials. There are some reports on the facts that the service life of dental composite mainly depends on surface wear phenomena and those failure process have significant relationship with the fracture toughness. Thus, wear resistance and fracture toughness are two main parameters that control the life of a dental composite.

In this study, bending strength ,bending elastic modulus and fracture toughness of three types of commercially available dental composite resin were obtained. The relation of fracture toughness and bending strength to filler fraction was made. Beside fracture surface finding, AE generation pattern during fracture toughness test was used to understand the fracture behavior of dental composite resin.

TEST MATERIALS AND METHODS

Test Materials and specimen preparation

Tested commercially available dental composite resins are listed in Table 1. The material includes each three kind of macrofilled type, microfilled type and hybrid type.

Table 1.Dental composite resins examined

Macrofilled Type	Hybrid Type	Microfilled Type
Clearfil F-Ⅱ	Bis-fil	Heliomolar
Hi-pol	P-50	Durafil
Concise	Graft	Silux-plus

A macrofilled type is generally contains the filler particles greater than 10 μm, but a microfilled type contains particles of submicrons. Especially in a microfilled type, prepolymerized resin particle sized 20-30 μm are added to increase the filler content.

Specimens for bending strength are bar-shaped ones with their length, width and thickness being 25mm, 2mm, 2mm respectively. Specimens for fracture toughness test are made in a split mold containing a razor blade which is fixed into a slit in the mold and their thickness(B)is 2.5mm and span, width are determined by thickness(W=2B,S=4W). The resulting notch/width ratio of specimen was maintained in a range of 0.45 to 0.55.

After packing the resin into the mold, the light cured types were illuminated in five 30 seconds illumination steps along its length for each side with tungsten halogen light illuminator. Chemically cured one was mixed to the manufacture's directions and packed and molded in a same manner as the light-cured type excepting for illumination. Fabricated specimens were tested after storing in distilled water at 37 ℃ for 24 hours.

Ten specimens for fracture toughness and five for bending specimens are tested ,and mean and standard deviation of toughness and strength value are obtained.

Experimental Procedure

The block diagram of testing procedure is shown in Fig. 1. The specimens were tested in three point bending. Bending strength and bending elastic modulus were calculated from the load-displacement curve. Fracture toughness, K_{IC}, was also calculated by peak load in load-time curve according to ASTM E-399.

In fracture toughness test, Physical Acoustic Corporation(PAC) 3000/3104 AE analyzer was used to detect released AE signals with two sensors attached on both ends of specimens. Sensors used were high-sensitivity, and low-noise type with which head-amp is equipped. Signals recorded by PAC system was analyzed as various parameters such as amplitude, events, energy etc..

The fracture surfaces were coated with thin film of Au in a vacuum evaporator and examined in the scanning electron microscope (Hitachi S-4300), so that the micro-fracture behaviors could be determined.

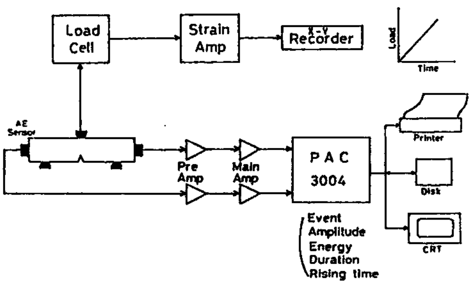

Fig.1 Block diagram of testing procedure.

RESULTS AND DISCUSSION

Fracture toughness and Bending strength values

Bending strength, bending elastic modulus and fracture toughness values obtained are listed in Table 2. Bending strength of dental composite resins ranges from 60 to 120 Mpa and elastic modulus from 2 to 7 Gpa. Fracture toughness of dental composite resin shows its values in the range of 0.8 to 1.5 Mpa$\sqrt{m}$. Macrofilled types shows the highest fracture toughness values of about 1.3 Mpa$\sqrt{m}$ and microfilled types have the lowest values of fracture resistance in range of 0.6 to 0.8 Mpa$\sqrt{m}$. Hybrid type shows the toughness values between the values of the two types.

