

# The effect of cavity configuration on the mechanical properties of resin composites

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국문초록

## 중합환경에 따른 복합레진의 물리적 성질에 관한 연구

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이 연구의 목적은 와동의 형태가 굴곡강도와 탄성계수 등 복합레진의 물리적 성질에 미치는 영향을 평가하는 것이다. 복합레진은 Clearfil™ AP-X(Kuraray, Japan)와 Esthet-X™(Dentsply, USA)가 이용되었으며, 상아질 접착제는 Clearfil™ SE Bond(Kuraray, Japan)와 Prime & Bond NT™(Dentsply, USA)를 사용하였다. 대조군의 시편은 split steel mold(25mm×2mm×2mm) 내에 상기 2종류의 복합레진을 충전하여 2개의 대조군 시편을 제작하였으며, 2.4 및 3.4의 C-factor를 부여하기 위한 유리 모형와동을 제작하고, 와동 내에 상기 2종류의 복합레진을 충전하기 전 유리와동의 내면은 sandblasting 처리하고 각각의 복합레진과 동일회사 제품의 상기 상아질 접착제로 처리한 후, 복합레진을 각각 충전하여 4개의 실험군을 제작하였다. 제작된 실험군 시편은 저속 diamond saw로 충전된 복합레진 부위의 중심부를 통과하도록 절단하여 레진기둥(25mm×2mm×2mm)이 되도록 제작하였다. 제작된 시편을 37℃의 증류수에 24시간 동안 보관 후, 만능시험기(EZ Test, Shimadzu, Japan)를 이용하여 분당 1mm의 crosshead speed로 3점 굴곡강도를 측정하였다. 또 Linometer(R&B, Korea)를 이용하여 복합레진의 중합수축량을 측정하였으며, 굴곡강도 측정 후 시편의 파단면은 주사전자현미경(S-2300, Hitachi, Japan)을 이용하여 관찰하였다. 실험결과와 통계분석은 95% 수준의 one-way ANOVA/Tukey's test를 이용하여 결과를 얻었다. 실험에 이용된 2종류 복합레진의 굴곡강도와 탄성계수는 C-factor의 증가에 따라 감소하였으며, 파단면 또한 C-factor의 증가에 따라 더 불규칙해지는 양상을 나타내었다.

본 실험의 결과 hybrid형 복합레진이 micro-hybrid형 복합레진에 비해 C-factor의 영향을 더 많이 받는 것으로 나타났으며, 와동의 C-factor 증가가 굴곡강도나 탄성계수와 같은 복합레진의 물리적 성질을 저하시킨다는 것을 의미하였다.

## 1. INTRODUCTION

Although dental composites has been improved and widely used, there are still some undesirable characteristics, which affect their performance.

One of them is the shrinkage that inevitably occurs during polymerization as monomer molecules are converted into a polymer network, exchanging Van der Waals spaces for shorter covalent bond spaces. This shrinkage produces contraction stress in confined structures such as a tooth cavity. The majority of the

contraction stress of composite occurs during the initial polymerization period after gelation, and the stress development rate decreases gradually with time<sup>1)</sup>. Before gelation, most of contraction stresses caused by polymerization shrinkage relieved by flow capacity of the resin composite. The contraction stress in dental composite plays an important role in marginal adaptation<sup>2)</sup>. The internal stress generated in the restricted environment of a tooth cavity can exceed the adhesive bond strength and produce a delamination of the restoration interface<sup>3)</sup>. In cases

**Table 1.** Resin composites used in this study.

	Clearfil™ AP-X	Esthet-X™
Type of Materials	hybrid composite	micro-hybrid composite
Batch No.	0526A	530059
Manufacturer	Kuraray Co., Osaka, Japan	Dentsply Caulk, Milford, USA
Filler content	84.5 W%	77.0 W%
Mean particle size	barium glass	bariumaluminio fluorosilicate glass : 0.6~0.8 $\mu$ m (0.02~2.5 $\mu$ m)
	silicone dioxide : 3.0 $\mu$ m (0.1~15 $\mu$ m)	

where higher bond strength is present, this stress may fracture the marginal tooth substrate and/or the composite restoration itself<sup>4)</sup>. Either case results in the formation of a marginal gap, allowing for the possible ingress of oral fluids and bacteria through leakage. It is not easy for leakage around the cavity wall to be clinically detected immediately after placement.

