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# Influences of Strain Rate and Water on Fracture Toughness of Heat Cured Denture Base Acrylic Resin

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***Abstract*** *The effects of strain rate and water on the fracture toughness ($K_{IC}$) of heat cured denture base acrylic resin were investigated using the single edge pre-cracked beam (SEPB) method. It was found that by drying notched specimens, the natural pre-crack needed for specimen preparation could be easily triggered and the length could be controlled. The results using dry specimens showed that the $K_{IC}$ was $1.04 \pm 0.03$ to $1.31 \pm 0.09$ MPa・$m^{1/2}$ and gently increased with increasing cross head speed (CHS). This was attributed to the decrease in the number of polymer chains drawn out along with the increase of CHS. From the observation of the fracture surface of the wet specimen, it was confirmed that slow crack growth (SCG) occurred during measurement and reached zero at the CHS of 100 mm/min. The $K_{IC}$ was extremely great at the lower CHS and then decreased with increasing CHS. The great $K_{IC}$ resulted from the stress dispersion due to the formation of craze and microcrack at the crack tip with the development of the SCG. Accordingly, $K_{IC}=1.05 \pm 0.06$ MPa・$m^{1/2}$ at the zero SCG was regarded as a appropriate value for the heat cured denture base acrylic resin with absorbed water.*

**Key words** : Fracture toughness, Heat cured denture base acrylic resin, Strain rate, Water, Single edge pre-cracked beam method

## INTRODUCTION

The cracking of dental base resin is a fatigue phenomenon[1~5] consisiting of the crack initiation and propagation. The mechanical parameter connected with this phenomenon is the fracture toughness ($K_{IC}$)[6], which is a measure of resistance in materials with small scale yielding. Thus, $K_{IC}$ has a great relevance to cracking in dental base resin. However, in resin both brittle and ductile fractures take place according to the circumstances. In evaluating the $K_{IC}$, therefore, it is important to elucidate the influences of the factors on $K_{IC}$. Moreover, in the preparation of pre-cracked specimens, it is desirable that the precrack length is as equal as possible in order to avoid the scattering of $K_{IC}$, as indicated by the study of R-curve behavior[7] which has been widely carried out in ceramics[8~11]. As recommended by ASTM D5045-95[12], the pre-crack is usually produced by tapping on a fresh razor blade placed in the machined notch. As a matter of course, it is difficult to obtain the same pre-crack length.

In the present investigation, by establishing a method for initiating the pre-crack, the strain rate dependence of $K_{IC}$ in both dry and water-absorbed specimens was examined. Moreover, the fracture surfaces were also observed in order to elucidate the influence of slow crack growth (SCG)[14] on $K_{IC}$.

## MATERIALS AND METHODS

### 1. Specimen preparation

Heat cured denture base acrylic resin (Acron clear, No. 5, GC Co., Tokyo, Japan) was mixed at a powder-liquid ratio of 100g/34.1g, and the dough was

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filled into a stainless steel die with an inner volume of 50×75×5 $mm^3$ and then pressed with a stainless plate. The insides of the die were finished to mirror-like surface. To allow a polymerization shrinkage cavity to form on one side of the resin plate, the wide surface side of the die was put parallel to the bottom of the pan. The water in the pan was heated at the rate of 2 ℃/min, and held for 30 minutes after boiling, and then cooled spontaneously to the room temperature.

The thickness of the polymerized resin plates was reduced to 4 mm by scraping on the shrinkage cavity side using waterproof abrasive paper and then polished with dental rouge. From these plates, beam specimens (4×8×50 mm, ASTM D5045-95[12] and E399-90[15]) with thickness B=4 mm and width W =8 mm were cut with a diamond cutter (MC-411, Maruto Inc., Tokyo, Japan).

**2. Initiation of pre-crack**

The specimen configuration is illustrated in Fig. 1, and photographs of the notch and pre-crack are shown in Fig. 2. Initially, the specimen was notched to 2 mm in depth and 0.1 mm in width in the middle of the beam using a diamond notch blade (Maruto Inc., Tokyo, Japan), and then dried in an incubator at 50 ℃ so as to trigger the natural crack easily and control the length as accurately as possible. The pre-crack was generated by a three-point bend load using a material testing machine (Autograph AG-5000C, Shimadzu Co., Kyoto, Japan). The pre-crack length was restricted to 4.0±0.1 mm by controlling the cross head while observing the extending pre-crack with an optical digital microscope (VH-6300, Keyence Co., Osaka, Japan), and the specimens kept for more than 1 week in the same incubator (dry specimen). To examine the influence of water on $K_{IC}$, half the pre-cracked specimens were soaked in the distilled water (wet specimen) for more than 1 week prior to measurement.

