TOSOH ZIRCONIA POWDER

TITLE: **SPECIFICATION AND TYPICAL PROPERTIES**

Grades: TZ-3Y-E, 3YS-E, 3YB-E, 3YSB-E, 3YSB-C

1. Standard Specification

		3Y-E	3YS-E	3YB-E	3YSB-E	3YSB-C
Chemical characteristics						
$ZrO_2+HfO_2+Y_2O_3+Al_2O_3$ *1	wt%	(>99.9)	(>99.9)	(>99.9)	(>99.9)	(>99.9)
Y_2O_3	wt%	5.15 ±0.20	5.15 ±0.20	5.15 ±0.20	5.15 ±0.20	5.15 ±0.20
Al_2O_3	wt%	0.25 ±0.10	0.25 ±0.10	0.25 ±0.10	0.25 ±0.10	0.25 ±0.10
SiO_2	wt%	≤0.02	≤0.02	≤0.02	≤0.02	≤0.02
Fe_2O_3	wt%	≤0.01	≤0.01	≤0.01	≤0.01	≤0.01
Na_2O	wt%	≤0.04	≤0.04	≤0.04	≤0.04	≤0.04
Loss on ignition (1000 °C)	wt%	≤1.2	≤1.2	3.6 ±0.6	3.3 ±0.6	5.5 ±1.0
Physical characteristic						
Specific surface area	m^2/g	16±3	7±2	(16±3)	(7±2)	(7±2)

*1: Calculated value ----- 100 - (SiO_2 + Fe_2O_3 + Na_2O)
Typical Hafnia content < 3.0 wt%.

Note: For binder grades, BET specific surface areas cannot be measured. We believe that a binder grade has the same BET value range as that of its base powder, for example, the range of BET value for TZ-3YB-E is the same as the range for TZ-3Y-E.

MODIFICATIONS

Valid August 2004

APPROVED BY

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Date: October 19, 2004

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2. Typical Properties

		3Y-E	3YS-E	3YB-E	3YSB-E	3YSB-C
Physical characteristics						
Crystallite size	nm	27	36	27	36	36
Particle size*1 D(50)	μm	0.6	0.6	(0.6)	(0.6)	(0.6)
Granule size*2 D(50)	μm	60	60	60	60	55
Bulk density	g/cm^3	1.3	1.2	1.1	1.2	1.35
Process characteristics*3						
Green density	g/cm^3	2.55	2.61	2.66	2.79	2.91
Sintered density	g/cm^3	6.05	6.05	6.05	6.05	6.05
Bending strength	MPa	1000	1500	1100	1400	1100
Fracture toughness	MPam$^{0.5}$	5	5	5	5	5
Hardness (HV10)		1250	1250	1250	1250	1250

*1: For typical particle size distributions, see Appendix-1.
*2: For typical granule size distributions, see Appendix-2.
*3: Values depend on process conditions. Above results are obtained by:
Uni-axial press
Molding pressure 70 MPa
Sintering Temp. TZ-3Y-E 1350 ^{o}C, 3YB-E & 3YS-E 1450 ^{o}C, 3YSB-E & 3YSB-C 1500 ^{o}C

