AE Monitoring of Microdamages in Bioceramics for Artificial Joints under Simulated Body Environment*

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Microfracture process of 3 mol% yttria stabilized zirconia (3Y-TZP) for artificial joints was evaluated using the acoustic emission technique. In order to investigate the effects of environment and strain rate on the microfracture process, four point bending tests were carried out in air and physiological saline (P.S.) at various loading rates. From the results of AE behavior, rapid AE increasing point was observed before the final unstable fracture. It was suggested from the previous work that the AE increasing point corresponds to the maincrack formation. The critical stress for maincrack formation, \( \sigma_C \), was determined from the bending stress at the AE increasing point. The critical stress as well as bending strength, \( \sigma_B \), decreased in physiological saline. In particular, the decrease in critical stress was remarkable. It was then understood that stress corrosion cracking (SCC) by water in physiological saline affected maincrack formation rather than the final fracture. Consequently, it was suggested that the evaluation of \( \sigma_C \) is essential for the reliability assessment of bioceramics.

**Key Words:** Zirconia, Bending Strength, Acoustic Emission, Microfracture Process, Maincrack Formation

1. Introduction

Bioceramics such as alumina and zirconia have been recently used for artificial joints because of the excellent biocompatibility \(^{(1)}\)–\(^{(3)}\). It is expected that the performance of artificial joints can be maintained for longer than 10 to 15 years. However, it has been reported that ceramic femoral heads had broken in a few years after the replacement. Thus, it should be recognized that the establishment of the technique to predict the fatigue strength and lifetime is required for the assessment of the long-term reliability of bioceramics.

On the other hand, the authors have investigated the microfracture process during bending tests of alumina ceramics using AE technique \(^{(4)}\), \(^{(5)}\). It is important that the rapid increase in AE energy was observed before the final fracture. It was confirmed that the location of AE signals were concentrated at that of final fracture after the rapid increase in AE energy, and the maincrack formation was observed using fluorescent dye penetrant technique. It was then understood that the microfracture process consists of microdamage accumulation, maincrack formation and its growth to final fracture. It was also suggested that \( \sigma_C \) could be significant in the fabrication, design and service of bioceramics.

Recently, partially stabilized zirconia has been increasingly used for artificial joints because of fully high strength and toughness due to the stress-induced phase transformation. The objective of the present study is to evaluate the microfracture process in zirconia during bending tests under simulated body environment using AE method. Four point bending tests were carried out in air and physiological saline under the various strain rates to investigate the dependence of microfracture process on environment and strain rate.

2. Experimental Methods

2.1 Specimens

The material used was 3 mol% yttria stabilized tetragonal zirconia polycrystal, 3Y-TZP, (Japan Medical Material Co., Japan) actually used for artificial joints. The mechanical properties are presented in Table 1. The values of density, Young’s Modulus and Poisson’s ratio were
given by the manufacturer, and the fracture toughness was measured by IF method according to JIS R 1607. The specimens and microstructure of the material are shown in Fig.1. The average grain size was 0.2 \( \mu \)m. It can be seen that the grain size distribution is quite uniform.

The size of specimen was 3 \( \times \) 4 \( \times \) 40 mm according to JIS R 1601. The tensile surface as well as chamfered edges were polished using 3 and 1 \( \mu \)m diamond slurry to remove the surface flaw. Specimens were then dried in a vacuum drying oven at 150\(^\circ\)C for 2 h in order to prevent the stress corrosion cracking due to water.

### 2.2 Bending tests

Figures 2 and 3 show the schematic diagram of the testing systems in air and physiological saline, respectively. Four point bending tests were conducted with an inner span of 10 mm and an outer span of 30 mm according to JIS R 1601 in both environments. Loading rates were selected as 0.1 and 0.01 mm/min to investigate the effect of the strain rate on the strength. During the tests in physiological saline at low strain rate, temperature was maintained at 36.0±1.5\(^\circ\)C using silicon rubber heater with electromagnetic shielding. Moreover, specimens were soaked in physiological saline using a super sonic bath for 30 minutes before bending tests to fit them to testing environment and prevent AE noise due to air bubbles. The AE signal was detected by two AE sensors (resonant frequency, 200 kHz) attached on both ends of specimen, and amplified by pre-amplifiers. Total gain was 80 dB, threshold level was 18 \( \mu \)V at the input terminal of pre-amplifiers, and the band pass filter was used with a range of 100 to 500 kHz.

### 3. Results and Discussion

#### 3.1 Bending tests and AE behavior

The microfracture process during bending tests was monitored using an AE technique. Figure 4 shows the bending stress and AE behavior in air at the loading rate of 0.1 mm/min. It was recognized that the rapid increasing point of AE energy is also observed in zirconia at 590 s, and the specimen fractured at 940 s. It is suggested from the previous work\(^{(4),(5)}\) that the main crack was formed at this point, and the critical stress for main crack formation \( \sigma_C \) can be determined from the bending stress at the increasing point.