**3. Measurement of fracture toughness ($K_{IC}$)**

The $K_{IC}$ measurements were carried out by means of the single edge pre-cracked beam (SEPB) method[15] using the same testing machine. A three point bend jig with a support span of 32 mm was placed in the glass vessel. In measuring the dry specimens, the upper part of the vessel was overlaid with a film so that air could not enter, and Ar gas

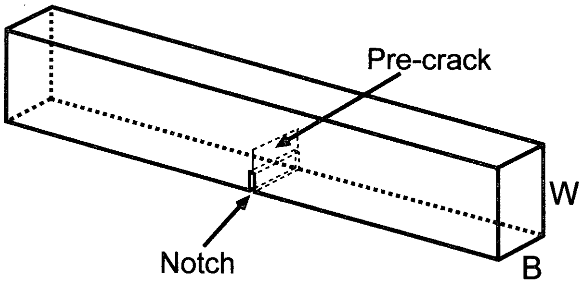


Fig. 1. Specimen configuration for the measurement of fracture toughness of heat cured denture base acrylic resin
W= 8 mm, B=4 mm

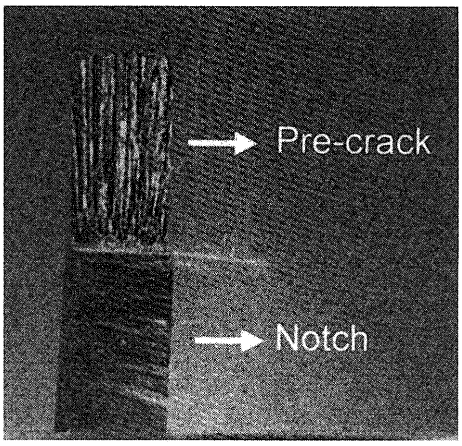


Fig. 2. Pre-crack initiated from the notch by the three point bend mode

was introduced. For the wet specimens, the jig was put in the glass vessel and covered completely with distilled water. The cross head speed (CHS) was changed from 0.01 to 100 mm/min by steps of 10×, and five specimens were tested at every CHS. The lengths of both the pre-crack ($a_0$) and the extended crack due to the SCG were measured and averaged from the values at three sites on the fracture surface. The $K_{IC}$ was calculated by Equation (1)[15] from the total length (a),

$$K_{IC} = (P_{max} \cdot S/B \cdot W^{3/2}) \cdot Y(\alpha) \quad (1)$$

where $P_{max}$ is the fracture load, S (=4W) is the support span of 32 mm, and $Y(\alpha)$ is the shape factor expressed by Equation (2),

$$Y(\alpha) = 1.5\alpha^{1/2}\{1.99 - \alpha(1-\alpha)(2.15 - 3.93\alpha + 2.7\alpha^2)\} / (1+2\alpha)(1-\alpha)^{3/2} \quad (2)$$

where $\alpha = a/W$.

**4. Observation of fracture surfaces**

After the measurement of $K_{IC}$, the fracture surfaces were observed with an optical digital microscope (VH-6300, Keyence Co., Osaka, Japan).

## RESULTS

### 1. Fracture toughness ($K_{IC}$)

With dry specimens, it was confirmed that SCG has not occurred at any CHS during $K_{IC}$ measurement. On the contrary, wet specimens experienced large SCG. Fig. 3 shows the relationship between the crack growth length and the CHS in wet specimens. The SCG was greater at lower CHS and zero at 100 mm/min. Fig. 4 shows the $K_{IC}$ of dry and wet specimens calculated using the total crack (pre-crack + crack by SCG) length, respectively. The $K_{IC}$ of dry specimens increased gradually with increasing CHS, but that of wet specimens decreased rapidly.

### 2. Fracture surface

Fig. 5 indicates the fracture surfaces of both dry and wet specimens at three CHS. The bottom part of each photograph is the pre-crack surface, and the crack extension direction is from bottom to top. The significant features of the pre-crack surface are that many linear scratches are observed in the extension direction, and many resin particles can be identified. In dry specimens, catastrophic fractures start from the tip of the pre-crack. The pattern of the fracture surface resembles that of the pre-crack, although that of 100 mm/min was a little different. In wet specimens, catastrophic fracture from the tip of the pre-crack took place only at 100 mm/min, and the others showed the distinctive fracture surface patterns produced by the SCG.