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TITLE: **SPECIFICATION AND TYPICAL PROPERTIES**

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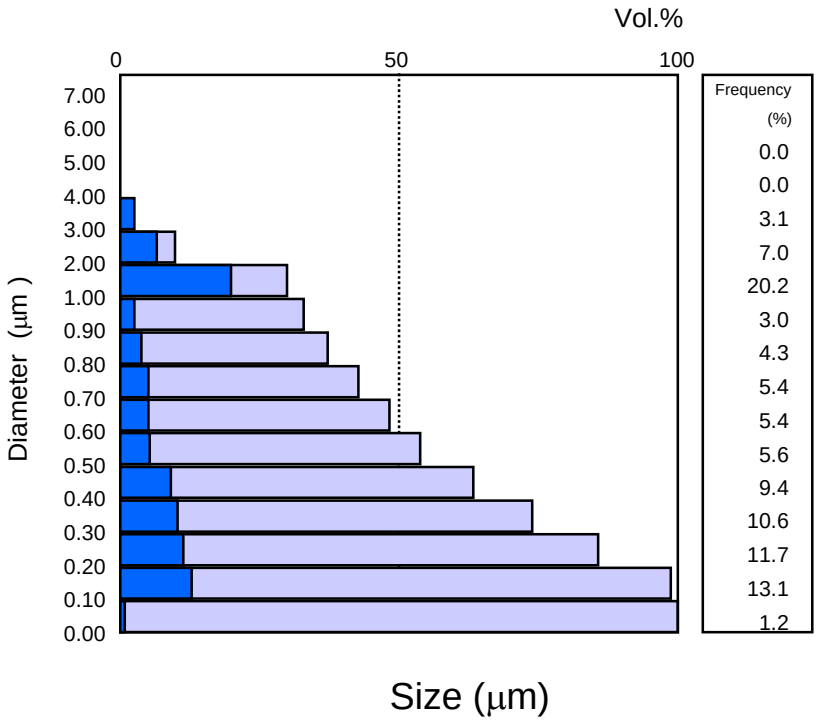
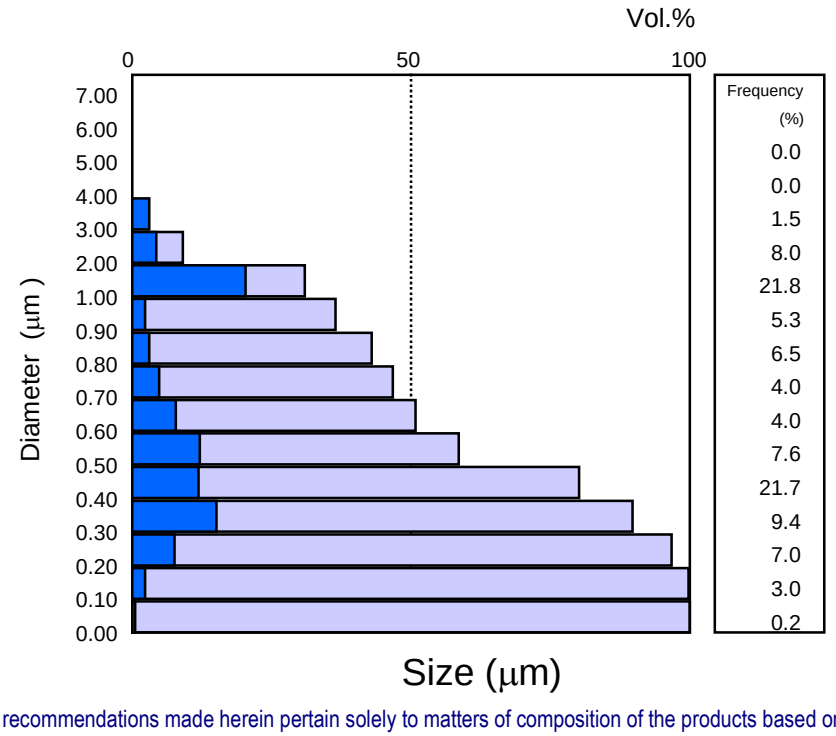
APPENDIX-1 Particle size distribution (typical)