However, the marginal leakage can be caused post-operative sensitivity, and may eventually produce discoloration of the margins and/or recurrent caries, and consequently may reduce the life of a restoration.

Due to the reasons mentioned above, throughout its lifetime, the interface between a dental resin and the restored tooth is subjected to the challenge of stresses, which can potentially cause de-bonding and lead to clinical failure of the restorative treatment. Before the restored tooth is subjected to functional load and thermal strains, an early interfacial stress build-up occurs during the polymerization in situ of resins bonded to tooth tissues. The setting reaction is, in fact, accompanied by volumetric shrinkage of varying magnitude, depending on the resin formulation<sup>5)</sup>. The magnitude of the polymerization contraction stress has been found to be dependent on the ratio of the bonded to free, unbonded surface area of the sample<sup>6,8)</sup>, on the material properties of the resin composite<sup>1,6,9,10)</sup> and on the amount of compliances of the substrate materials<sup>6,11,12)</sup>.

Among the material properties, which are thought to reduce early interfacial stress build-up is the ability to undergo plastic flow during the early phase of polymerization. Plastic flow allows part of the shrinkage to take place without stress build-up at the

interface and will determine the proportion of remaining shrinkage that manifests itself as determination rigid contraction<sup>7)</sup>. As a result of the rate-dependent character of visco-elastic behavior, slower rates of polymerization reaction have been shown to be associated with lower stress build-up and better interfacial integrity scores<sup>1,13-16)</sup>.

The elastic modulus of the shrinking material has also been found to be a factor on which shrinkage stresses depend. In vitro studies have shown that the interfacial stress during the setting shrinkage of a resin composite is positively correlated with the rigidity of the setting material<sup>17)</sup>. The elastic modulus also increases as the polymerization reaction proceeds<sup>18)</sup>.

Polymerization shrinkage and elastic modulus are in close relationship with the survival of the interfacial bond. However, so far, little has been done to establish how close that relationship is, starting with task of a systemic characterization of the relevant properties of resin composites.

In this aspect, the present study was designed to evaluate the effect of cavity configuration on the mechanical properties of resin composites by measuring flexural strength, elastic modulus, polymerization shrinkage and examination of the fractured surfaces of resin composites cured in the cavities with different configuration.

## II . MATERIALS AND METHODS

Two composite resins(Table 1) and their corresponding dentin bonding agents(Table 2) were used in this study. The 48 specimens of control group and two experimental groups of each resin composite

**Table 2.** Dentin bonding agents used in this study.

	Clearfil™ SE Bond	Prime & Bond NT™
Type of materials	self-etching system	self-priming system
Batch No.	primer : 00184A adhesive : 00175A	etching : 971202 adhesive : 98060000601
Manufacturer	Kuraray Co., Osaka, Japan	Dentsply Caulk, Milford, USA
Composition	primer : MDP, HEMA, water, initiator adhesive : MDP, HEMA, dimethacrylate, initiator, microfiller	etching : 36% phosphoric acid with colloidal silica adhesive : PENTA, UDMA, Resin R5-62-1, T-resin, D-resin, nanofiller, cetylaminehydrofluoride and acetone
Instructions for use	primer application : 20sec gentle air dry adhesive application light-cure 10sec	etching : 15sec rinse & blot dry adhesive application : 20sec dry : 10sec light-cure : 10sec

**Table 3.** Code of control and experimental group by C-factor. (n=8)

Material	Control (c<1.0)	C=2.4	C=3.4
Clearfil™ AP-X	ACO	AC2	AC3
Esthet-X™	ECO	EC2	EC3

were made according to the configuration factor (Table 3).

