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Application News

No. i217

Dynamic Ultra Micro Hardness Tester

Hardness Test for Zirconia Particles

Introduction

Fracture strength in a compression test is generally used in strength evaluations of fine particles such as various types of industrial raw materials. Recently, however, there have also been requests for evaluation techniques of the hardness of particles.

As a test method, specimens for hardness evaluations are prepared by embedding the fine particles which are the object of measurement in resin and then polishing the specimen surface. This article introduces an example in which the hardness of zirconia powder was measured using a Shimadzu DUH™-211S dynamic ultra micro hardness tester.

Zirconia powder is now used in a diverse range of products, beginning with materials for dental treatment, taking advantage of its characteristic of high hardness.

Testing Apparatus and Specimens

The testing apparatus used in this experiment was a Shimadzu DUH-211S dynamic ultra micro hardness tester (Fig. 1 shows the appearance of a representative system). Two types of zirconia powder with different particle diameters (100 µm and 30 µm) were used in the evaluation. Samples were prepared by fixing the particles in an embedding resin and polishing the specimen surface. Table 1 shows the details of the samples used in the test.

Table 1 Test Samples

1) Sample name	Zirconia powder	
2) Sample symbol	A	B
3) Particle diameter (µm)	100	30
4) Sample dimensions (mm)	φ25 × 10 t (embedding resin)	

Fig. 1 Overview of DUH-211S Dynamic Ultra Micro Hardness Tester

Test Conditions

Table 2 shows the test conditions (loading jig and loading conditions). Loading was applied with a diamond triangular pyramid indenter having a tip angle of 115 degrees. Fig. 2 shows an image of the loading method.

Table 2 Test Conditions

1) Testing machine	DUH-211S dynamic ultra micro hardness tester (see Fig. 1)
2) Indenter	The diamond triangular pyramid indenter with tip angle 115 degrees
3) Type of test	Load-unload test
4) Test force (mN)	49
5) Loading rate (mN/s)	2.685
6) Holding time (s)	5

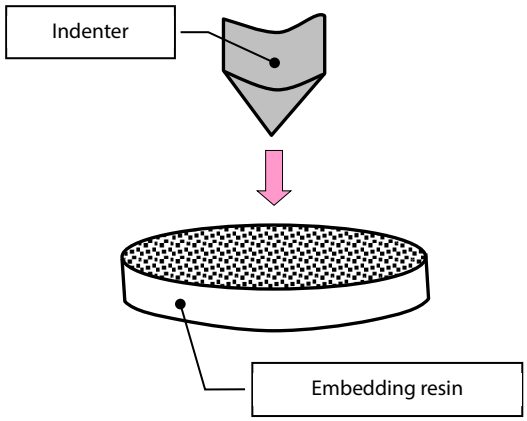


Fig. 2 Loading Image

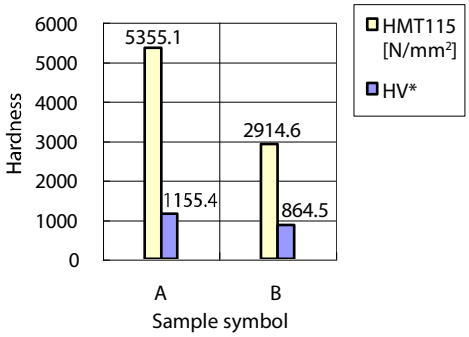
Test Results

Fig. 3 shows the results of the hardness measurements by a load-unload test conducted under the conditions shown on the previous section (average of the measured values at multiple points for both samples A and B). Fig. 4 shows the test force-indentation depth curves for representative measurement points.

Hardness test results (average values)					
Sample name	Sample symbol	Fmax [mN]	hmax [μm]	HMT115 [N/mm²]	HV*
Zirconia	A	49.37	0.543	5355.1	1155.4
	B	49.40	0.742	2914.6	864.5

Note 1) The symbols shown in the above table have the following meanings.
Fmax: Maximum test force
hmax: Maximum indentation depth
HMT115: Martens hardness obtained with a 115 degrees triangular pyramid indenter
HMT115 = Fmax / (26.43 × hmax²)
HMT115 is value which includes machine compliance correction (Cf correction) by the rigidity of the testing machine and indenter area function correction (Ap correction) for the influence of the roundness of the indenter tip.
HV*: Converted Vickers hardness (reference value)

From these results, it can be understood that sample A has a higher Martens hardness (HMT115) and a higher converted Vickers hardness (HV*) than sample B.



Sample symbol and hardness functions

Fig. 3 Test Results (Measured Values)

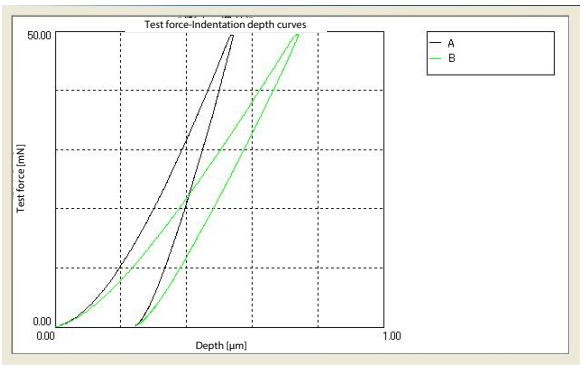
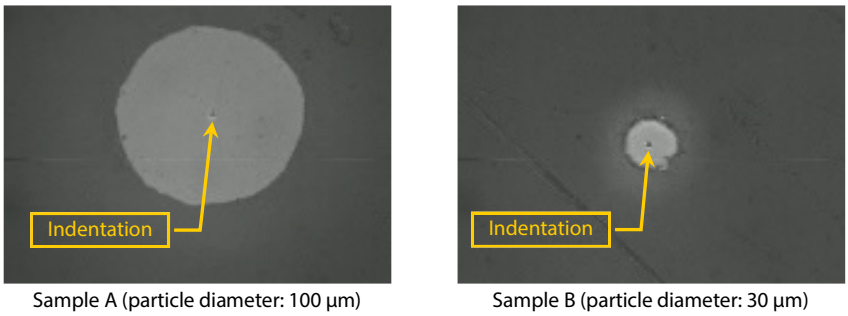


Fig. 4 Test Results (Test Force-Indentation Depth Curves)

Fig. 5 shows enlarged images of the indentations of the two samples after unloading, as photographed with a microscope.

If a hardness test is conducted with a sharp-tipped indenter under a condition in which fine particles are loosely distributed and not fixed, accurate measurement will be impossible due to scattering of the particles. However, as described in this example, it is possible to prepare samples with a polished fine and flat surface, even when testing fine particles, if the particles are embedded in resin and polished.

Because the DUH-211S dynamic ultra micro hardness tester is capable of measuring hardness by forming indentations with extremely low force while aiming at the center of the particles, it is an effective tool for testing fine particles, as shown in this experiment.



Sample A (particle diameter: 100 μm) Sample B (particle diameter: 30 μm)

Fig. 5 Results of Microscopic Observation of Test Specimens

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