TZ-3Y-E

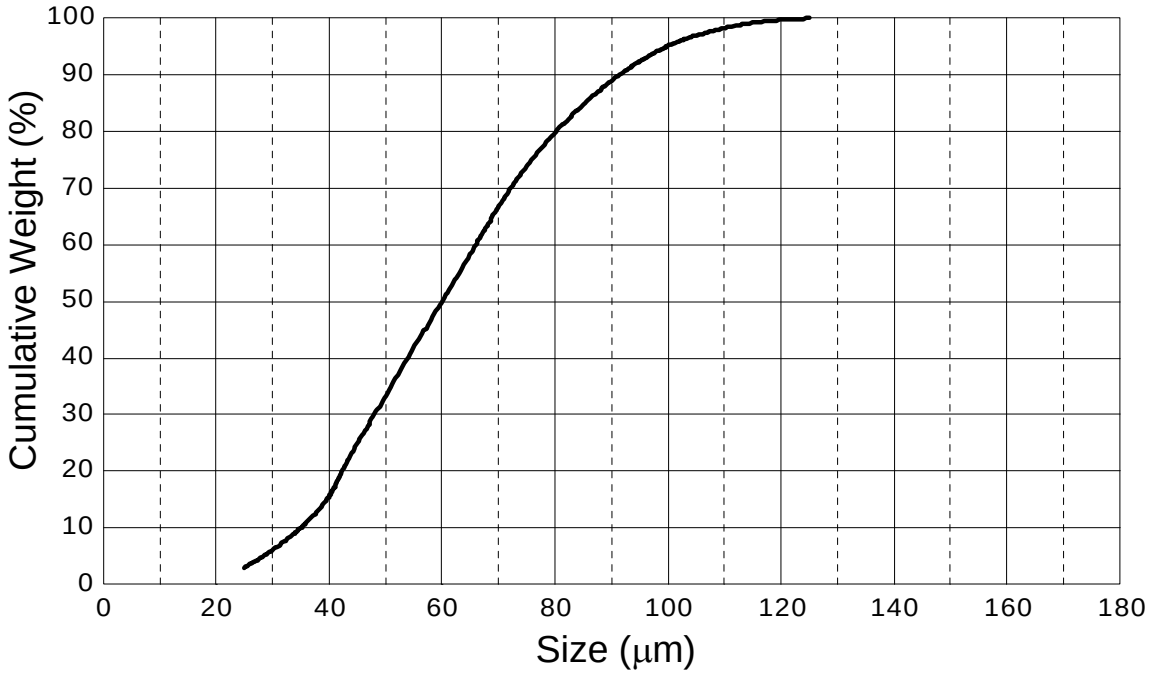


TZ-3YS-E

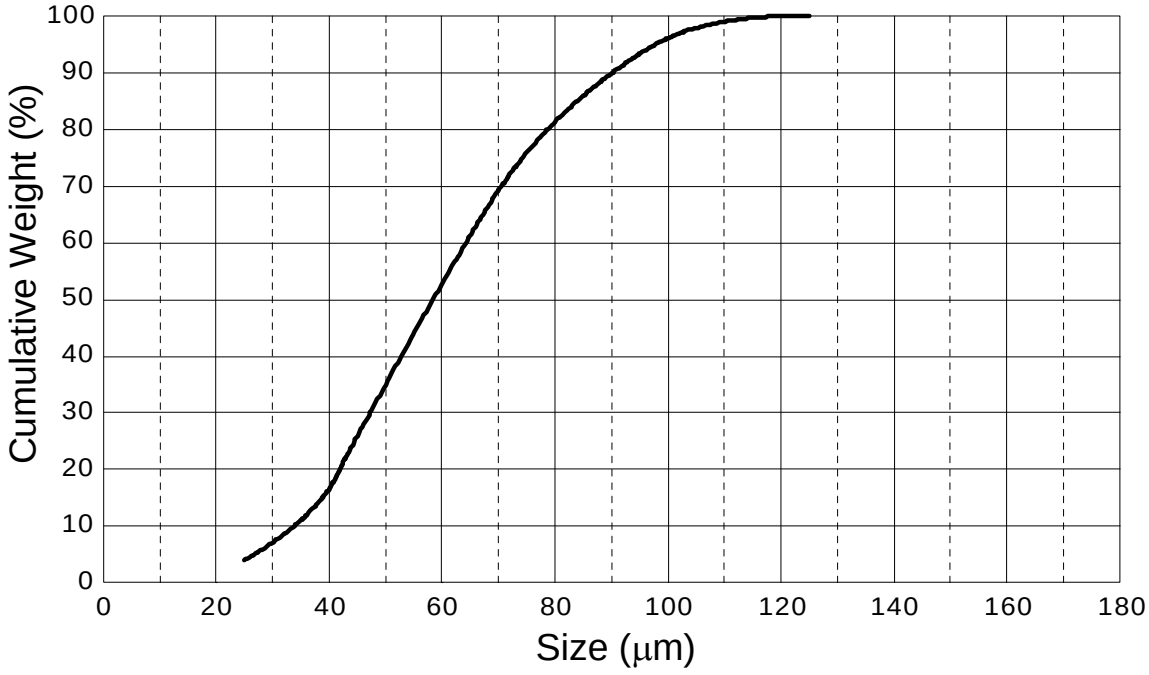


APPENDIX-2 Granule size distribution (typical)