### 1. Specimen fabrication

The specimens of control group of each resin composites were made in a split steel mold (25×2×2mm, Fig. 1) with top and bottom surfaces covered by a clear matrix. The specimens(n=8) were made with light irradiation(60s) in the curing unit(LABO-LIGHT LV 1, GC, Japan) on both the top and bottom surfaces. The specimens were stored in distilled water at 37°C for 24hours prior to testing. In experimental groups, the specimens of each resin composite with 2.4 configuration factor(C-factor)(AC2 & EC2) were made in a simulated cavity(25×5×3mm) made of glass plates. Before filling the resin into the cavity, the inner surfaces of the glass cavities were sand-blasted and treated with silane and corresponding

dentin bonding systems. The specimens were made with light irradiation(60s) in the curing unit on both the top and bottom surfaces. Then the specimens were cut with slow speed diamond saw (ISOMET, Buehler, USA) under copious water to make resin bars(25×2×2mm). The storage and testing procedures of specimens were carried as same as control groups.

The specimens of each resin composite with C-factor of 3.4(AC3 & EC3) were made as the same procedures as the specimens with C-factor of 2.4, but the size of the simulated cavity(25×5×5mm) was different.

### 2. Flexural strength and elastic modulus determination

Flexural strength and elastic modulus were evaluated in three-point bending by International Standards Organization(ISO) 4049 for testing of resin-based filling materials reported previously<sup>19)</sup>. The specimens were tested on a universal testing machine(EZ Test, Shimadzu, Japan, Fig. 2) in bending on a span of 20mm at a crosshead speed of 1mm/min(Fig. 3). The crosshead travel of the universal testing machine was synchronized with the motion of the strip chart. The slope of the linear portion of the load-versus-time curve was used in the

calculation of the modulus, according to the followed equation :

$$\text{Flexural strength calculation : } 3 P \cdot L / 2 b \cdot W^2$$

- P : Maximum load at fracture
- L : Length of the span(20mm)
- b : Thickness of the specimen
- w : Width of the specimen

$$\text{Elastic modulus calculation : } P \cdot L^3 / 4 d \cdot b w^3$$

- P : Change of the load
- L : Length of the span
- d : Crosshead travel
- b : Thickness of the specimen
- w : Width of the specimen

### 3. Polymerization shrinkage determination

The amount of the linear contraction of two resin composites were determined with Linometer(R & B, Korea). A small amount of resin composite(about 30mg) was placed on the thin stainless steel disk(25mm diameter, 0.5mm thickness) and covered with slide glass. The resin composite was cured for 60seconds. Since the disk was attached to the computerized sensor, if polymerization contraction occurs, the steel disk was raised and the sensor detected how long distance the disk moved.

The shrinkage data was obtained for 60 seconds with data interval of 0.5 second.

### 4. Examination of the fractured surface

After flexural strength testing, a fragment of the fractured specimens was selected randomly, sputter coated with gold and examined the fractured surfaces

with scanning electron microscope(S-2300, Hitachi, Japan). Then the characteristics of the specimens of control and experimental groups were compared.

### 5. Statistical analysis

The data obtained from above experiments was evaluated using one-way analysis of variance(ANOVA) / Tukey's test at  $\alpha \leq 0.05$  level.

## III . RESULTS

### 1. Flexural strength

The flexural strength was decreased with increasing of configuration factor of the specimen. The flexural strength of ACO was much higher than that of AC2 and AC3, and there was significant difference among the test groups using Clearfil™ AP-X( $p < 0.05$ ). In the groups using Esthet-X™, each flexural strength showed no significant difference( $p > 0.05$ ).

### 2. Elastic modulus

The elastic modulus was decreased with increasing of configuration factor. The elastic modulus of control groups was significantly higher than that of experimental groups, though there was no significant difference between experimental groups for each resin composite ( $p > 0.05$ ).

### 3. Polymerization shrinkage determination

The polymerization shrinkage of Clearfil™ AP-X is much less than that of Esthet-X™. Probably, the fact is due to the difference of filler content between two resin composites (Table 1).

**Table 4.** Flexural strength (MPa±S.D.) of the experimental groups.

Resin composite	Control	C=2.4	C=3.4
Clearfil™ AP-X	200.79±25.01 <sup>a</sup>	174.77± 9.7 <sup>b</sup>	131.07±15.7 <sup>c</sup>
Esthet-X™	117.48±20.72 <sup>a</sup>	112.45±23.7 <sup>a</sup>	93.44±17.71 <sup>a</sup>

#### 4. SEM examination of fractured surface

Fractured surface of the specimens of group ACO(Fig. 7, 8) showed smooth and homogenous appearance, but those of the specimens of the group AC2(Fig. 9) and AC3(Fig. 10) showed irregular and rough features. Especially, the specimen of group AC3 showed crack lines at the fractured surface. It means that there was stress concentration within the

resin mass during polymerization. The examination of the groups using micro-hybrid composite(Fig. 11, 14) was similar to that of hybrid composite, but general features of fractured surfaces were smoother than those of hybrid composite.

