X-RAY EXAMINATION OF FRACTURE SURFACE OF DENTAL MATERIAL

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ABSTRACT

X-ray diffraction observation of metal fracture provides fracture analysis with useful information on the mechanics and mechanical conditions of fracturing. This method is called “X-ray Fractography” and has been developed especially in Japan as a new engineering tool for fracture analysis[1-3]. X-ray fractographic technique was applied to fracture surface of dental material. The relationship between X-ray parameters and fracture mechanics parameters plays a key role in determining the mechanical condition of fracture of dental material.

INTRODUCTION

In the present paper, the fracture toughness tests of dental material were conducted for pre-cracked as well as notched specimens. Pre-cracking was introduced to the trace of Vicker’s indentation by longitudinal compression. The first part described the effect of the notch-tip radius on the fracture toughness value and the plastic zone size determined from the residual stress distribution beneath the fracture surface. Then, these results were discussed in connection with fracture mechanics and fracture processes.

EXPERIMENTAL PROCEDURE

Material and Fracture tests
The dental material used in this experiment was Hydroxyapatite (HAp) made by NGK Spark Plug Co., Ltd. Main component of HAp is Ca₁₀(PO₄)₆(OH)₂, and the crystal structure is
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hexagonal system. Specimens were sintered in an electrical furnace. The bending strength was 100Mpa. Pre-cracking was introduced to the trace of Vicker’s indentation by longitudinal compression as shown in Figure 1. The loading plane on the end surfaces was adjusted. Figure 2 shows bluntly notched specimen used for fracture tests. The notch was made with diamond blades and finished by chemical polishing. The radius of the notch-tip varied between 0.1mm and 0.5mm. All the specimens were fractured under three points of bending as shown in Figure 3. The crack extension was monitored on the basis of load-displacement record.

Figure 1 Schematic illustration of pre-cracking method

Figure 2 Dimension of test specimen (in mm)

Figure 4 Schematic illustration of X-ray irradiated area on the fracture surface.(in mm)

Table 1 X-ray conditions for stress measurement

<table>
<thead>
<tr>
<th>X-ray optics</th>
<th>Collimated beam</th>
</tr>
</thead>
<tbody>
<tr>
<td>Characteristic radiation</td>
<td>Cr-Kα</td>
</tr>
<tr>
<td>Detector</td>
<td>PSPC</td>
</tr>
<tr>
<td>Diffraction line</td>
<td>HAp252</td>
</tr>
<tr>
<td>Diffraction angle, deg</td>
<td>139.7</td>
</tr>
<tr>
<td>Tube voltage, kV</td>
<td>30</td>
</tr>
<tr>
<td>Tube current, mA</td>
<td>60</td>
</tr>
<tr>
<td>Irradiated area</td>
<td>φ1(0.785mm²)</td>
</tr>
<tr>
<td>Peak determination</td>
<td>Half value breadth method</td>
</tr>
</tbody>
</table>
X-ray Measurement
The distribution of the residual stress near the fracture surface was measured with the X-ray diffraction method. X-ray equipment used was a Rigaku Rint 2500 stress analyzer. The condition of X-ray observation are given in Table 1. The diffraction profile of the (252) plane was obtained by Cr-Kα-X-rays from the area φ1mm² and was measured by removing successfully the surface layer by electron polishing as shown in Figure 4.

RESULTS AND DISCUSSION

Determination of X-ray Elastic Constant and Residual stress
Figure 5 shows \(2\theta-\sin^2\Psi\) curves obtained for the specimen under applied strains of \(\varepsilon_a=0, 200\) and \(400\times10^{-6}\). Three curves can be approximated by straight lines. The slope M of \(2\theta-\sin^2\Psi\) diagrams was determined by a least square regression program. The relation between M and \(\varepsilon_a\) is shown in Figure 6. The X-ray stress constant H was determined from Figure 5 by using the following equation.

\[
H = \frac{E_M}{\partial M / \partial \varepsilon_a} \quad (1)
\]

By using \(H=-286.9\)MPa/deg, the residual stress \(\sigma_R\) is determined where \(E_M\) is the mechanical Young’s modulus the measurement of M through

\[
\sigma_R = H \cdot M \quad (2)
\]

The residual stress before fracture toughness test was about 80MPa.
Fracture Toughness Tests Results