Table 2. Bending strength ,bending elastic modulus and fracture toughness of dental composite resins.(Means and standard deviation)

Type	Brand	σ_B (MPa)	E_B (GPa)	K_{IC} (MPa$\sqrt{m}$)
Macro - filled	Clearfil F-II	83.14(3.91)	6.38(0.19)	1.42(0.08)
	Hi-pol	94.82(7.87)	7.15(0.16)	1.36(0.12)
	Concise	91.55(8.57)	4.68(0.19)	1.31(0.08)
Hybrid	Bisfil	88.61(7.22)	6.20(0.20)	1.31(0.05)
	P-50	111.40(3.16)	6.85(0.19)	1.03(0.06)
	Graft	120.69(6.36)	7.19(0.31)	1.60(0.08)
Micro - filled	Heliomolar	60.24(1.80)	2.04(0.03)	0.85(0.11)
	Durafil	65.43(6.22)	1.84(0.12)	0.65(0.04)
	Silux-plus	62.03(3.23)	4.14(0.15)	0.85(0.08)

The relation of fracture toughness and bending strength to filler volume fraction is shown in Fig. 2. Filler volume fraction of dental composite resins is distributed in the range of 40 to 70% from type to type, in where hybrid types show the highest filler content among three because of the higher packing ratio with the different sized fillers mixing. Where as, microfilled type and macrofilled type shows its volume fraction in about 40% and 60%,respectively.

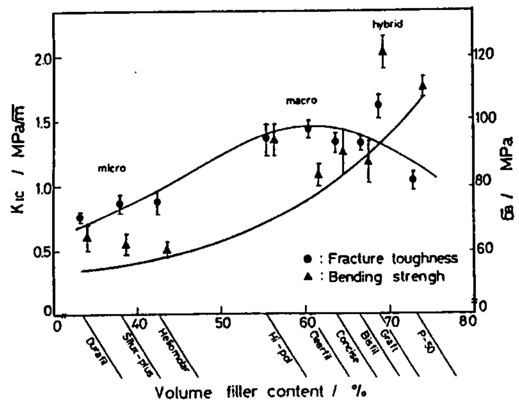

Fig. 2 The relation of bending strength and fracture toughness to filler volume fraction.

Bending strength increases with the filler fractions. Microfilled type, which has the lowest filler content and small particle size, shows the lowest values. By increasing the filler fractions and filler size, bending strength is increased. Hybrid types ,which have the highest filler content, show the highest values of about 2 times of microfilled types. Mechanical properties such as bending strength are increased by fraction of reinforced filler in dental composite resins.
On the other hand, fracture toughness has not the same tendency to bending strength. Hybrid type has the lower toughness value than that of macrofilled type in spite of higher filler fraction. That is, the value of K_{IC} reaches maximum to a certain filler fractions and begins to decrease. Before the fracture resistance reaches maximum, the increase of fracture toughness depends on the fraction of second phases.

Fractography

SEM fractographs of fracture surfaces of dental composite resin are shown in Fig. 3. Macrofilled type shows very rough surface(a). This means that large energy is required for crack to propagate. The delamination between matrix and fillers or the fracture of filler causes roughness to resulted surface. In hybrid types, less rough fracture surfaces than macrofilled type are observed(b).
The fracture surfaces, it can be recognized that cracks propagate through the interface between filler and matrix or

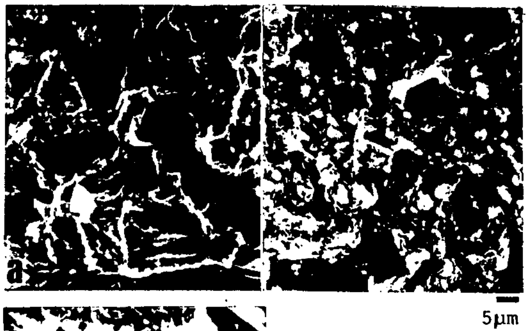

Fig. 3 Scanning electron micrograph of fracture surface of dental composite resins.
a)macrofilled type
b)hybrid type
c)microfilled type

matrix itself. Because filler size of hybrid type is smaller than that of macrofilled type, if crack extends through the interface between matrix and filler as same manner in two types, resulting surface of hybrid type is more flat than that of the macrofilled type.
Microfilled type shows some characteristic feature (c). Fracture of 20-30µm sized prepolymerized particles are observed. Roughness of microfilled types would seems apparently somewhat similar to that of macrofilled types. Prepolymerized resin blocks are relatively large and irregular in shape, which cause fracture surfaces to be rough. These particles can be seen either cleaved or protruded from the fracture surface. As a whole, microfilled types show low resistance to crack extension, because prepolymerized particles have almost no resistance to crack propagation.