#### IV. DISCUSSION

The contraction stress of resin composite depends upon the type and level of filler included. Generally, an increased filler loading contribute to reduced polymerization shrinkage, since the overall polymerization shrinkage depends on the amount of polymer matrix presented<sup>20,21)</sup>. The higher stiffness leads to increased stress for a given contraction strain, according to Hooke's law. ( $E = \kappa\sigma/\epsilon$ ,  $E$  : Elastic modulus,  $\kappa$  : Proportional constant,  $\sigma$  : Stress,  $\epsilon$  : Strain). Therefore, the composite stiffness and the amount of contraction both play important roles in the generation of stress in dental composite restoration. In this study, hybrid type resin composite showed higher mechanical properties(flexural strength and elastic modulus) than micro-hybrid

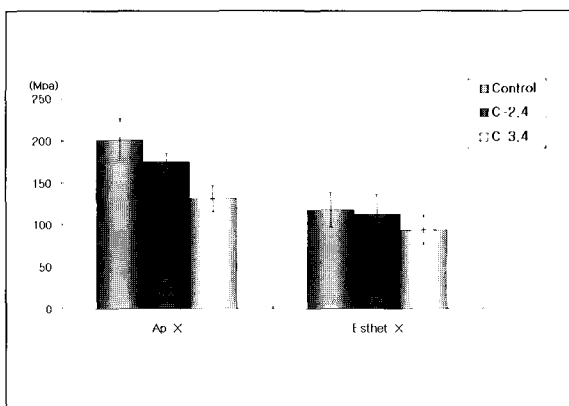


Fig. 4. Flexural strength of the experimental groups.

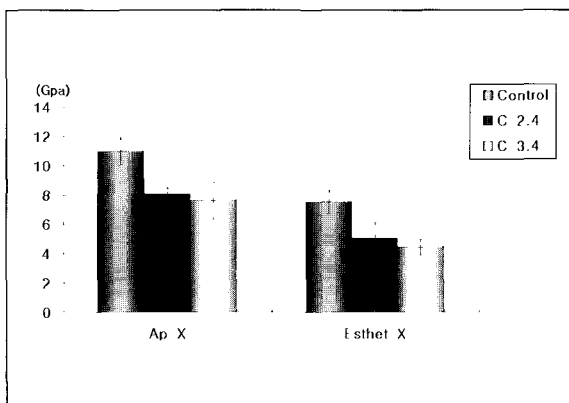


Fig. 5. Elastic modulus of the experimental groups.

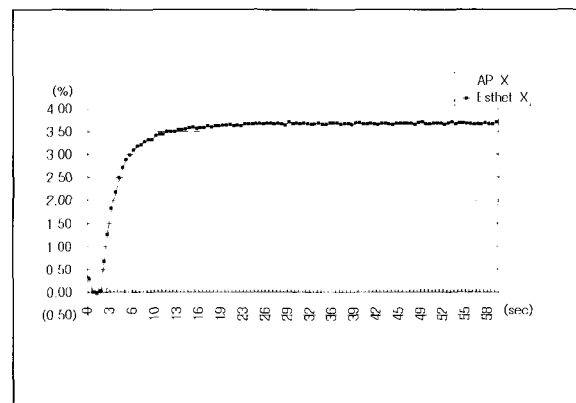


Fig. 6. Polymerization shrinkage of two resin composites.

