RELATIONSHIP BETWEEN GRAIN SIZE AND STRENGTH FOR 3Y-TZP CERAMICS

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It is put forward in this paper that the relationship of the grain size and the strength is out of accord with that of Hall-Petch relationship when the grain size is below a micron(<1μm) for ZrO_2 or TZP ceramics. Especially in the nanocrystalline, the smaller the grain sizes were, the lower the strengths were. The relationship of the grain size and the flexural strength found here accords with the following equation:

$\sigma_b = aD^{-b/D}$

Where σ_b is the flexural strength and D is the grain size, a and b are constants of materials.

INTRODUCTION

It is well known for metals that the Hall-Petch(1)'s equation represented well the relationship of the yield stress and the grain size; this equation is as follows:

$$\sigma_y = \sigma_i + K_y d^{-1/2} \tag{1}$$

The yield stress of a material is inversly proportional to the square root of the grain size.

Rice(2) summarised a large amount of works which had been published, and found that the fracture strengths of ceramics were also inversly proportional to the

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square root of the grain size, completely similar to the equation of Hall-Petch. Oxide ceramics with ionic bonds, nitride ceramics with covalent bonds, and also phosphatic ceramics accorded with as follows:

$$\sigma_f = \sigma_0 + Kd^{-1/2} \tag{2}$$

The fine grain can increase the strength of metal. This is because the grain bondaries hinder plastic deformation. But below micron grain size in the structural ceramics, it was found by Zheng et al(3-5) that was out of accord with the equation (2).

However, the unterstanding of the relationsahip of fracture strength and grain size is still rather lacking so far. This requires systematical research. So, the objective of this paper is to investigate the relationship between the flexural fracture strength and the grain size which were from the nanometer(100 nm) to the micron(1.33 μm) size at room temperature for both the hot-pressed and the pressureless sintered 3Y-TZP ceramics.

EXPERIMENTAL PROCEDURE

Nanopowder

The superfine powder was prepared by coprecipitation method with zirconium oxychloride and yttria nitrate as starting materials, with ammonia as the precipitant treatment. The specific surface area of power is 32.94m^2/g by the BET methods of nitrogen adsorption. TEM(JEM-200CX) was used to observe the size and morphology of first-order particles, the average 20nm sized superfine power(particles) were obtained.

3Y-TZP Materials

3Y-TZP ceramics were obtained by both the pressureless sintering and hot-pressing the 20 nm sized 3Y-TZP powder. Densities were measured by the Archimedes method, and the average grain size, by the line intercept method by SEM, TEM, especially in the nanocrystalline(<120 nm), with the aid of a AFM. The mechanical properties were obtained from the average of measurement data for three specimens from each.

It was known by x-ray diffraction that almost 100% tetragonal phase was obtained at room temperature.

Specimens

The specimen dimension was 3mm × 4mm × 36mm for the three-point bending test. The testing surfaces of the specimens were polished down to a 1μm diamond paste finish and the four long edges of each sample were rounded.

Bending Test

Load-controlled static three-point bending tests were conducted on a computer-controlled 100KN Instron tensile testing machine at room temperature.

RESULTS

Relationship of Bending Strength and Grain Size for Structural Ceramics

Fig.1 showed that the flexural strength increased with increasing both the hot-pressed and the pressureless-sintered grain sizes for 3Y-TZP ceramics. The experimental data of the flexural strength and the grain size were drawn up, and the result was as follows:

$$\sigma_b = aD^{-b/D} \tag{3}$$

σ_b is the flexural atrength; D is the grain size; a and b are the constants for the material. The experimental data were treated by minimal two-multiplication, and the results were obtained as follows:

$a_1 = 1860$; $b_1 = 31.25$ (Hot-Pressed)
$a_2 = 937.4$; $b_2 = 20.8$ (Pressureless-Sintered)

It was found that the fitted curve for the pressureless sintered 3Y-TZP ceramics was accorded better with the experimental data than that of the hot-pressed(see Fig.1). The average error range between the experimental data and the fitting curve was 4.5% for the pressureless sintered materials; 6.1% for the hot-pressed one.

Both Fig.1 and equation (3) showed that the σ_b would trend to a certain value with increasing grain size. This is also in accordance with reality, because any material has the maximum value of its strength.

