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Fine ceramics (advanced ceramics, advanced technical ceramics) — Test method for fracture resistance of silicon nitride materials for rolling bearing balls at room temperature by indentation fracture (IF) method

Céramiques techniques — Méthode d'essai de résistance à la rupture des matériaux nitrure de silicium pour billes de roulement à billes à température ambiante par la méthode de rupture par indentation

Reference number
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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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ISO 14627 was prepared by Technical Committee ISO/TC 206, *Fine ceramics*.

Fine ceramics (advanced ceramics, advanced technical ceramics) — Test method for fracture resistance of silicon nitride materials for rolling bearing balls at room temperature by indentation fracture (IF) method

1 Scope

This International Standard describes a test method that covers the determination of fracture resistance of silicon nitride bearing balls at room temperature by the indentation fracture (IF) method, as specified in ISO 26602.

This International Standard is intended for use with monolithic silicon nitride ceramics for bearing balls. It does not include other ceramic materials.

This International Standard is for material comparison and quality assurance.

Indentation fracture resistance, $K_{I,\ IFR}$ as defined in this International Standard is not to be equated to fracture toughness determined using other test methods such as K_{Isc} and K_{Ipb}.

NOTE $K_{I,\ IFR}$ is an estimate of a material's resistance to cracking as introduced by an indenter and has correlations with wear resistance and rolling contact fatigue performance as well as machining processes used for silicon nitride materials since these properties are governed by the resistance to crack extension in localized damage areas. By contrast, fracture toughness, K_{Isc} and K_{Ipb} are intrinsic properties of a material and are relevant to macroscopic and catastrophic fracture events with long cracks, rather than those phenomena caused by microscopic and successive damage accumulation associated with short cracks.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 4287:1997, *Geometrical Product Specifications (GPS) — Surface texture: Profile method — Terms, definitions and surface texture parameters*

ISO 6507-2:2005, *Metallic materials — Vickers hardness test — Part 2: Verification and calibration of testing machines*

ISO 6507-3:2005, *Metallic materials — Vickers hardness test — Part 3: Calibration of reference blocks*

ISO/IEC 17025:2005, *General requirements for the competence of testing and calibration laboratories*

ISO 17561:2002, *Fine ceramics (advanced ceramics, advanced technical ceramics) — Test method for elastic moduli of monolithic ceramics at room temperature by sonic resonance*

ISO 26602:2009, *Fine ceramics (advanced ceramics, advanced technical ceramics) — Silicon nitride materials for rolling bearing balls*

3 Terms and definitions

For the purpose of this document, the following terms and definitions apply.

3.1
fracture resistance
$K_{\mathrm{I,\,IFR}}$
fracture resistance measured by the IF method

[ISO 26602:2009]

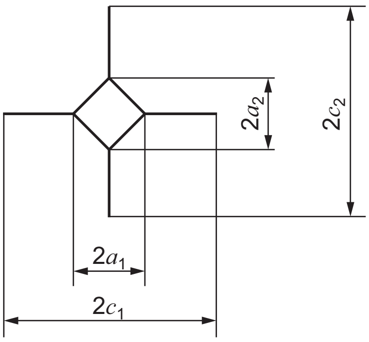
NOTE The indentation fracture resistance, $K_{\mathrm{I,\,IFR}}$, as used here is not to be equated to fast fracture toughness K_{Ic}. $K_{\mathrm{I,\,IFR}}$ is an estimate of a material's resistance to cracking as introduced by an indenter. K_{Ic} is considered to be an intrinsic property of a material and is independent of the test method.

3.2
preprocessed ball
densified unit of material from a material lot before final shaping and finishing

[ISO 26602:2009]

4 Principle

This International Standard is for material comparison, quality assurance of silicon nitride materials for rolling bearing balls as described in ISO 26602. The method determines the indentation fracture resistance value, $K_{\mathrm{I,\,IFR}}$, by forcing a Vickers indenter (diamond pyramid) into the surface of a test piece and measuring the lengths of both the diagonals and the associated cracks of the indentation that are left in the surface after removal of the indenter. See Figure 1.