Table 5. Elastic modulus (GPa S.D.) of the experimental groups

Resin composite	Control	C=2.4	C=3.4
Clearfil™ AP-X	10.96±0.94 <sup>a</sup>	8.04±0.44 <sup>b</sup>	7.60±1.26 <sup>b</sup>
Esthet-X™	7.49±0.81 <sup>a</sup>	5.07±1.01 <sup>b</sup>	4.47±0.54 <sup>b</sup>

composite for all configuration factor ( $p < 0.05$ ). Generally, it is accepted that an increased filler level should contribute to increased mechanical properties and reduced polymerization shrinkage. From the polymerization shrinkage graph in this study, the gel-point of hybrid composite was found to be occurred earlier than micro-hybrid composite. During the period before occurring gel-point (pre-gel stage), the developing contraction stresses can be relieved due to the flow capacity of the material, but after gel-point (post-gel stage), the flow capacity of the material decreased and the developing contraction stress can cause microdefect or cracks in the materials. The specimen being stressed, the defect and/or cracks played a role as stress raiser and the mechanical properties decreased. The lower elastic modulus and higher shrinkage of the micro-hybrid composite is indirect evidence that flow capacity is achieved mainly by increasing the proportion of monomer in the formulation of the composite pastes. So despite of the less polymerization shrinkage, hybrid composite was affected more than micro-hybrid composite by increase of C-factor.

In SEM, the fractured specimens of the groups of Clearfil™ AP-X showed rougher surfaces than those of Esthet-X™. The phenomenon was maybe due to the difference of filler particle size between the resin composites. The mean filler particle size of hybrid and micro-hybrid composite was  $3.0\mu\text{m}$  and  $0.6\text{--}0.8\mu\text{m}$ , respectively. Indeed, the specimens with lower configuration factor showed smoother appearance than those with higher configuration factor. Generally the fact is accepted that the higher the configuration factor of the cavity, the more stresses develop. So resin composites filled in the cavity with higher configuration factor experience severe contraction stresses during polymerization and the forces can cause crack in the resin mass. Flexural and/or tensile strength are significantly changed by the presence of defects due to generation of stress concentrations. In brittle materials such as dental resin composites or porcelain, microcracks which exist on the surface or inside the material, decrease the material strength<sup>22</sup>.

It is supposed that as the configuration factor of the cavity increased, the flow capacity of the resin com-

posites decreased and more stresses occurred that cause detrimental effect on the mechanical properties of the resin composites in the restricted polymerization environment.

There have been many studies about the effect of polymerization shrinkage on dentin bonding<sup>23-29</sup>, and it is shown that polymerization shrinkage can cause contraction stress which has detrimental effect on the bond strength between resin composites and tooth structures. But, there are few studies about the effect of contraction stresses on the mechanical properties of the resin composites. In this study, the flexural strength and the elastic modulus of resin composites had a tendency to decrease with increase of the C-factor. In case of hybrid type composite, the flexural strengths of test groups showed significant difference ( $p < 0.05$ ), but those of micro-hybrid composite were not significantly different ( $p > 0.05$ ). It may be due to the filler content of the resin composite. Hybrid type composite used in this study had higher filler content (84.5wt%) than micro-hybrid type resin composite (77.0wt%).

So, hybrid composite showed less polymerization shrinkage than micro-hybrid composite. But despite of the less shrinkage, the higher elastic modulus ( $10.96 \pm 0.94\text{GPa}$ ) and earlier emergence of gel-point of hybrid composite makes it more influenced by increase of C-factor than micro-hybrid composite.

In clinical situation, many dentists have faced to problem related to the contraction stress of resin composite. Actually, the contraction stress of resin composites and bonding agents under clinical situations is much more complex than what was previously thought. The high values of contraction stress reported from some studies for specific configurations of restorations may overcome the bond strength of resin to dentin. This might explain a large number of bond failures and gap formation frequently observed in studies with bonding agents. However, an early interfacial stress build-up occurs during the polymerization in situ of resins bonded to tooth structures. Moreover, under the rigid set-up situation (in case that bond strength is higher than contraction stress), the contraction stresses may affect the mechanical properties of resin composites.

There are several solutions to the competition

between the bond strength of resins to dentin and the forces of polymerization contraction.

One approach of these solutions is to develop dentin bonding agents that are able to develop immediate bond strengths that are greater than those developed by polymerization contraction and are equal to those obtained to acid-etched enamel. But, this can cause another problem, that is, the restrict polymerization environment decrease the mechanical properties of the resin composites.

