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Glass and glass-ceramics — Knoop hardness test

Verres et vitrocéramiques — Essai de dureté Knoop

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Foreword

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Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

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Introduction

For testing the hardness of glass and glass-ceramics, two methods of test have been discussed, namely the Vickers hardness test and the Knoop hardness test. Both tests involve certain difficulties when carried out. The results of a round robin testing survey, carried out in 1974 by the International Commission on Glass (ICG) to compare both procedures, showed that, for the specific task of testing glass, the Knoop hardness test is preferable.

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Glass and glass-ceramics — Knoop hardness test

1 Scope

This International Standard describes the method of test for determining the Knoop hardness number of glass and glass-ceramics.

The Knoop hardness number characterizes the behaviour of glass and glass-ceramics concerning a permanent change of the surface after indentation with a solid material.

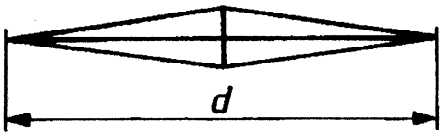

Figure 1 — Base area of the indentation produced by the Knoop indenter

2 Definition, symbol and designation

The Knoop hardness number, HK, is proportional to the quotient of test force F and the projected area A_p of the permanent indentation given by numerical value equation (1):

$$\mathrm{HK} = 0{,}102\,\frac{F}{A_p}$$

$$= 0{,}102\,\frac{F}{d^2 \times 0{,}5\left(\cot\frac{172{,}5°}{2} \times \tan\frac{130°}{2}\right)}$$

$$= 14{,}229 \times \frac{0{,}102F}{d^2} \qquad \ldots (1)$$

where

F is the test force, in newtons;

A_p is the projected area, in square millimetres, of the permanent indentation;

d is the length, in millimetres, of the long indentation diagonal.

NOTE 1 The factor 0,102 in numerical value equation (1) became necessary through the introduction of the SI-unit newton for the test force instead of kilogram-force to avoid changing of the value of the Knoop hardness number.

This indentation is regarded as a straight pyramid with a base area as shown in figure 1, a long indentation diagonal d and identical transverse area angles to those of the indenter (see figure 2).

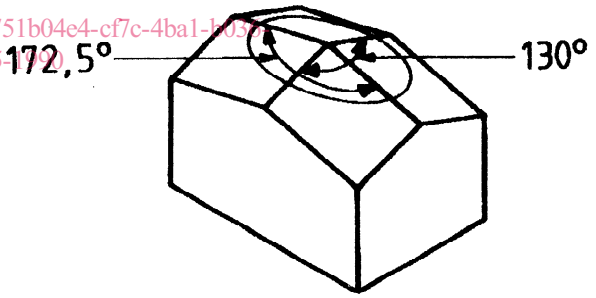

Figure 2 — Knoop indenter

The symbol HK is supplemented:

a) by a number indicating the test force used, expressed in newtons multiplied by 0,102 and therefore equal to the test force expressed in kilogram-force, and

b) by a number indicating the duration of test force application in seconds.

EXAMPLE

490 HK 0,1/20 means the Knoop hardness number 490 HK resulting from an applied test force of 0,980 7 N (× 0,102 equals 0,1). This test force was applied for 20 s.

3 Principle

The Knoop indenter is forced vertically into the surface of a test specimen of glass or glass-ceramics by different test forces for 20 s. Within a 6 min period after removal of the indenter, the length of the long indentation diagonals, left in the surface, are measured using a microscope.

4 Apparatus

4.1 Hardness tester

The hardness tester shall meet the following requirements:

a) the relative error of the test force shall be not greater than 1 %;

b) the design of the loading device shall be such that the indenter is vertically lowered, free from stress and vibration, on to the surface of the test specimen at a rate of (0,20 ± 0,05) mm/min;

c) the length of the long indentation diagonal shall be measurable with an uncertainty less than 0,5 µm;

d) the microscope measurement shall be carried out with the effective numeric aperture of the objective used of $0{,}7\ ^{+0,1}_{0}$;

e) the light used for the measurement shall be yellow-green.