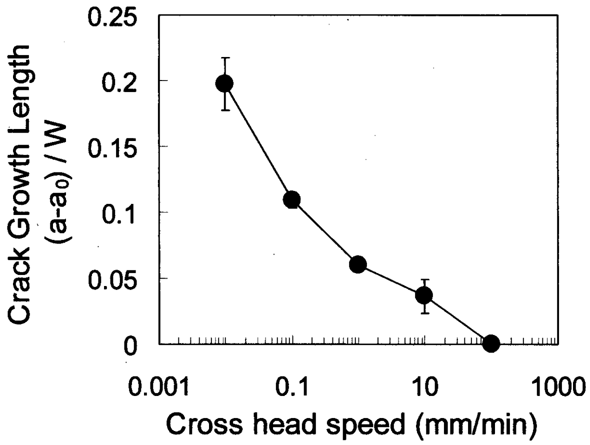


Fig. 3. Effect of cross head speeds on crack growth length in wet specimen
a : total crack length, $a_0$ : pre-crack length

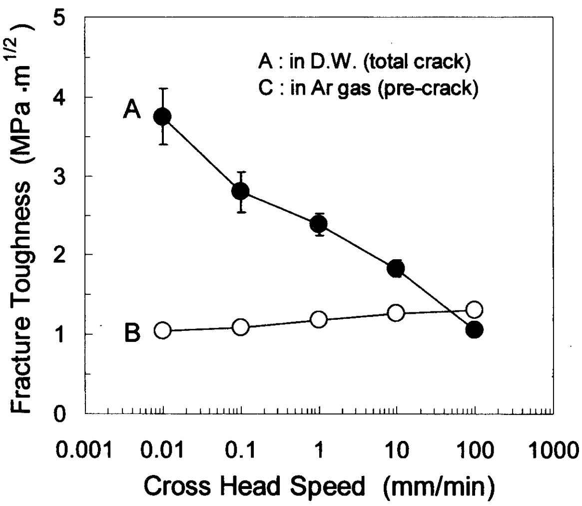


Fig. 4. Fracture toughness ($K_{IC}$) of heat cured denture base acrylic resin A (wet specimen) and B (dry specimen) were measured in distilled water and Ar gas, and calculated using total crack length and pre-crack length, respectively.

## DISCUSSION

### 1. Pre-crack initiation

So far, the pre-crack in a resin has been initiated by hitting the edge of a razor blade put in the notch[12]. This procedure has produced scattering in both length and angle. Nishikawa et al.[13] attempted to initiate natural pre-cracks by the three point bend mode, but it was not always easy to trigger a natural crack from the notch. In this study, however, it was found that, by drying the specimens in an incubator at 50 ℃ for more than 1 week, the pre-crack could be easily triggered. By keeping the specimens in the same incubator after initiating the pre-crack, it was expected that the residual stress near the crack tip would be relaxed.

### 2. Effect of water on slow crack growth (SCG)

As shown in Fig. 5, the patterns which appeared on the fracture surface of dry specimen resembled those of the pre-crack at every CHS except 100 mm/min. The tip of the pre-crack was clearly identified at every CHS. From these observations on the dry specimen, it was confirmed that catastrophic fracture occurred from the pre-crack tip when the stress intensity factor ($K_I$) reached the critical value ($K_{IC}$). In the wet specimen, however, quite a different pattern appeared, that is, a curved stripe along the direction of crack extension and a stripe perpendicular to that one. As such striped patterns were never observed in the dry specimens, it was clear that