Figure 7 shows the record of load versus crack opening displacement for crack and $\rho=0.50\text{mm}$. The relation is linear up to the point of the fracture. The fracture toughness, $K_\rho$, of bluntly notched specimen is plotted against the square root of notch-tip radius in Figure 8. As seen in the figure, the relationship between $K_\rho$ and notch-tip radius $\rho$ can be divided into three regions: region I where $\rho$ is smaller than the radius $\rho I$ (30$\mu$m), region II where $\rho$ is above $\rho I$ and below 1mm, and region III where $\rho$ is larger than 1mm. $K_\rho$ was constant is region I and III, where it increased linearly with $\sqrt{\rho}$ in region I. I approaches a constant value $K_\rho$, i.e., the fracture toughness of the pre-cracked specimen, as $\rho$ becomes smaller.

Plastic zone size

The plastic zone size, $\omega_y$, can be defined as the depth where the residual stress approaches to zero as shown in Figure 9. In Figure 10, the depth was plotted against the toughness divided by the bending strength $\sigma_b$, the value of $\omega_y$ was proportional to the square of $K_\rho/\sigma_b$, i.e.,

$$\omega_y = \alpha \left( \frac{K_\rho}{\sigma_b} \right)^2 \quad (3)$$

where $\alpha = 0.05$. According to our previous studies,[4] the $\alpha$ value was 0.07 for Si$_3$N$_4$ ceramics. Equation(3) was extremely important for determining fracture toughness of silicon nitride ceramics from the X-ray measurement of the residual stress near the fracture surface. Levy et al [5] derived $\alpha = 0.15$ on the basis of the elastic-plastic finite element method for elastic perfectly plastic material. The $\alpha$ value different from 0.15 is now assumed to be caused by the difference

Figure 7 Load vs crack opening displacement

Figure 8 Relation between fracture toughness value and square root of notch-tip radius
of the yield stress in the plastic zone from that in simple tension test. The bending stress in the plastic zone $\sigma_{B'}$ is evaluated. The bending stress in the plastic zone $\sigma_{B'}$ is evaluated from the following equation

$$\omega_y = 0.05 \left(\frac{K\rho}{\sigma_B}\right)^2 = \alpha \left(\frac{K\rho}{\sigma_B}\right)_B^2$$  \hspace{1cm} (4)$$

$$\sigma_{B'} = \left(\frac{0.05}{\alpha}\right)^{1/2} \cdot \sigma_B$$  \hspace{1cm} (5)$$

From the previously published data [6] of $\alpha$ measured for the fracture surface of various steel and ceramics, the value of $\sigma_{B'}$ was calculated by using equation(5) and correlated to $\sigma_B$ in Figure 11. The following linear relation is obtained between $\sigma_{B'}$ and $\sigma_B$:

$$\sigma_{B'} = -54 + 1.5 \sigma_B$$  \hspace{1cm} (6)$$

The result of the present study agrees with this equation. From equations(5) and (6), the $\alpha$ value is given as a function of $\sigma_B$.

$$\alpha = 0.05 \left(\frac{\sigma_B}{-54 + 1.5 \sigma_B}\right)_B^2$$  \hspace{1cm} (7)$$

In the analysis of failure accidents, the apparent stress intensify factor can be determined from the measurement of the plastic zone by using $\alpha$ obtained from equation(7).

![Figure 9 Residual stress distribution near fracture surface.](image)

![Figure 10 Relation between plastic zone depth and stress intensity factor devided by bending strength.](image)

![Figure 11 Yield stress determined from plastic zone size.](image)
CONCLUSIONS

The main results obtained in the present study are summarized as follows:

① The relation between the fracture toughness $K\rho$ and notch-tip radius $\rho$ can be divided into three regions.

② The residual stress on the fracture surface was compression. The compressive residual stress slightly increased and then gradually diminished at a certain depth. The residual stress become almost constant.

③ The plastic zone size $\omega_y$ was determined. It is related to the stress intensity factor $K\rho$ as

$$\omega_y = \alpha \left( \frac{K\rho}{\sigma_B} \right)^2$$

where $\sigma_Y$ is the yield stress and $\alpha = 0.05$. The published data on the $\alpha$-value determined for various kinds of steel are expressed by the following function of $\sigma_B$,

$$\alpha = 0.05 \left\{ \frac{\sigma_B}{(1.5\sigma_B - 54)} \right\}^2$$

The difference in $\alpha$ between the materials was used to evaluate the actual yield strength of the material in plastic zone around the crack tip.

REFERENCES