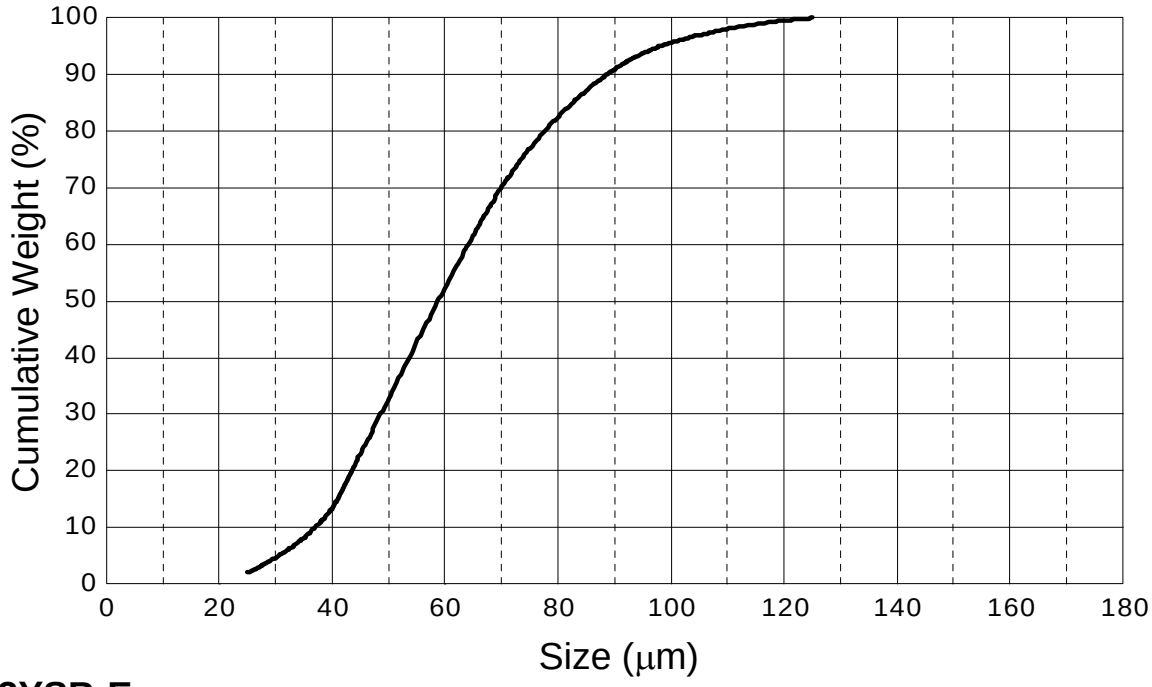
TZ-3Y-E



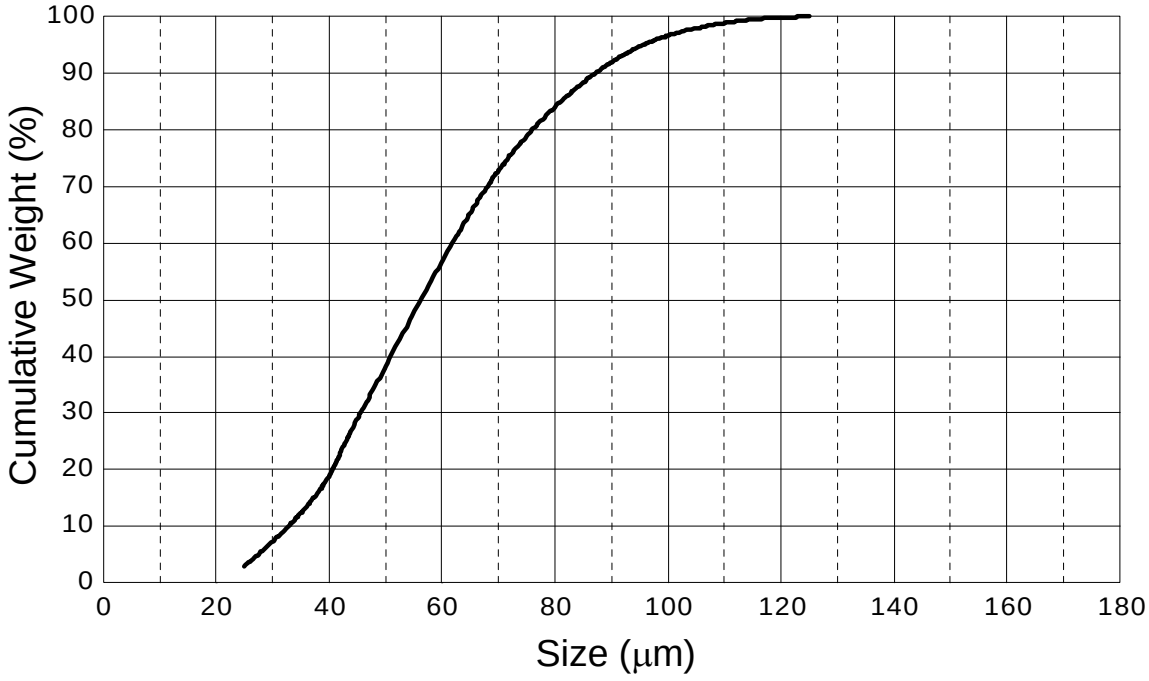
TZ-3YS-E



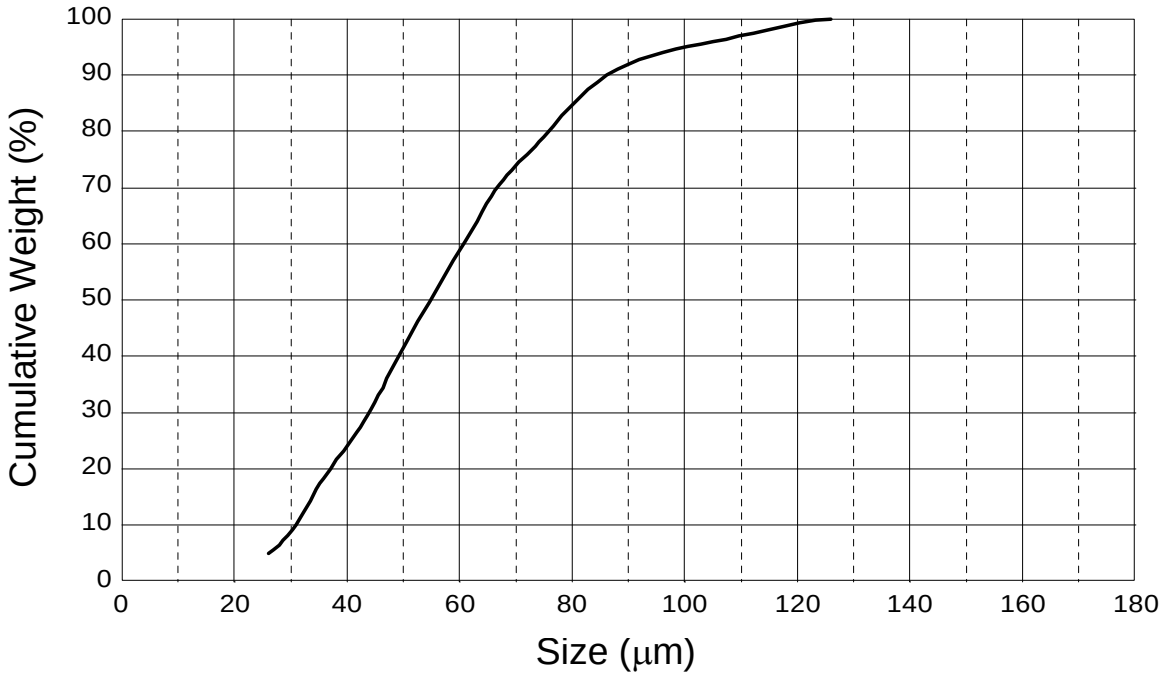
TZ-3YB-E



TZ-3YSB-E



TZ-3YSB-C



Quality of each lot is assured according to the following:

1. Chemical composition

Chemical composition is determined by the following methods:

Y_2O_3	X-ray fluorescence
Al_2O_3	Inductive Coupled Plasma
SiO_2	X-ray fluorescence
Fe_2O_3	X-ray fluorescence
Na_2O	Atomic absorption

2. Loss on ignition

A sample of 3 grams of powder is put into a crucible. The crucible is placed in an oven. After heating for 1 hour at 1000 ^{o}C, the weight loss is measured. Loss on ignition is calculated as a percentage of the weight of the sample.