Acoustic Emission Behavior

Result of Acoustic Emission(AE) release during fracture toughness test are shown in Fig. 4. There are load-time curves with AE events and amplitude distribution. In macrofilled type, AE is detected from the early stage at about 20% of maximum load. Rather small amplitude of AE events are released over the entire loading range. It is considered that this phenomena is related to the fracture of particles of 5-10 µm around the crack tip before crack extension.

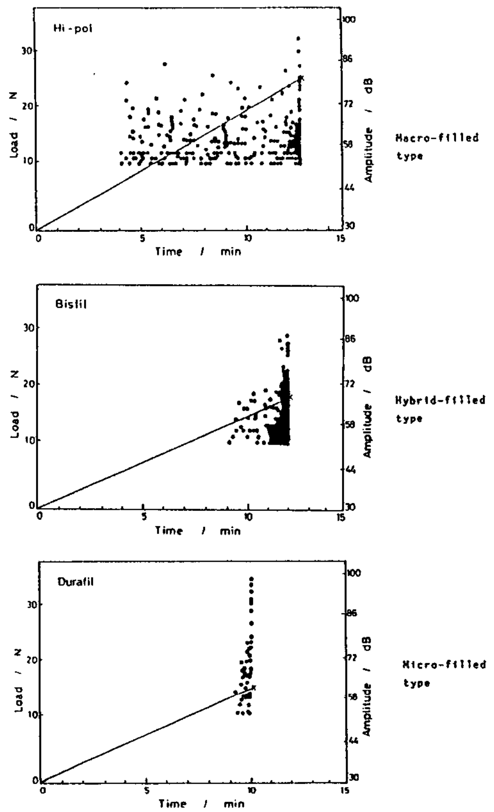

Fig. 4 AE release behavior of composite resins during fracture toughness test.

In microfilled type, AE is released to start at the maximum load and only small numbers of events are detected. But in this case the signals are somewhat large in amplitude, which is the result of the fracture of prepolymerized particles when main crack extends.

Fig. 5 shows the relation between fracture toughness and released AE event counts and filler contents of dental composite resins. As a whole, the fracture toughness values and released AE event counts show the same tendency,that is, the more AE events, the larger fracture toughness values. Main source of released AE in macrofilled type is thought to be the fracture of 5-10μm of fillers before crack extension , which enhance the fracture resistance of this type of composite resin and these fillers play an important role in fracture toughness of dental composites.

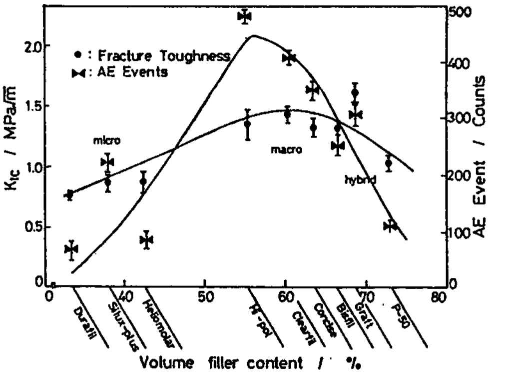

Fig .5 Relationship between AE event counts, fracture toughness values and volume fraction of dental composite resins.

CONCLUSIONS

Mechanical properties of dental composite resins are obtained. Bending strength of dental composite resins shows its values in the range of 60 to 120 Mpa and elastic modulus from 2 to 7 Gpa. Fracture toughness shows its values in the range of 0.8 to 1.5 Mpa√m. The bending strength and fracture toughness to filler fraction is become clear.

In acoustic emission behavior during fracture toughness test, characteristic generation patterns are confirmed on each type.

Fracture toughness and AE event show the same tendency,the more AE events, the larger fracture toughness.

From the fracture surface finding, AE behavior and measured fracture toughness values, fracture behavior of dental composite resin is understood.

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