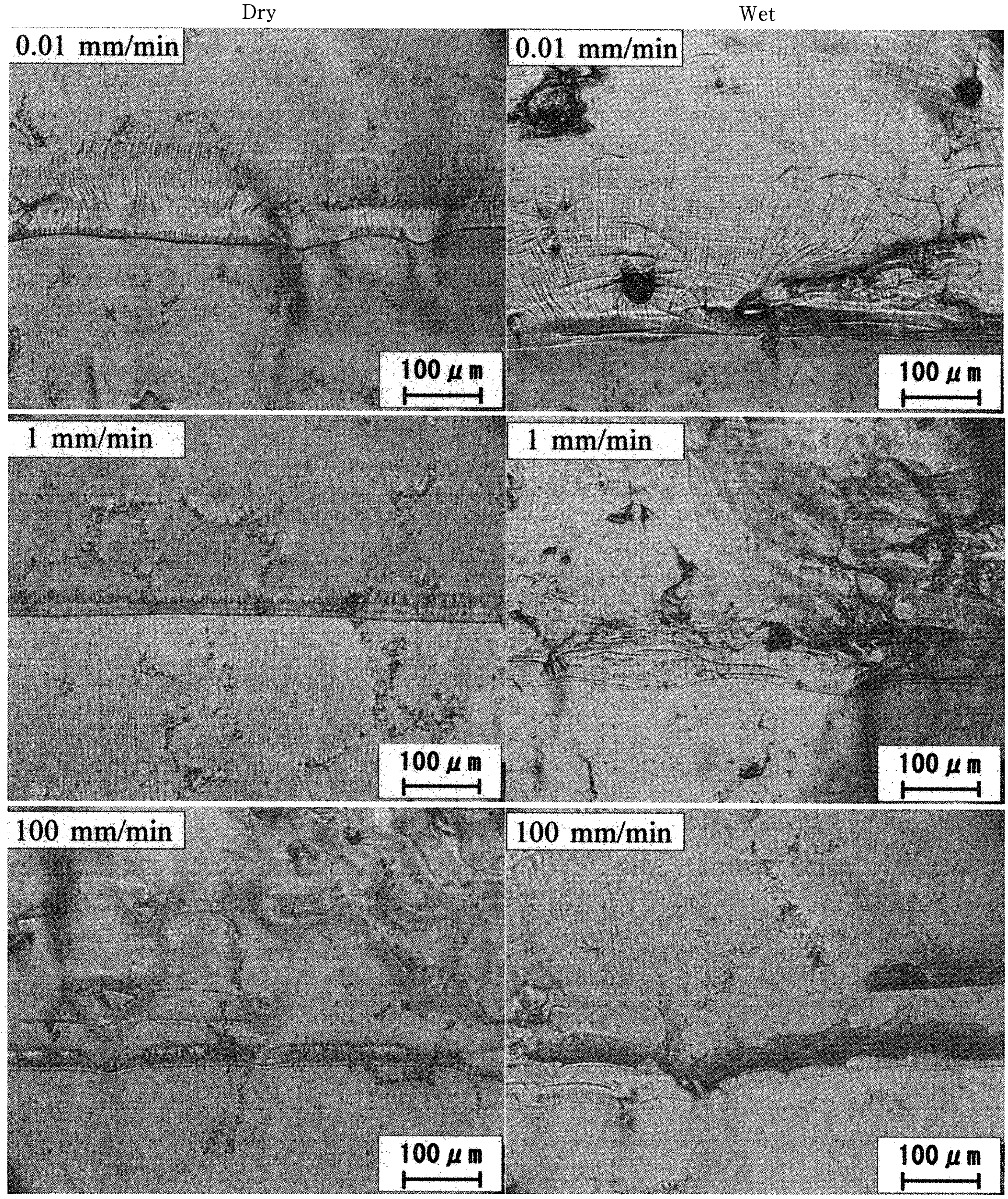


Fig. 5. Fracture surfaces after measurement of $K_{IC}$

these patterns were formed by the action of water.

Fig. 6 shows a peculiar pattern created by the SCG which proceeded at the CHS of 0.01 mm/min, where the main crack has propagated from the bottom to the top. The patterns indicated by the white arrows would be wakes of crack like concentric circles which have started to grow from the defects ahead of the main crack tip. We consider that the concentric circle cracks were prevented from extending to the rear because the main crack ran into them, but the front side would be able to grow together with the main crack. Accordingly, the crack growth lengths in Fig. 3 were determined by judging that these observed patterns on the fracture surfaces were not due to brittle fracture but rather the SCG induced by the water.

**3. Effect of strain rate and water on fracture toughness ($K_{IC}$)**

As expressed in Equation (1), both the crack length and the load causing the catastrophic fracture are needed to calculate the $K_{IC}$. In dry specimens, as the SCG has not occurred at all, the $K_{IC}$ was given from $a_0$ and $P_{max}$ as shown in Fig. 4. Hence, the gentle increase in curve (B) with increasing CHS would

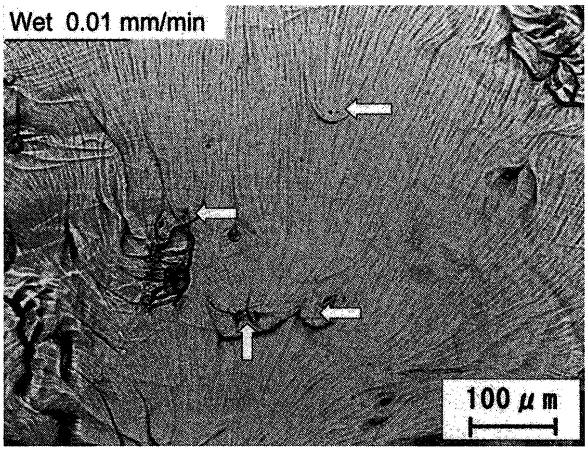


Fig. 6. Pattern created by SCG in wet specimen
The crack propagation direction is from bottom to top. White arrows indicate the starting points of new crack initiated ahead of the main crack tip before it has come.