3. Specific surface area (B.E.T.)

Equipment: TriStar 3000 (Micromeritics)*

A sample of 2 grams of powder is degassed under vacuum for 2 hours at 250 ^{o}C. After degassing, specific surface area is measured by the Nitrogen gas adsorption method (5 points), using the above equipment.

4. Crystallite size

Equipment: X-ray diffractometer (Rigaku)*
Crystallite size is calculated by the following numerical equation.
The reported value is determined by the average of 2 samples.

$$CS = \frac{91.435}{\sqrt{(HW/40)^2 - (0.1675)^2}}$$

CS: Crystallite size

HW: Half width of T(111)

5. Granule size and distribution

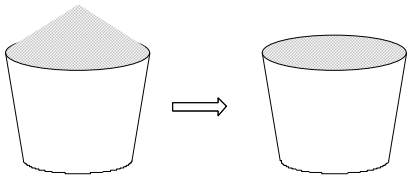
Equipment: Sieves (JIS Z-8801)
125 μm, 106 μm, 90 μm, 75 μm, 63 μm, 45 μm, 38 μm, 25 μm
Tapping machine

A sample of 50 grams of powder is put on the top sieve. After 30 minutes of tapping, the weight of the powder remaining on each sieve is measured. The results are entered in a graph showing granule size and cumulative weight. The granule size ($D_{(50)}$) is determined from the graph.

6. Bulk density

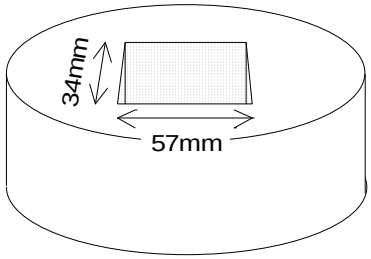
Equipment: Powder Characteristics Tester (HOSOKAWA MICRON)*
Powder is poured gently through a vibrating sieve (710 μm) into a measuring cup (100 cc) until the powder runs over. The excess powder is removed carefully from the cup by using a scraper.

The bulk density is calculated from the weight of the powder in the cup.



7. Green density

Equipment: Uni-axial press
Mold (shown at right)
Vernier calipers

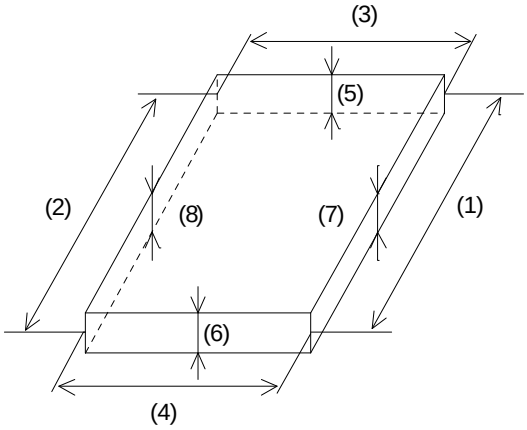


A sample of 25 grams of powder is put uniformly into the cavity of the mold.

Press the powder with 70 MPa pressure and hold the pressure for 30 seconds. From each powder batch, two green bodies are produced. The weight of each green body is measured.

Dimensions of the green bodies are measured using vernier calipers.