Influence of Density

Another important influence was the density of the materials for the strength of structural ceramics, this is the characteristic of ceramic materials that differs from metals. So, the strengths of ceramics were controlled by both the grain sizes and the densities. The relationship of the grain size, the density and the strength was drawn in the three-dimensional diagram(see Fig.2)

The strength of ceramics depends strongly on its pore size and content. When the densities were below 99% of the theoretical density, the strengths increased exponentially with increasing the grain size from 100 nm to 200 nm (see Fig.2). While the densities were above 99.9% of that, the strengths increased linearly with increasing the grain size(above 600 nm) for both the hot-pressed and the pressureless-sintered 3Y-TZP ceramics.

DISCUSSION

Relationship of Strength and Grain size, Density

The flexural strengths depended strongly on the density when below 99% of the theoretical density; on the grain size, above the 99.9% of that. So, the strengths of ceramics materials are controlled by two factors, namely, the grain size and the density.

The pores gave rise to stress concentration, in turn, resulting in a marked drop in the strength. The smaller the grain size was, the larger the volume fraction of grain boundary was, and the larger the degree of disorder was. So, it was possible for the pore content to be increased at the grain boundaries of nanocrystalline(100 nm or so), so that its strengths were lower in the pressureless sintered ceramic materials.

The larger the grain sizes were, the easier the transformation from tetragonal(t) to monoclinic(m), the larger the toughening effect was and the higher the strength became for 3Y-TZP ceramics.

Defect Types

The strengths of the hot-pressed 3Y-TZP ceramics were markedly higher than that of the pressureless-sintered (see Fig. 1 and 2). This was because the different sintering methods resulted in the different defect types. The main internal defects were the microcracks along the grain boundaries for the hot-pressed 3Y-TZP ceramics, and the pores along the grain boundaries and at the joints of poly-grains in the pressureless sintered ceramic. Due to the pore-induced stress concentration, these were more fatal to failure than the microncracks. This was why the strengths of the pressureless sintered ceramics were far lower than those of the hot-pressed.

CONCLUSION

The relationship between the flexural strength and the grain size and density at room temperature for hot-pressed and pressureless sintered 3Y-TZP ceramics was investigated.

1. The flexural strengths increased with increasing grain size from 100 nm to 1.33 μm. The relationship of the flexural strength and the grain size was concluded as follows:

$$\sigma_b = aD^{-b/D}$$

σ_b is the flexural atrength; D stands for the grain size; a and b are the constants for a material. The above relationship is the opposite to the Hall-Petch relationship. It is suitable for ZrO_2 and TZP ceramics.

2. The strength was controlled by the density when below 99% of the theoretical density; above it, by the grain size.

3. The strengths of the hot-pressed 3Y-TZP ceramics were markedly higher than that of the pressureless sintered. This is because the pores produced in pressureless sintering gave rise to a stress concentration, and resulted in the drop in strength. So, the pores were more fatal to failure than the microcracks.

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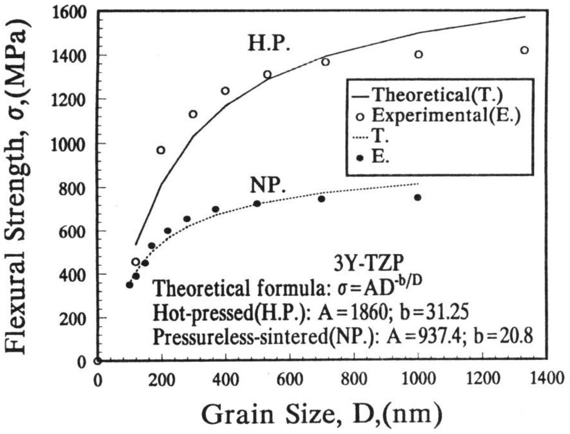

Fig. 1 The relationship between flexural strength and grain size at room temperature for both the hot-pressed and the pressureless sintered 3Y-TZP ceramics.

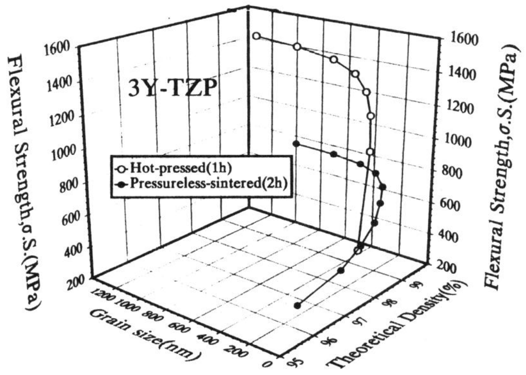

Fig. 2 The relationship between flexural strength and grain size as well as density at room temperature for both the hot-pressed and the pressureless sintered 3Y-TZP ceramics.