NOTE 2 The effective numerical aperture probably differs with the data on the objective. It is recommended to inquire about this from the manufacturer of the hardness tester.

4.2 Knoop indenter

The Knoop indenter consists of a diamond-tipped right pyramid with a rhomboid base as shown in figure 2, whose axis is coincident with the axis of the test force. The Knoop indenter shall be of a shape so that the factor

$$0{,}5\left(\cot\frac{172{,}5°}{2}\times\tan\frac{130°}{2}\right)=70{,}28\times10^{-3}$$

in numerical value equation (1) has a deviation no greater than 1 %.

The faces and edges of the Knoop indenter shall be smooth and free from cracks or other faults.

It is recommended to use only Knoop indenters which have been certified by a testing authority.

4.3 Verification of apparatus

It is recommended to test the function of the hardness tester regularly, including the Knoop indenter, by measurements of standardized test plates of known hardness, preferably made of glass.

Inspect the Knoop indenter microscopically from time to time to detect any breakages or damage to the edges.

5 Test specimen

The test surface of the specimen shall be flat. It shall be fire-polished or optically ground and polished.

Immediately before testing, it shall be polished with water and cerium oxide until dry.

The minimum thickness of the specimen shall be 0,5 mm.

6 Procedure

The hardness test shall be carried out at a temperature from 18 °C to 28 °C, unless otherwise agreed upon.

Place the test specimen on the stage of the hardness tester so that the test surface of the test specimen lies in a plane normal to the axis of the test force and the axis of the indenter (diamond). During the test, the test specimen shall remain in this position.

Clean the Knoop indenter by pressing it into copper or steel of low hardness, or by means of a suitable solvent not harmful to the hardness tester.

Apply the test forces for 20 s to produce the indentations, the long diagonal dimensions of which are used to calculate the Knoop hardness number. During the indenting time, no shocks or vibrations shall be allowed to change the test force. Within a 6 min period after removing the test force, measure the length of the long indentation diagonal twice, using the microscope. The values obtained shall not differ by more than 2,0 µm.

For the determination of the Knoop hardness number, apply the test force of 0,980 7 N. To reduce uncertainty arising from only one test force value, apply in addition to the test force of 0,980 7 N, on the same day, test forces of two other values which are not likely to create excessive fracture.

In the case where only the test force of 0,980 7 N is used, repeat the procedure at least five different times. For three different test forces, repeat the procedure once the next day.

Apply the different indentations on the same test specimen; the distance between indentations shall

be at least three times the length of the short indentation diagonal.

In the case that a measurement of the long indentation diagonal is impeded by dust particles at the end of the indentation diagonal or if the measuring results of one indentation differ by more than 2,0 µm, this indentation and the measurements shall be repeated.

Determine the arithmetic means of the long indentation diagonals for each condition of indentation.

In the case where three values of test force are used, plot the results on a graph as follows:

Plot the values of lg F on the abscissa and the values of lg d on the ordinate. If all the measurements were carried out properly (for instance, no dust particles, no excessive fractures, etc.), all the points are situated on a straight line. The 0,980 7 N point shall be determined from the straight line and used as d for the calculation of the Knoop hardness number using numerical value equation (2) (see clause 7). Sometimes a point is somewhat apart from the straight line, due to difficulties in the measurement of this point. In this case, repeat the measurements, including a fourth test force.

7 Expression of results

The Knoop hardness number HK 0,1/20 is calculated from numerical value equation (2)

$$\mathrm{HK0{,}1/20} = \frac{1{,}423}{d^2} \quad \ldots (2)$$

Insert the length d, in millimetres, of the long indentation diagonal obtained either by direct measurement or by determination from the straight line for a test force of 0,980 7 N in numerical value equation (2).

8 Test report

The test report shall include the following information:

a) reference to this International Standard;

b) type and designation of the glass or the glass-ceramics;

c) history of the test specimen, if known;

d) Knoop hardness number HK 0,1/20 rounded to the nearest 10 HK 0,1/20;

e) a statement whether the Knoop hardness number was obtained by direct measurement or by determination from the straight line.

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