A second solution is to cover the bonded surfaces with an elastic cavity liner. This acts as a "shock-absorber" and places a low modulus material between the relatively rigid dentin and resin composite<sup>30</sup>. The resin interdiffusion zone generated as the main bonding mechanism of most of the current dentin bonding systems may also function as a stress relaxation layer, since it has a lower modulus than the underlying mineralized dentin<sup>31</sup>.

A third solution is to insert resin composites in increments to reduce the volume of the resin that is shrinking during polymerization. The use of reduced amounts of resin to be polymerized at each increment has been shown to reduce the stresses generated to the cavity walls<sup>15</sup>. Incremental filling techniques actually lower the C-factor to below 1, since there is usually almost as much free surfaces as bonded surface in any single increment.

A fourth solution is development of the new materials. Several researchers are attempting to develop new resin composites that do not shrink when they polymerize<sup>32</sup>. For example, Spiro- orthocarbonate monomers have been synthesized that expand during polymerization through a double ring-opening process<sup>33</sup>. Further improvements are being developed to cause sufficient double ring-opening to generate enough expansion to overcome the shrinkage generated by conventional dimethacrylates.

In this study, hybrid type resin composite was more influenced than micro-hybrid composite by increase of C-factor. This fact is due to the difference of time, which gel-point appeared and filler content between the two resin composites. In case of hybrid composite, high filler content makes it more stiff and the earlier emergence of gel-point decrease the flow capacity of the material. The polymerization shrink-

age was proved to have an effect on flexural strength and elastic modulus of composites. The results of this study shown that the higher the C-factor of cavity, the lower the mechanical properties of the resin composites.

Then the contraction stress of composites could be caused by polymerization shrinkage affect both bond strength of resin to tooth structures and mechanical properties of resin composites itself.

## V. CONCLUSION

To achieve successful resin composite restoration, many factors such as volumetric shrinkage of resin mass, configuration factor of the cavity and the type of the resin can be considered. This study was designed to evaluate the effect of configuration factor of cavity on the mechanical properties of resin composites such as flexural strength and elastic modulus. From the results of this study, it can be concluded as follows :

1. The flexural strength of both resin composites was decreased as the C-factor increased. But there were no significant difference among the groups in micro- hybrid resin composite. ( $p>0.05$ ).
2. The elastic modulus of both resin composites was decreased with increasing the configuration factor. The elastic modulus of control groups(with C-factor<1) was significantly higher than that of experimental groups ( $p<0.05$ ).
3. The amount of polymerization shrinkage of hybrid composite was less than that of micro-hybrid composite. Although hybrid composite was less shrink than micro-hybrid composite, the flexural strength of the former was much affected by increase of the configuration factor.
4. In SEM, the higher the C-factor of the specimens, the rougher the fractured surfaces were. Generally, the fractured surfaces of the Clearfil™ AP-X showed more irregular pattern than Esthet-X™, probably due to the difference of the filler particle size and flow capacity between two resin composites.

This study suggests that the mechanical properties of resin composites can be affected by the configura-

tion factor that plays an important role in the confined curing environment.