Weibull plots of bending strength, \( \sigma_B \), and the critical stress for main crack formation, \( \sigma_C \), obtained for each environment at 0.1 mm/min are shown in Fig.5. It is worth noting that the specimen with high fracture strength does not always have high critical stress for main crack formation, although the difference between those values at higher probability is smaller.

Average values of \( \sigma_B \) and \( \sigma_C \) for each environment are summarized in Table 2. Due to the stress corrosion cracking by water in physiological saline, \( \sigma_B \) was 15% lower in physiological saline than in air, on the other hand, \( \sigma_C \) was 45% lower. Therefore, it is understood that the testing environment more significantly affects the main-

**Table 1** Mechanical properties

| Density | Young’s Modulus | Poisson’s Ratio | Fracture Toughness(IF) |
|---------|-----------------|----------------|-----------------------|
| [g/cm\(^3\)] | [GPa] | [-] | [MPa·m\(^{1/2}\)] |
| 6.08 | 210 | 0.31 | 8.7 |

Fig. 1 Specimens and microstructure of material

Fig. 2 Schematic diagram of the testing system in air

Fig. 3 Schematic diagram of the testing system in P.S.

Fig. 4 Bending stress and AE behavior in air at 0.1 mm/min
crack formation.

**3.2 Effect of strain rate on strength**

Figure 6 shows the dependence of strength on strain rate. It can be seen in the result of tests in air that the lower the strain rate was, the lower the bending strength was. The critical stress for main crack formation also slightly decreased in physiological saline at low strain rate, due to the stress corrosion cracking by water in physiological saline.

![Graph showing the dependence of strength on strain rate.](image)

**Fig. 5** Weibull plots at 0.1 mm/min

**Table 2** Average values of $\sigma_B$ and $\sigma_C$ in each environment

| Environment | $\sigma_B$ (MPa) | $\sigma_C$ (MPa) |
|-------------|-----------------|-----------------|
| In air      | 1450 (100%)     | 1240 (85%)      |
| In P.S.     | 720 (100%)       | 400 (55%)       |

**3.3 Fracture process**

Fracture process was investigated by the observation of crack path and fracture surfaces using a scanning electron microscopy. Figure 7 shows the SEM micrograph

![SEM micrograph of crack branching and crack deflection.](image)

**Fig. 7** SEM micrograph of crack branching and crack deflection

**Fig. 8** SEM fractographs

(a) In air at 0.1 mm/min  
(b) In P.S. at 0.1 mm/min  
(c) In air at 0.01 mm/min  
(d) In P.S. at 0.01 mm/min
of tensile surface (in air at 0.1 mm/min). Several crack branchings as indicated by circles and crack deflections, which might be resulted from the phase transformation toughening, were observed.

The fracture surfaces and fracture origins in each environment at each strain rate are shown in Fig. 8. Crack propagation process was then examined based on fracture mechanics. From the figure, the fracture origin and the shape of crack at the onset of unstable growth to final failure can be identified. Both are approximated by semi-ellipsoidal surface crack. Then, the stress intensity factor $K_I$ can be calculated by (6),

$$K_I = 1.1\sigma \sqrt{\frac{\pi c}{Q}}$$  \hspace{1cm} (1)

where $c$ is a crack depth, $\sigma$ is the applied stress and $Q$ is a flaw shape parameter (see Fig. 9). It is considered that the fracture origin was initial maincrack subjected to the uniform stress of the critical stress for maincrack formation, $\sigma_C$, while the maincrack grew to the final unstable fracture under the uniform stress of bending stress, $\sigma_B$. Therefore, the critical stress intensity factor for the semi-ellipsoidal crack at fracture origin, $K_{I_{m}}$, and for the final unstable fracture, $K_{I_{f}}$, could be calculated using Eq. (1). The values of $K_{I_{m}}$ and $K_{I_{f}}$ are tabulated in Table 3. The critical stress intensity factor increased from $K_{I_{m}} = 0.60 – 3.90$ to $K_{I_{f}} = 20.0 – 22.0$ MPa·m$^{1/2}$ as cracks grew from 1.5 – 24 to 80 – 150 µm, which might be contributed by R-curve behavior due to the toughening mechanisms by stress-induced phase transformation as well as crack deflection and crack branching.

4. Conclusions

Four point bending tests were carried out in air and physiological saline at various strain rates, and microfracture process was monitored using AE technique. Furthermore, based on fracture mechanics, crack propagation process was examined from the SEM fractography. Consequently, following conclusions were obtained.

(1) Bending strength, $\sigma_B$, and the critical stress for maincrack formation, $\sigma_C$, decreased in physiological saline comparing with air. Since the decrease in $\sigma_C$ was larger, it was understood that testing environment significantly affects the maincrack formation.

(2) $\sigma_B$ as well as $\sigma_C$ decreased with the decrease in strain rate.

(3) The stable crack growth followed by unstable growth was observed on the fractured surfaces. R-curve behavior was also suggested by fracture mechanical analysis.

It was strongly suggested that the critical stress for maincrack formation can be the design and evaluation parameter of bioceramics and the prediction of fatigue strength and lifetime would be enabled based on the concept of $\sigma_C$ and R-curve behavior.

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