SURFACE FRACTURE TESTING ON ALUMINA CERAMICS WITH SPHERICAL INDENTER AND INSTRUMENTED HARDNESS TESTER

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SUMMARY
Two kinds of mechanical tests were conducted for discussing the surface fracture on alumina ceramics. In spherical indentation test, the size of the circular (Hertz) crack was discussed. The diameter decreased with increasing the average grain size of alumina, but the decreasing ratio of the circular crack diameter was smaller than that of the bending strength. We could not calculate the surface stress for forming the circular crack, as the measured diameter was larger than the calculated values. In instrumented hardness test, we discussed energy consumption for forming indents. The energy consumption ratio was independent of the grain size on alumina ceramics, but this ratio was smaller on alumina single crystal. It can be related to the grain boundary fracture on the ceramics. The results were not different between the tests with Vickers indenter and with Knoop indenter.

Keywords: ceramics, alumina, hardness, Hertz crack, grain size

1 INTRODUCTION
For understanding the wear properties on ceramic materials, it is important to evaluate the mechanical properties on the surface of ceramics [1, 2]. In this study, we used spherical indentation test and instrumented hardness test for understanding the mechanical properties on the surface of alumina ceramics. For discussing the surface fracture, spherical indentation test was carried out, and the size of the circular (Hertz) crack was measured. In the instrumented hardness test, the energy for forming an indent on the surface was measured. We prepared some alumina ceramics with different average grain size, and the grain size dependence of the result was discussed.

2 EXPERIMENTS
2.1 Specimen
Commercially supplied alumina sintered body (Type LX05 by Toshiba Tangaloy Co.) was tested [3]. The average grain size was changed with annealing this sample in air, under different temperature and annealing time. Four kinds of specimens with different average grain size were prepared. Table 1 shows the average grain size and the bending strength of each specimen.

<table>
<thead>
<tr>
<th>Grain size (µm)</th>
<th>Bending strength (MPa)</th>
<th>S.D.</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.1</td>
<td>652</td>
<td>39</td>
</tr>
<tr>
<td>1.8</td>
<td>555</td>
<td>40</td>
</tr>
<tr>
<td>4.4</td>
<td>415</td>
<td>23</td>
</tr>
<tr>
<td>5.7</td>
<td>369</td>
<td>21</td>
</tr>
</tbody>
</table>

Table 1: Average grain size, bending strength and its standard deviation of four alumina specimens

2.2 Spherical indentation test
The schematic figure for spherical indentation test is shown in Figure 1. A bearing ball with 2mm diameter made of silicon nitride was used for the indenter. A Rockwell hardness tester was used for applying the test force. The applied test forces were 441, 588, and 980 N. If the applied force was too small, we could not observe the Hertz crack, as the indenter contacted elastically. If the applied force was too large, more than two circular cracks were observed. In the case we could obtain one circular crack, the diameter of the circular crack was measured.

The measured diameters of the circular cracks were discussed with the following analysis. From the Hertz analysis, the radius of the contact circle, a, and the maximum tensile stress on the surface, \( \sigma \), can be calculated with these equations:

\[
a = \left( \frac{4kr}{3} \right)^{1/3} F^{1/3} \quad (1)
\]

\[
k = \left( \frac{9}{16} \right) \left[ (1-v^2) + (1-v'^2) \frac{E'}{E} \right] \quad (2)
\]

\[
\sigma = \frac{1}{2} \frac{F}{ \pi a^2} \quad (3)
\]

Figure 1: Spherical indentation test and the parameters

where, \( r \): radius of contact ball, \( E \), \( v \): Young's modulus and Poisson's ratio of flat specimen, \( E' \), \( v' \): Young's modulus and Poisson's ratio of contact ball, \( F \): contact force.

These equations indicate that the diameter of the crack is proportional to the surface fracture stress, as the radius of the contact circle is proportional to the surface tensile stress.
stress. It means that we can compare the crack diameter and the strength directly.

For discussing the surface fracture, fracture toughness test in indentation fracture (IF) method was conducted. We measured the median crack length in Vickers indentation, and calculated the fracture toughness [4]. We conducted single edge pre-cracked beam (SEPB) method for obtaining the fracture toughness values for the comparison.

### 2.3 Instrumented hardness test

The relation between the applied load and the indenter penetration displacement was measured, using instrumented hardness tester (Type MZT-4, Akashi Co.) with Vickers or Knoop indenter made of diamond. The energy for forming the indent was calculated from the loading-unloading curve. Figure 2 shows a typical example of the obtained result. From this figure, we could calculate the consumed energy for forming indent during the hardness test.