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사진부도 ①

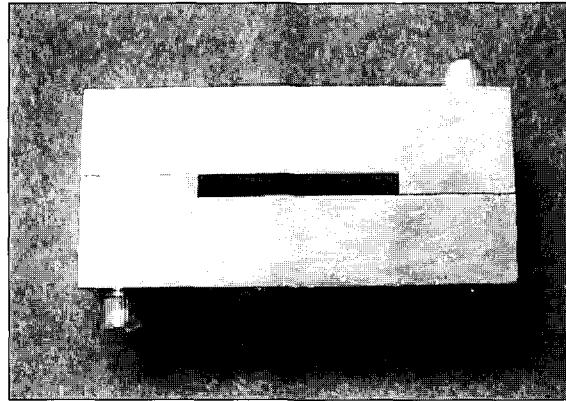


Fig. 1. Split steel mold used in this study

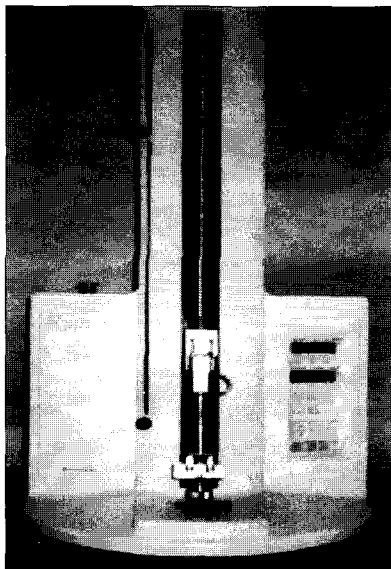


Fig. 2. Universal testing machine

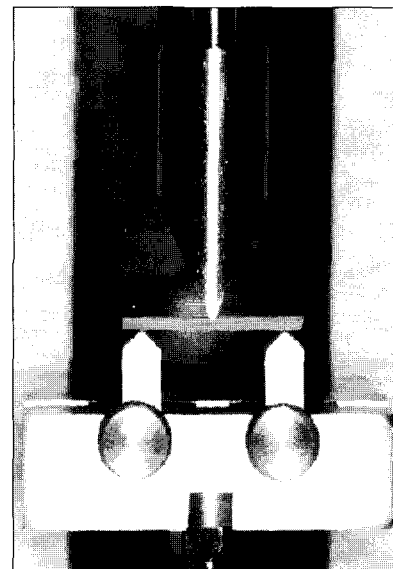


Fig. 3. 3-point bending procedure

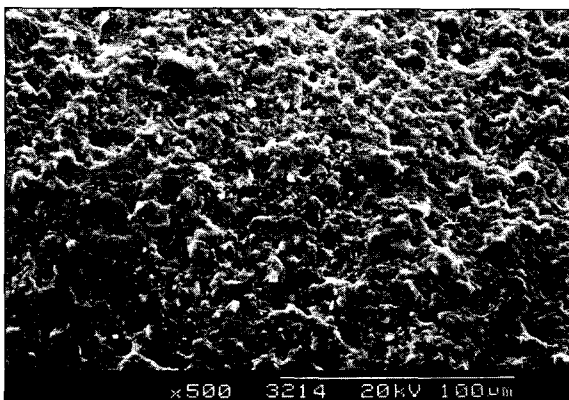


Fig. 7. The fractured surface of the ACO group ( $\times 500$ )

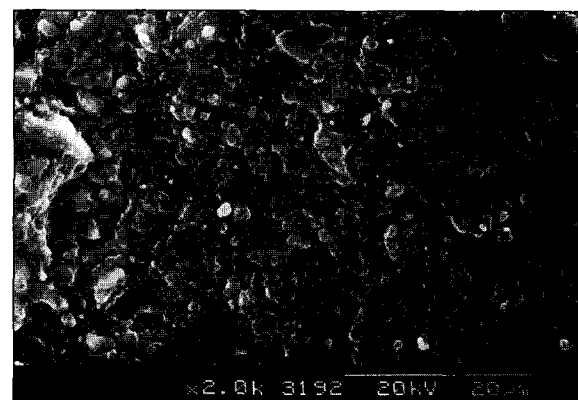


Fig. 8. The fractured surface of the ACO group ( $\times 2,000$ )

사진부도 ②

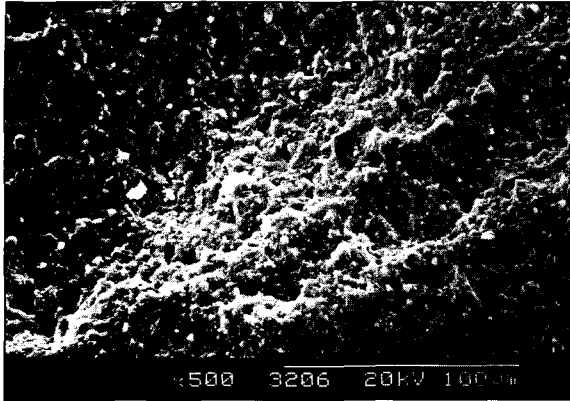


Fig. 9. The fractured surface of the AC2 group(×500)