Key
$2a$ is the diagonal length of the indent
$2c$ is the diagonal length of the crack

Figure 1 — Crack lengths and diagonal sizes of the Vickers indentation

5 Apparatus

5.1 Testing machine

The testing machine shall be in accordance with ISO 6507-2.

5.2 Indenter

The indenter shall meet the specification for the Vickers indenter. See the test method in ISO 6507-2. The diamond should be examined before the test, and if it is loose in the mounting material, chipped, or cracked, it shall be replaced.

5.3 Verification by standard reference materials

The reference materials which are in accordance with ISO 6507-3 shall be used to verify the testing machine and their Vickers hardness shall not vary from the hardness of the material to be measured by more than 20 %.

6 Test specimens

6.1 General

The ground and polished sections of the preprocessed balls or the finished products shall be used for the indentation fracture (IF) method. The surface finishing shall be done to avoid residual stress.

6.2 Thickness

The thickness of the specimen shall be large enough so that the crack lengths are not affected by variations in the thickness. As long as the thickness of the specimen is more than five times the crack length ($2c$), the test will not be affected. In general, a specimen thickness of more than 3 mm is suitable.

6.3 Surface finish

Specimens shall have a ground and polished surface so that the crack lengths can be measured accurately. The surface roughness, R_a, as defined in ISO 4287 shall be not more than 0,1 μm. Any grinding-induced damage layer at the surface shall be removed completely by polishing so that the crack lengths are not affected by any residual stress at the surface layer.

NOTE If the specimen test area is insufficient to permit at least five valid indentations (see Figure 2) to be obtained using the spacing criterion given in 7.6, e.g. if bearing balls are employed, then it is permitted either to employ a larger test-piece from the same material lot, or several randomly selected bearing balls from the same lot.

7 Procedure

7.1 Specimen placement

Place the specimen on the stage of the machine so that the specimen will not rock or shift during the measurement. The specimen surface shall be clean and free from any grease or film.

7.2 Specimen leveling

The surface of the specimen being tested shall lie in a plane normal to the axis of the indenter.

7.3 Clean the indenter

The indenter shall be cleaned prior to and during a test series. A cotton swab with ethanol, methanol or isopropanol may be used. Indenting into soft copper also may help remove debris.

7.4 Application of test load

Indentations shall be made using a Vickers indenter under the following conditions:

— Load 196,1 N

— Dwell Time 15 s

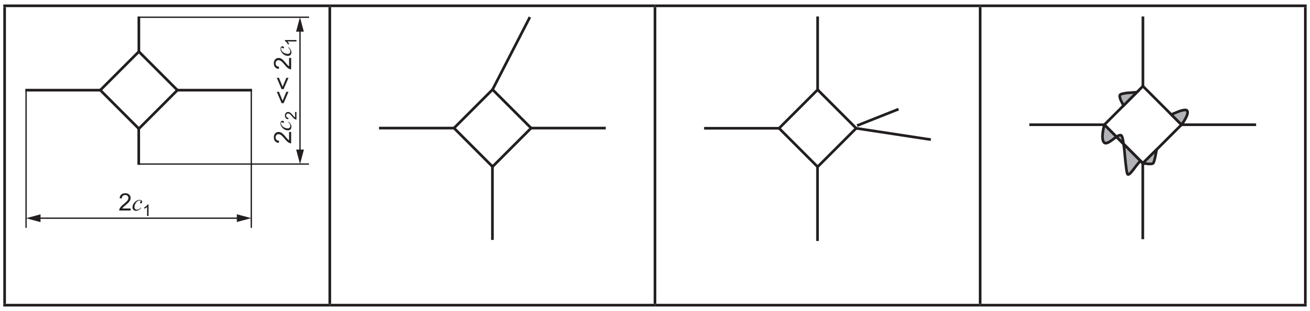
If indentations made at the test load of 196,1 N lead to no acceptable indentations (see Figure 2), use a lower test load of 98,07 N. The test load employed shall be described in the test report to show the precision of measurements.