We estimated that the ratio of the consumed energy to the total deformation energy during the test could indicate the surface fracture, as if only the elastic deformation happens, this consumed energy ratio becomes zero. We changed the maximum test load from 9.8 mN to 980 mN with 7 levels, and obtained the energy consumption ratio, explained in Figure 2. We also carried out instrumented hardness test on alumina single crystal (sapphire) specimen to obtain data for comparison.

3 RESULTS

#### 3.1 Spherical indentation test

Figure 3 shows the average grain size dependence of the Hertz crack diameter and the bending strength on alumina ceramics. Both of the crack diameter and the bending strength decreased with increasing the average grain size. However, the decreasing ratio was smaller on the result of crack diameter.

![Figure 3: Grain size dependence of the Hertz crack diameter and the bending strength on alumina ceramics.](image)

Figure 4 shows the grain size dependence of the fracture toughness measured in IF and SEPB method. Both results show that the measured fracture toughness increased with increasing the average grain size.

#### 3.2 Instrumented hardness test

Figure 5 shows the relation between maximum test force and the energy consumption ratio on the instrumented hardness test with Vickers indenter. In some test conditions on small test force, the energy consumption ratio became smaller. However the scattering of the data is large, so we cannot discuss the test force dependence of the energy consumption ratio. We could not see any grain size dependence of the energy consumption ratio, but the energy consumption ratio was smaller on alumina single crystal (Sap in Figure 5).
We used Knoop indenter for the instrumented hardness test in same test conditions. We expected that the elastic recovery is more likely to occur with Knoop indenter, because the Knoop indenter has the blunt tip angle. However, the results were not so different from that with Vickers indenter (Figure 6).

Figure 5: The relation between the maximum test force and the energy consumption ratio for forming the indent in instrumented hardness test with Vickers indenter, on different grain size specimens; '1.1', '1.8', '4.4' and '5.7' mean the average grain size of tested alumina, in µm, and 'Sap' means the data on alumina single crystal (sapphire).

Figure 6: The relation between the maximum test force and the energy consumption ratio for forming the indent in instrumented hardness test with Knoop indenter, on different grain size specimens; '1.1', '1.8', '4.4' and '5.7' mean the average grain size of tested alumina, in µm, and 'Sap' means the data on alumina single crystal (sapphire).

4 DISCUSSION

4.1 Spherical indentation test

The surface fracture measured in spherical indentation showed different grain size dependence from bending strength, and fracture toughness. The bending strength is controlled by the defect size in the ceramics, but the surface fracture is less influenced by the defects in the materials.

The fracture toughness in IF method is similar to the result of macroscopic fracture in SEPB method. It increased with increasing the grain size. We expected that the results form IF method can indicate the property of surface fracture, as it measures the length of the median cracks on the surface. However, the crack length in this method was around 0.2 mm, which is long enough compare to the grain size. In this reason, we estimated the result by IF method is dominated by the macroscopic fracture property of the specimen.

We tried to calculate the surface stress for circular crack with the Hertz analysis. The calculated diameter of the contact circle was 0.268 mm on the test force of 588 N, and 0.244 mm on 441 N. The measured diameter was larger than these values, as shown in Figure 3. We could not make good explanation for this difference. We need some computational analysis for discussing this difference.

4.2 Instrumented hardness test

In instrumented hardness test, we expected that the energy consumption ratio becomes smaller in the tests with smaller maximum test force. If we use a small enough test force, the indenter can be supported by only the elastic deformation of the specimen, and the energy consumption becomes zero. However, we could not observe the test force dependence of the energy consumption clearly, on both of the results obtained with Vickers indenter and Knoop indenter. We shall use a spherical indenter for obtaining this elastic/plastic limit of these specimens.

In the case of circular crack test, we could see the grain size dependence of the crack diameter, but in instrumented hardness test, we could not see the grain size dependence. In the former test, the fracture mode is in tension but in the latter test, the compressive stress was applied on the specimen. This difference of the stress mode can be related to the difference of the grain size dependence, as the fracture of ceramics in tension and in compression are different.

The energy consumption ratio did not depend on the grain size of alumina ceramics, but this ratio was smaller on alumina single crystal. The formation of indent on ceramics needs some grain boundary fracture, so it requires additional energy for forming indents, compare to the single crystal.

5 CONCLUSIONS

Spherical indentation test and instrumented hardness test were carried out on alumina ceramics, for understanding the mechanical properties on the surface. In the spherical indentation test, the influence of the average grain size on the surface cracking stress is less than on the bending strength, because the surface fracture is not controlled by the size of the defects in the ceramics. Indentation fracture test was used for determining the
fracture toughness on the surface, but we estimated the obtained values represent the fracture toughness of the bulk material. In the instrumented hardness test, we could not see the grain size dependence of the energy consumption for forming indents during the test. The energy consumption became smaller on single crystal specimen than on ceramic specimen. The fracture at the grain boundary could consume the energy for forming the indents.

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7 REFERENCES