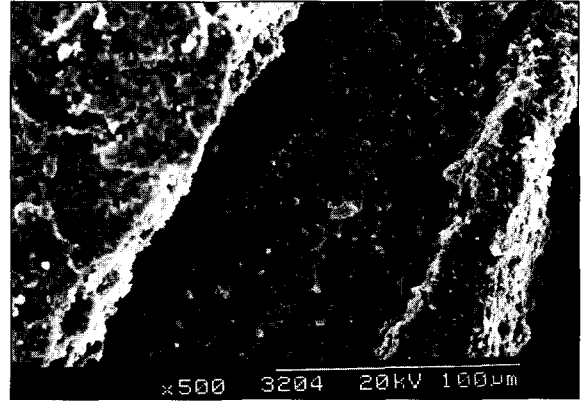


Fig. 10. The fractured surface of the AC3 group(×500)

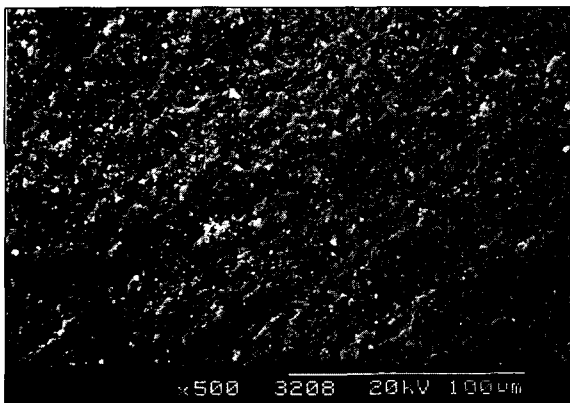


Fig. 11. The fractured surface of the ECO group(×500)

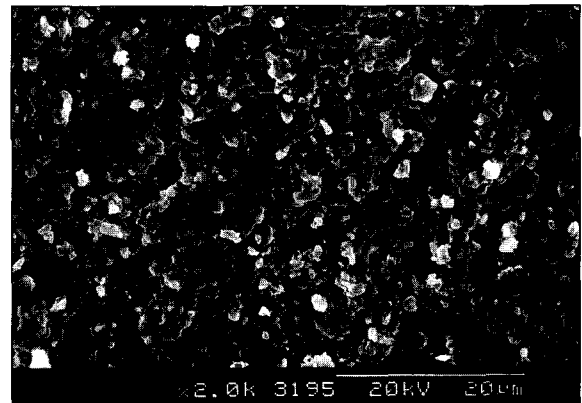


Fig. 12. The fractured surface of the ECO group(×2,000)

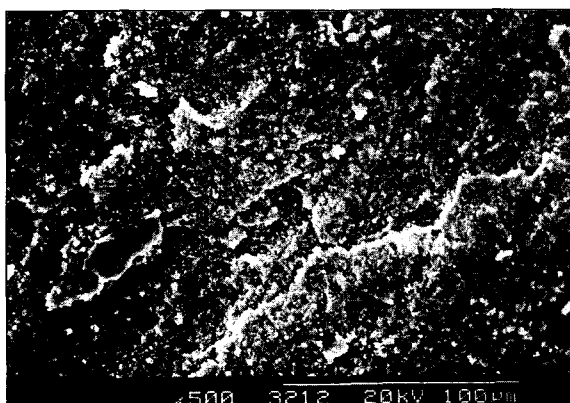


Fig. 13. The fractured surface of the EC2 group(×500)

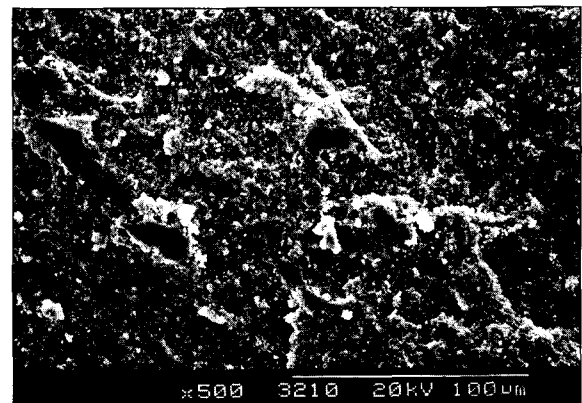


Fig. 14. The fractured surface of the EC3 group(×500)