NOTE 1 $K_{\mathrm{I,\ IFR}}$ measured at a load of 98,07 N may be slightly smaller than that obtained at 196,1 N, especially for those silicon nitrides with self-reinforced microstructures which produce rising R-curve behavior. Also, the accuracy of the measurement of crack length may become worse when the indentation size gets smaller at the test load of 98,07 N.

NOTE 2 If the thickness of the tiny ball specimen is not more than five times the crack length ($2c$), or the specimen test area is insufficient for a single indentation to satisfy the spacing criterion given in 7.6 (see Figure 3), a test load lower than 98,07 N is permitted by mutual agreement between the customer and vendor.

7.5 Acceptability of indentations

Only indentations whose four primary cracks emanate straight and radially from each corner shall be accepted. Indentations with badly asymmetrical, split or forked cracks or with gross chipping shall be rejected. If the difference between the horizontal crack length and the vertical one is more than 10 % of the mean value of the horizontal and vertical ones, the result shall be rejected.

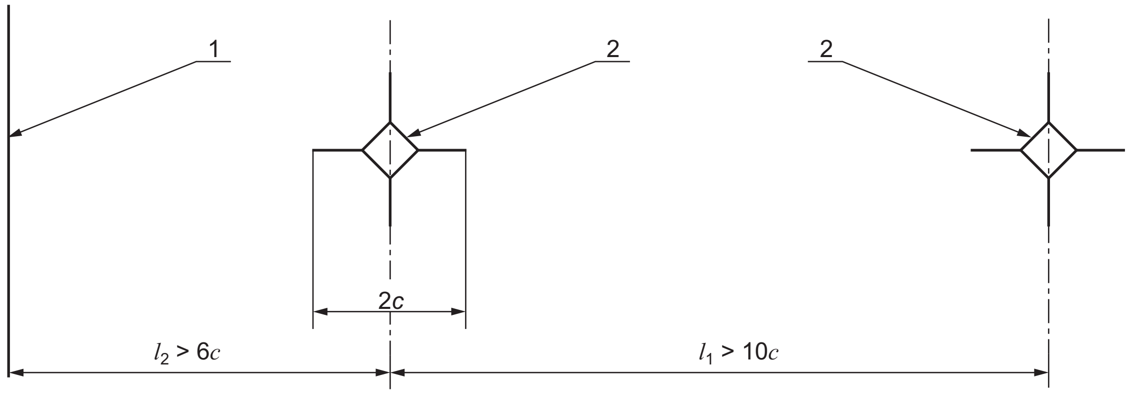


asymmetrical **asymmetrical** **badly split** **chipping and ragged edges**

Figure 2 — Guidelines for the unacceptable indentations

7.6 Number of indentations

The number of valid indentations shall be not less than five. The spacing of indentations shall not be less than five times the diagonal length of cracks. No indentation shall be closer to the test-piece edge than three times the diagonal length of the crack.



Key

1 edge of test piece

2 indentations

$2c$ is the diagonal length of the crack

l_1 is the distance between centres of indentations

l_2 is the distance from the centre of indentation to the edge of sample

Figure 3 — Closest permitted spacing between indentations and from indentation to the test piece edge for Vickers indentations

7.7 Measurement of indentation size

Measure both diagonals of each hardness impression as “2*a*” values to within 2 μm within 10 min after unloading, e.g. with an optical microscope attached to the hardness tester, a metallurgical microscope or a traveling microscope.

7.8 Measurement of crack size

Enlarge the indentation by at least 100x using an optical microscope attached to the hardness tester, a metallurgical microscope or a traveling microscope, and measure visible tip-to-tip crack lengths associated with the hardness impression as “2*c*” values to within 10 μm within 10 min after unloading. Crack observations with higher total magnification, e.g. not less than 200x by using a Charge Coupled Device (CCD) camera or a traveling microscope, are preferred to minimize measuring errors.