Length (1) and (2)
Width (3) and (4)
Thickness (5), (6), (7) and (8)

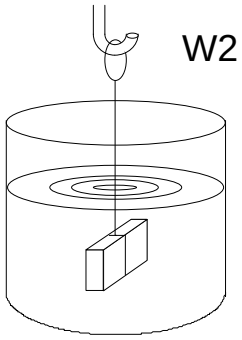


Green density is calculated by the following numerical expression.
Green density value is determined by the average of the 2 pieces.

$$G.D. = \frac{\text{weight}}{\frac{(1)+(2)}{2} \times \frac{(3)+(4)}{2} \times \frac{(5)+(6)+(7)+(8)}{4}} \times 1000$$

8. Sintered density

Equipment: Electric furnace
Precision balance



The above-mentioned green bodies are sintered at 1350 ^{o}C or 1450 ^{o}C or 1500 ^{o}C (heating rate: 100 ^{o}C/Hr, sintering temp.: 3Y-E 1350 ^{o}C, 3YB-E & 3YS-E 1450 ^{o}C, 3YSB-E & 3YSB-C 1500 ^{o}C, holding time : 2 hours).
The following weights are measured:

W1: Weight of sintered body at room temperature.
W2: Keep the sintered body in boiling water for 1 hour. After cooled down to R.T., the weight (W2) is measured in the water at R.T.
W4: After removing water from the surface, weight (W4) of the sintered body is measured.

Sintered density is calculated by the following numerical expression.
Sintered density value is determined by the average of the 2 pieces.

$$S.D. = \frac{W1 \times WDT^*}{W4 - W2}$$

*WDT: density of water at T ^{o}C

9. Bending strength (3-point bending test)

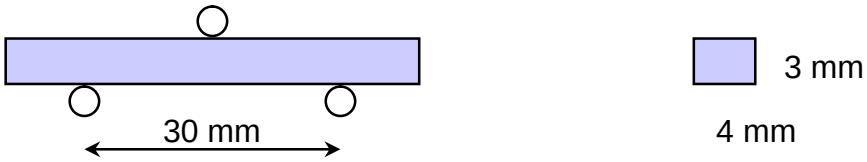
Equipment: Autograph DLS-R-500 (Shimadzu)*

Test pieces are made according to JIS R1601 with additional polishing by whetstone (#200) and chamfering by sandpaper (#400).

Size: 3 $^{\pm 0.1}$ x 4 $^{\pm 0.1}$ x 45 mm

Measurement details are in JIS R1601.

Cross head speed: 0.5 mm/min



The bending strength value is determined by an average of 10 samples.

10. Fracture toughness (Single Edge Precracked Beam method)

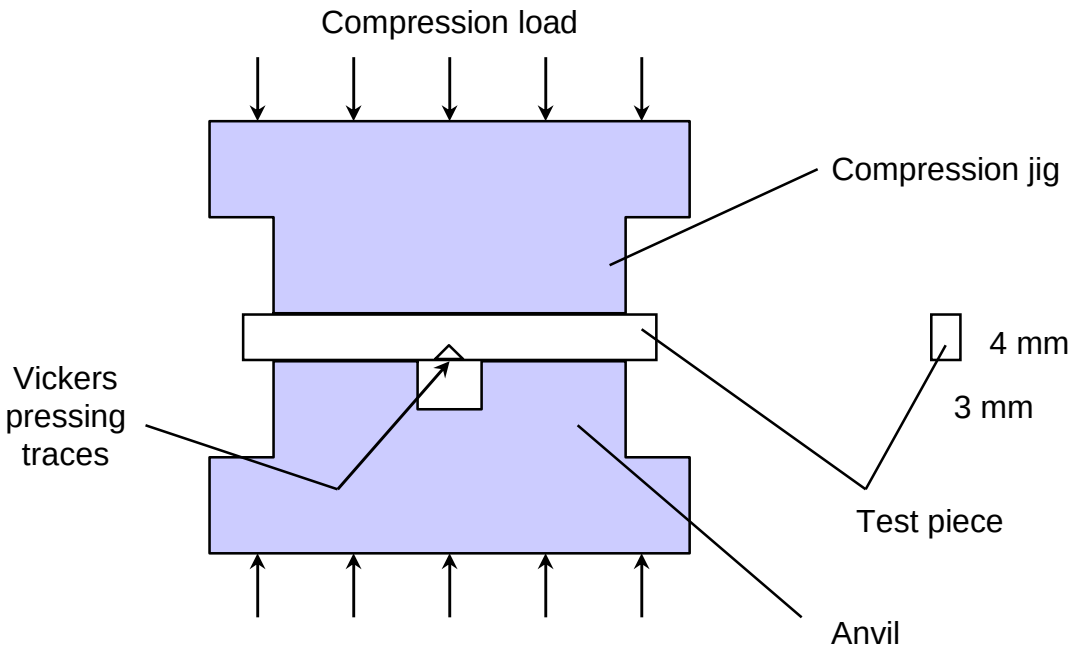
Equipment: Autograph DLS-R-500 (Shimadzu)*
Vickers Hardness Tester
Pre-crack Introducing Jig

Test pieces are made according to JIS R1607.