be the effect of the strain rate only, related to the number of survived polymer chains. Near the crack tip, when the stress is applied, some of the polymer chains will be pulled out or cut. This is a time dependent phenomenon, that is, the shorter the stress applying time is, the more polymer chains will survive up to a catastrophic failure. Therefore, the greater the strain rate is, the more the $K_{IC}$ will increase and approach the value of resin itself.

On the contrary, considering that the $K_{IC}$ of the wet specimen in which the SCG occurred during loading was extremely large at lower CHS as shown in Fig. 4, the nature of the crack tip in SCG might be different from that of the pre-crack. This can be undoubtedly attributed to the water, but there is also no doubt that the crack length corresponding to $P_{max}$ would be equivalent to the total value measured on the fracture surface. Because the water at the tip of the pre-crack decreases the intermolecular force of the polymer chains, a craze[16~20] is formed easily at the pre-crack tip and grows into the crack. As a result, the SCG can proceed under a stress intensity factor ($K_I$) smaller than $K_{IC}$. With the development of the SCG, many more crazes are produced and change into a branched crack. These crazes and microcracks cause stress dispersion and the load necessary for rupturing the specimen is increased. This is a reason for the extreme large $K_{IC}$ at lower CHS. Therefore, the $K_{IC}=1.05\pm0.06$ MPa·m$^{1/2}$, at which the SCG had not occurred as shown in Fig. 3, can be regarded as the most appropriate value for heat cured acrylic resin including absorbed water.

## CONCLUSION

In preparing the single edge pre-cracked beam (SEPB) specimen for the fracture toughness ($K_{IC}$) measurement of the heat cured denture base acrylic resin, it was found that, by drying at 50 ℃, the natural pre-crack could be easily triggered from the machined notch with the three point bend mode. The $K_{IC}$ of the dried specimens was $1.04\pm0.03$ to $1.31\pm0.09$ MPa·m$^{1/2}$ and gently increased with increasing strain rate. Specimens with absorbed water had greater toughness at lower strain rates and the values decreased with increasing strain rate. The most appropriate value was regarded as $K_{IC}=1.05\pm0.06$ MPa·m$^{1/2}$, at which the slow crack growth (SCG) never occurred.

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# 義歯床用加熱重合レジンの破壊靱性値に及ぼす歪み速度と水の影響

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キーワード：破壊靱性値，義歯床用加熱重合レジン，歪み速度，水，予亀裂導入破壊試験法

**抄録**　義歯床の割れは臨床上大きな問題であるにもかかわらず，その原因は現在でも明らかになっていない．亀裂の発生と最も関連性が深い性質は破壊靱性値($K_{IC}$)であるが，条件によって値が全く異なるという問題がある．そこで，本研究では義歯床用加熱重合アクリルレジンについて，三点曲げによる予亀裂導入破壊試験法を用いて，$K_{IC}$に及ぼす歪み速度と水による影響を検討し，正確な$K_{IC}$の決定を試みた．その結果，先ず予亀裂を導入する前に試験片を50℃で1週間以上乾燥させることにより予亀裂の導入が極めて容易になり，方向と長さを正確に設定できることが明らかになった．これらの乾燥試験片の場合，脆性破壊が予亀裂の先端から進行したことが破断面の観察から確認され，$K_{IC}$は歪み速度の増加とともに緩やかに増加することがわかった．これは歪み速度が増加するほど引き抜かれずに残る高分子鎖の数が多いためであると考えられた．また，予亀裂導入後に吸水させた試験片では歪み速度が小さいほど測定中に低速亀裂成長が進行していたことが破断面の観察から確認され，クロスヘッドスピードが100 mm/minの時に零になることがわかった．同時に$K_{IC}$はひずみ速度の増加とともに顕著な減少を示した．この原因はSCGが進行するほど亀裂先端付近にクレーズと微少亀裂の発生が顕著になり，その分だけ応力が分散されて破断荷重が増加したためであると考えられた．そこで，SCGがまったく起きていなかった場合の$K_{IC}=1.05\pm0.06$ MPa･m$^{1/2}$が吸水試験片の破壊靱性値として適切であると考えた．