NOTE The achievable precision of crack size measurement depends upon the microstructure of material, the surface finish and the magnification of a microscope. Reading crack lengths of indentations in bearing-grade silicon nitrides with a total magnification of 100x will enable the precision suggested in 7.8 to be achieved by well-trained operators. The calculated indentation fracture resistance, $K_{\mathrm{I,\,IFR}}$, is sensitive to the precision of the crack length measurements, as is obvious from Equation (3) in 8.3. Thus, employing higher magnification greater than 100x is preferable to ensure the validity of the test results.

The magnification employed shall be described in the test report to show the precision of the measurements.

8 Calculation

8.1 For each indentation, calculate the mean values of 2*a* and 2*c*.

$$2a = \frac{2a_1 + 2a_2}{2} \quad \text{and} \quad 2c = \frac{2c_1 + 2c_2}{2} \tag{1}$$

8.2 For each indentation, calculate the Vickers hardness value as follows:

$$HV = 0{,}001\,854 \frac{P}{(2a)^2} \tag{2}$$

where:

HV is the Vickers hardness, in gigapascals (GPa);

P is the applied load, in newtons (N);

a is the mean half-length diagonal value, in millimetres (mm).

8.3 For each indentation, calculate the indentation fracture resistance by Niihara's method as follows:

$$K_{\mathrm{I,IFR}} = 0{,}000\,978 \left(\frac{E}{HV}\right)^{0{,}4} \left(\frac{P}{c^{1{,}5}}\right) \tag{3}$$

where:

$K_{\mathrm{I,\,IFR}}$ is the indentation fracture resistance, in megapascals square root metre (MPa·m$^{1/2}$);

E is the elastic modulus, in gigapascals (GPa);

HV is the Vickers hardness, in gigapascals (GPa);

P is the applied load, in newtons (N);

c is the mean half-crack length, in millimetres (mm).

NOTE It is proven that at loads of 98,07 N and above only half-penny-type cracks are introduced for silicon nitride ceramics, References [1, 2]. Therefore, the ratio of $2c/2a$ and the equation for the Palmqvist crack have not to be considered.

The sonic resonance method which is in accordance with ISO 17561 shall be used to measure the elastic modulus of silicon nitrides, provided that the test-piece is available from the same material lot as the bearing balls. If the test piece for the sonic resonance method is not available from the same material lot, it is permitted to use literature data.

9 Test report

The test report shall be in accordance with ISO/IEC 17025, unless there are valid reasons for not doing so. The report of the results of an indentation fracture resistance test shall include the following items:

a) the name and address of the testing establishment;

b) the date of the test, a unique identification of the report and of each page, customer name and address, signatory of the report;

c) a reference to this International Standard, i.e. determined in accordance with ISO 14627;

d) a description of the test material, elastic modulus, batch codes, date of manufacture, as appropriate;

e) geometry and dimensions of test specimen;

f) sampling conditions of test specimen from material and its machining conditions (when a specimen is heat treated, its conditions are included);

g) name of testing machine and its type;

h) name of the microscope and its type;

i) magnification used to observe and measure the cracks;

j) environment of test, relative humidity, temperature;

k) indentation load;

l) elastic modulus used to calculate the indentation fracture resistance as well as the measuring method, i.e. determined with ISO 17561. If literature data was used, the source of the data should be provided;

m) a list of test results (number of valid indentations measured, as well as the total number of indentations, mean indentation fracture resistance and the standard deviation);

n) deviations from the specified procedures, if any;

The report of the results of a indentation fracture resistance test should preferably also include the following items:

o) kinds of additives and sintering method of test material;

p) mechanical properties of test material such as bending strength, Vickers hardness, etc.

Bibliography

[1] LUBE, T. Indentation crack profiles in silicon nitride, *J. Eur. Ceram. Soc*., **21**, 2001, pp. 211-218

[2] MIYAZAKI, H., HYUGA, H., YOSHIZAWA, Y., HIRAO, K. and OHJI, T. Crack profiles under a Vickers indentation in silicon nitride ceramics with various microstructures, *Ceram. Int*., **36**, 2010, pp. 173-179

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