Size: 3 $^{\pm 0.1}$ x 4 $^{\pm 0.1}$ x 18 mm

Three Vickers pressing traces (dents) are made with a diamond point on the surface of each test piece by a load of 98.07N.

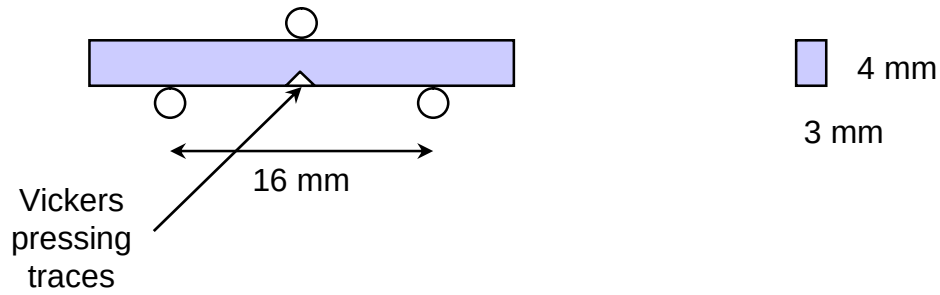
To generate a precrack, the test piece is placed between the compression jig and the anvil as shown below.



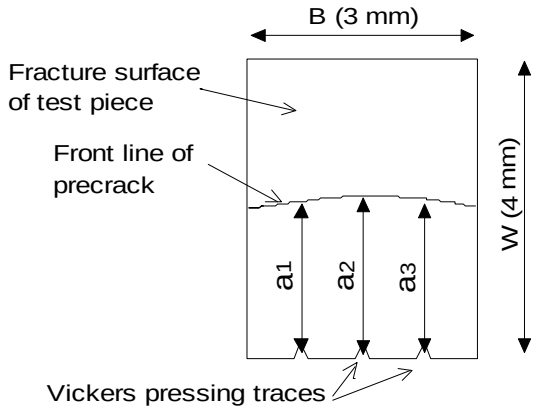
The compression load is increased until a ‘popping’ sound is detected, and then the test piece is immediately removed from the load.

A three-point bending test is done on each test piece.

Span:	16 mm
Cross head speed:	0.5 mm/min



After fracture, the length of the precrack is measured.



$$a = \frac{a_1 + a_2 + a_3}{3}$$

a: length of precrack (m)

Fracture toughness (K_{IC}) is calculated by the following numerical expression:

$$K_{IC} = \left(\frac{PS}{BW^{3/2}}\right)\left[\frac{3}{2}\left(\frac{a}{W}\right)^{1/2} \cdot Y\left(\frac{a}{W}\right)\right]$$

P: fracture load (N)
S: span length (m)

$$Y\left(\frac{a}{W}\right) = \frac{1.99 - \frac{a}{W}\left(1 - \frac{a}{W}\right)\left\{2.15 - 3.93\frac{a}{W} + 2.7\left(\frac{a}{W}\right)^2\right\}}{\left(1 + 2\frac{a}{W}\right)\left(1 - \frac{a}{W}\right)^{3/2}}$$

11. Hardness

Equipment: Vickers Hardness Tester (MATSUZAWA SEIKI)*

The surface of the test piece is polished with a diamond powder slurry (size: 3 μm). A Vickers pressing trace is made by 98.07N of pressure. The diagonal length of the Vickers pressing trace is measured by using a microscope.

The hardness is calculated by the following numerical expression:

$$HV10 = \frac{0.1891 \times F}{d^2}$$

F: load (98.07N)
d: diagonal length of Vickers pressing trace

** The name in parentheses indicates the testing machine manufacturer.*